Certified Carbon Isotope ($\delta^{13}C_{VPDB-LSVEC}$) Reference Materials

UME CRM 1309 - Sucrose
UME CRM 1310 – Glucose
UME CRM 1311 - Fructose
UME CRM 1312 – Honey (Unadulterated)
UME CRM 1313 – Honey (Adulterated)

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ABBREVIATIONS

ANOVA	Analysis of variance
CRM	Certified Reference Material
EA	Elemental Analyzer
IAEA	International Atomic Energy Agency (Austria)
IJS	Institute Jozef Stefan (Slovenia)
IRMS	Isotope Ratio Mass Spectrometry
ISO	International Organization for Standardization
LGC	Laboratory of the Government Chemist (UK)
LTS	Long Term Stability
$MS_{between}$	Mean square between-bottle from ANOVA
MS_{within}	Mean square within-bottle from ANOVA
NIM	National Institute of Metrology (China)
NMIA	National Measurement Institute (Australia)
RSD	Relative standard deviation
RSIL	Reston Stable Isotope Laboratory (USA)
SI	International System of Units
STS	Short Term Stability
TRaNS	Random Stratified Sampling Software
VPDB-LSVEC	Vienna Peedee Belemnite-Lithium carbonate prepared by H. J. Svec

SYMBOLS

α	Significance level
δ	Delta value
N	Number of replicates per unit
n	Number of laboratories with accepted results
s	Standard deviation
s_{bb}	Between-bottle standard deviation
$s_{bb,rel}$	Relative between-bottle standard deviation
ss	Standard deviation of accepted means of participating laboratories
s_{wb}	Within-bottle standard deviation
$s_{wb,rel}$	Relative within-bottle standard deviation
u_{bb}	Standard uncertainty related to possible between-bottle heterogeneity
$u_{bb,rel}$	Relative standard uncertainty related to possible between-bottle heterogeneity
u^*_{bb}	Standard uncertainty of heterogeneity that can be hidden by method repeatability
$u^*_{bb,rel}$	Relative standard uncertainty of heterogeneity that can be hidden by method repeatability
u_{char}	Standard uncertainty related to characterisation
$\overline{u}_{labs}$	Mean of standard uncertainties reported by the participating laboratories
u_{lts}	Standard uncertainty related to long term stability
u_{sts}	Standard uncertainty related to short term stability
$\bar{t}$	Mean of all time points
t_α	Two-tailed t-critic value (t test)
t_i	Time point for each paralel

ABSTRACT

This report explains the various stages of production of Certified Reference Materials of UME CRM 1309 (sucrose), UME CRM 1310 (glucose), UME CRM 1311 (fructose), UME CRM 1312 (unadulterated honey), UME CRM 1313 (adulterated honey) and certification of their $\delta^{13}C_{VPDB\text{-}LSVEC}$ (delta) values. These materials have been produced according to the requirements of the ISO 17034:2016 [1]. Sucrose, glucose and fructose materials (with at least 99% purity) and honey samples (unadulterated and adulterated) were obtained from different producers and were used as starting raw materials of candidate reference materials.

Homogenity between units and stability tests were carried out according to ISO 33405:2024 [2]. Characterization strategy was planned to utilize one or more laboratories applying one or more independent methods according to the ISO 17034. Preliminary measurements, filling, labelling, homogenity, short and long term stability tests were carried out in TÜBİTAK UME. Characterization study was performed by organising interlaboratory comparisons. UME CRM 1309 (sucrose), UME CRM 1310 (glucose), UME CRM 1311 (fructose), UME CRM 1312 (unadulterated) and UME CRM 1313 (adulterated honey) were sent to local laboratories for the measurement of the $\delta^{13}C_{VPDB\text{-}LSVEC}$ values. In addition, the reference material UME CRM 1312 was sent to the national metrology institutes participating the CCQM-K140 key comparison organized by CCQM-IAWG (Consultive Committee for Amount of Substance, Inorganic Analysis Working Group), and coordinated by LGC for determination of the $\delta^{13}C_{VPDB\text{-}LSVEC}$ values. The $\delta^{13}C_{VPDB\text{-}LSVEC}$ values of UME CRM 1312 obtained from the CCQM-K140 comparison results are given under the “Additional Information” section. When the results of UME CRM 1312 obtained from CCQM-K140 and local interlaboratory comparison studies were compared, it can be concluded that the $\delta^{13}C_{VPDB\text{-}LSVEC}$ values in both studies are not significantly different from each other.

INTRODUCTION

Honey is a commonly used food product having increasing commercial importance in recent years. Having a major place among the agricultural products consumed in Türkiye, it is becoming more important to understand whether it is adulterated or not. The offical paper published the Turkish Food Codex for honey by the Ministry of Food, Agriculture and Livestock outlined the quality parameters for honey [3].

It is not always possible to determine added sugars in honey by classical laboratory methods. So, isotope ratio of $\delta^{13}C_{VPDB\text{-}LSVEC}$ method is used to determine the adulteration of honey. Both honey itself and its protein content (extracted from honey) are efficiently analyzed by Isotope Ratio Mass Spectrometry (IRMS) to determine $\delta^{13}C_{VPDB\text{-}LSVEC}$ values in order to detect adulteration in honey. $\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰) values are expected to vary from -23 to -28 for unadulterated honey and -9 to -15 for adulterated honey [4,5]. Therefore, in order to provide the field laboratories suitable reference materials, it is needed to have reference materials with $\delta^{13}C_{VPDB\text{-}LSVEC}$ certified values within the the described range.

Certified reference materials (CRM) are used for method validation as well as calibration or correction of raw measurement results. Use of CRMs is one of the most important tools for assuring

measurement quality. Proper use of CRM is also important as well as correct choice of the appropriate CRM .

Production and certification of UME CRM 1309, UME CRM 1310, UME CRM 1311, UME CRM 1312 and UME CRM 1313 were carried out in accordance with the requirements of the ISO 17034 and ISO 33405 by using TUBITAK UME infrastructure. Production and certification of these materials are planned considering the needs of field laboratories, and lack or cost of available CRMs. The average value obtained from the accepted comparison study results has been used in the value assignment of the CRMs.

PARTICIPANTS

Laboratories contributing to the sampling, processing, homogeneity, stability and characterization and international intercomparison studies are given in Table 1.

Table 1. Institutions and organizations involved in the production and certification process

Activity	Laboratory
Project Management and Data Evaluation	• TÜBİTAK Ulusal Metroloji Enstitüsü (UME), Gebze, Kocaeli, Türkiye
Sampling and processing	• TÜBİTAK Ulusal Metroloji Enstitüsü (UME), Gebze, Kocaeli, Türkiye
Homogenity study	• TÜBİTAK Ulusal Metroloji Enstitüsü (UME), Gebze, Kocaeli, Türkiye
Stability study	• TÜBİTAK Ulusal Metroloji Enstitüsü (UME), Gebze, Kocaeli, Türkiye (Short-term and long-term stability studies)
Characterization study (in alphabetical order)	• Akdeniz Üniversitesi, Gıda Güvenliği ve Tarımsal Araştırmalar Merkezi, Kampüs, 07058, Antalya, Türkiye • Altıparmak Gıda San. ve Tic. A.Ş. Çavuşbaşı Cad., No:70 Çekmeköy, İstanbul, Türkiye • Arıgıda Kontrol ve Araştırma Laboratuvarı, Bağlar Mah. Endüstri Sok. No:7 Kozan, Adana, Türkiye • Ege Üniversitesi (Argefar), İlaç Geliştirme ve Farmakokinetik Araştırma ve Uygulama Merkezi, Çevre ve Gıda Analizleri Laboratuvarı, 35100, Bornova, İzmir, Türkiye • Türkiye Atom Enerjisi Kurumu (TAEK), Sarayköy Nükleer Araştırma ve Eğitim Merkezi, Saray Mah. Atom Cad. No:27, 06983, Kazan, Ankara, Türkiye • Türk Standartları Enstitüsü (TSE), TSE Gebze Kalite Kampüsü, Cumhuriyet Mah. Çayırova tren istasyonu yanı, 2258 Sok. Gebze, Kocaeli, Türkiye • TÜBİTAK Ulusal Metroloji Enstitüsü (UME), Gebze, Kocaeli, Türkiye
CCQM-K140 Intercomparison Study for UME CRM 1312 (in alphabetical order)	• Jozef Stefan Institute (IJS), Jamova 39, 1000 Ljubljana, Slovenia • LGC Ltd, Queen`s Road, Teddington, Middlesex, TW11 OLY, UK • National Measurement Institute, 105 Delhi Rd, North Ryde, NSW 2113, Australia • National Institue of Metrology, No.18, Bei San Huan Dong Lu, Chaoyang District, China • TÜBİTAK Ulusal Metroloji Enstitüsü (UME), Gebze, Kocaeli, Türkiye

MATERIAL PROCESSING

Candidate reference materials, sucrose (Sigma-Aldrich, Cas no: 57-50-1, Cat. No: S7903), glucose (Merck, Cas No: 50-99-7, Cat No: 346351) and fructose (Sigma-Aldrich, Cas No: 57-47-7, Cat. No: F0127) with purities of ≥99% were purchased from local representatives. Unadulterated and adulterated honey were purchased from local grocery stores. Sucrose, glucose and fructose in powder form were homogenised with 3D mixer (Willy A. Bachofen AG Maschinenfabrik, TURBULA® T10-B, Switzerland) and honey samples were homogenised with a mechanical stirrer (Heidolph, RZR 2051 Control, Germany). Homogenised products were weighed and filled into the 5 ml amber glass bottles as 200 bottles for each product. Filling proccess was completed by filling powder and honey as approximately 1 and 2 grams in to each bottle, respectively.

$\delta^{13}C$ values of supplied samples were determined for the preevaluation of the raw material's isotope ratios of $^{13}C/^{12}C$. It was aimed to determine the difference in their $\delta^{13}C_{VPDB\text{-}LSVEC}$ values in order to choose unadultrated and adulterated honey samples. The $\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰) values of unadulterated and adulterated honey samples varies from -23 to -28 and from -9 to -15, respectively [4,5]. In the preliminary studies, the $\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰) values of honey samples were found approximately as -24 and -12 and these honey samples were described as unadulterated and adulterated honey, respectively. The $\delta^{13}C_{VPDB\text{-}LSVEC}$ value of unadulterated honey and its extracted protein content were determined seperately to select unadulterated candidate reference material. The criterion to specify the honey as unadulterated is that the difference between the $\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰) value of honey and its protein should be less than 1 [4,5]. After analyzing this honey sample, the value was found to be less than 1 and this sample was reserved as candidate reference material for production and certification. The $^{13}C/^{12}C$ isotope ratio determination was performed by EA-IRMS (Thermofinnigan MAT 253, Germany) instrument. All candidate reference material units (except stability test units) were stored at reference temperature (18 °C) for homogeneity, characterization and post-certification monitoring until analysis. Target $\delta^{13}C_{VPDB\text{-}LSVEC}$ range of all candidate reference materials are given in Table 2.

Table 2. Target $\delta^{13}C_{VPDB\text{-}LSVEC}$ values of all candidate reference materials

Material	Target range, $\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰)
Sucrose (UME CRM 1309)	-25 – -28
Glucose (UME CRM 1310)	-10 – -12
Fructose (UME CRM 1311)	-10 – -12
Honey, unadulterated (UME CRM 1312)	-24 – -27
Honey, adulterated (UME CRM 1313)	-11 – -18

HOMOGENEITY

Homogeneity study between the units is performed to show that assigned value is valid for all units within the stated uncertainty. Homogeneity study between the units is performed with number of samples representing the whole batch. In this project, 20 units (10 spare) are selected by using random stratified sampling software (TRaNS) and were reserved for the study of homogeneity between units. Homogeneity tests were carried out for all candidate CRMs by measuring 3 sub-samples under the repeatability conditions. The method used for these measurements was validated

and the samples to be analysed were introduced to the instrument by random order to find out any trend arising from analytical and/or filling sequences. Certified reference materials and samples were analysed in the same run. All measurements were carried out using EA-IRMS. All the data for homogeneity measurements are given in Annex 1 and the plots are given in Annex 2.

The data for all CRMs were evaluated statistically by regression analysis for the presence of any trend in analytical and filling sequence. After evaluation of data, no trend was found for any CRM candidate at 95% and 99% confidence levels.

Grubbs test was applied to all data for the presence of outlier at 95% and 99% confidence levels. According to data obtained for each CRM, it was found that the distribution was found normal and no outlier was found (Table 3).

Table 3. Statistical Evaluation Result of Homogeneity (CRM 1309-1313)

CRM	Is there a Trend?		Is there an Outlier?		Distribution
	Analytical sequence	Filling sequence	All data	Unit averages	All data
Sucrose (UME CRM 1309)	No	No	No	No	Normal/unimodal
Glucose (UME CRM 1310)	No	No	No	No	Normal/unimodal
Fructose (UME CRM 1311)	No	No	No	No	Normal/unimodal
Honey, unadulterated (UME CRM 1312)	No	No	No	No	Normal/unimodal
Honey, adulterated (UME CRM 1313)	No	No	No	No	Normal/unimodal

Analysis of Variance (ANOVA) is a statistical tool used to estimate the uncertainty contribution from homogeneity of the materials. All data were examined for normal data distribution using Shapiro-Wilk test and histograms before applying one way ANOVA test. All CRMs showed normal distribution on Shapiro-Wilk test and histogram diagrams. Uncertainities of homogeneity between units were evaluated with one way ANOVA for all CRMs. The following equation (1) is used for repeatability of method (s_{wb}) and equation (2) is used for the calculation of standard deviation between units (s_{bb}).

$$s_{wb} = \sqrt{MS_{within}} \quad (1)$$

where,

MS_{within} : mean of square of variance within the unit

s_{wb} equals to “s” of the method as long as sub samples represent the whole unit.

$$s_{bb} = \sqrt{\frac{MS_{between} - MS_{within}}{n}} \quad (2)$$

where,

$MS_{between}$: mean of square of variance between units

n : number of replicates per unit

$MS_{between}$ is found to be smaller than MS_{within} in conditions for which the heterogeneity of the material is smaller than heterogeneity that can be determined by the applied analytical method or measurement fluctuations that may have occurred randomly. In these cases, since s_{bb} can not be calculated, u^*_{bb} is calculated as heterogeneity contributing to uncertainty including method repeatability using equation (3).

$$u^*_{bb} = \frac{s_{wb}}{\sqrt{n}} \sqrt[4]{\frac{2}{\nu_{MSwithin}}} \qquad (3)$$

where,

$\nu_{MSwithin}$: degree of freedom of MS_{within}

The uncertainity values obtained from the homogeneity study are given in Table 4.

Table 4. Results of Homogeneity Study

CRM	Average value $\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰)	$s_{wb,rel}$ %	$s_{bb,rel}$ %	$u^*_{bb,rel}$ %	$u_{bb,rel}$ %
Sucrose (UME CRM 1309)	-26.75	0.43	0.21	0.14	0.21
Glucose (UME CRM 1310)	-11.05	0.90	$MS_{between}<MS_{within}$	0.29	0.29
Fructose (UME CRM 1311)	-10.74	0.67	$MS_{between}<MS_{within}$	0.22	0.22
Honey, unadulterated (UME CRM 1312)	-24.18	0.26	0.12	0.08	0.12
Honey, adulterated (UME CRM 1313)	-11.58	0.57	0.26	0.18	0.26

The values of $MS_{between}$ were found to be smaller than the values of MS_{within} for CRM 1310 and CRM 1311. For these two cases, $\mathbf{u^*_{bb}}$ is calculated and used as the uncertainty contribution due to homogeneity.

STABILITY

Stability studies were carried out with the simulation of conditions in the laboratory, considering environmental conditions that may occur during shipment to the user and storage conditions.

Short Term Stability Study

Stability studies were performed with isochronous design which is cited in the ISO 33405 [2]. For the Short Term Stability (STS) test, two different temperatures (4˚C and 60 ˚C) and 4 time points (1, 2, 3 and 4 weeks) were tested. 10 samples for each CRM were selected by TRaNS. 8 samples were subjected to the test temperatures for the specified time intervals.

Samples were moved to +18˚C (reference temperature) after completion of the test time. All samples were analysed at the same time. Three replicate samples were prepared from each unit and were analyzed by IRMS-EA under the repeatability conditions for $^{13}C/^{12}C$ isotope ratio delta values.

The data for each temperature were first examined by single Grubbs test for both 95% and 99% confidance intervals to find out outliers. Number of detected outliers are given in the Table 5. Since no technical reason can be found to reject these data, all outliers were included in the STS calculations.

Values calculated for each time point were plotted against the time for the assessment of short term stability. The relationship between variables were analyzed in order to determine if any significant change exists in $\delta^{13}C_{VPDB\text{-}LSVEC}$ values with the testing time (*regression analysis*). It was found that the slopes were not significantly different than zero for all CRMs in the 95% confidence interval. All data related to short term stability are given in Annex 3 and plots are given in Annex 4.

Uncertainty calculations are done using equation (4). Maximum time for transfer is chosen as 4 weeks.

$$u_{lts} = \frac{RSD}{\sqrt{\sum (t_i - \bar{t})^2}} \times t \qquad (4)$$

where,

RSD : relative standard deviation obtained from all data in STS

t_i : time point for each replicate

$\bar{t}$: mean of all time points

t : maximum time suggested for transfer (4 weeks)

Results obtained from short term stability tests are given in Table 5.

Table 5. Short Term Stability Test Results

CRM	4 °C u_{sts}	60 °C u_{sts}	Number of outliers in 95% confidence interval*		Number of outliers in 99% confidence interval*		Is there a significant trend in 95% confidence interval?		Is there a significant trend in 99% confidence interval?	
			4 °C	60 °C	4 °C	60 °C	4 °C	60 °C	4 °C	60 °C
Sucrose (UME CRM 1309)	0.17	0.09	1	1	1	1	No	No	No	No
Glucose (UME CRM 1310)	0.06	0.05	-	-	-	-	No	No	No	No
Fructose (UME CRM 1311)	0.07	0.08	-	-	-	-	No	No	No	No
Honey, unadulterated (UME CRM 1312)	0.05	0.04	1	-	-	-	No	No	No	No
Honey, adulterated (UME CRM 1313)	0.05	0.04	-	-	-	-	No	No	No	No

* Single Grubbs Test

Result of this study showed that the CRMs can be transferred to the end users without applying any cooling elements if the ambient temperature is not exceeding 60 °C and duration is not exceeding 4 weeks. u_{sts} at 60 °C calculated for 4 weeks is used as the contribution of short term stability to the CRM uncertainty.

Long Term Stability Study

Shelf life of the produced CRMs are determined by the long-term stability studies. 25 ˚C was choosen as the test temperature for long term stability tests and totally 52 units (26 spare) for each CRM were reserved for this study. Samples were selected by TRaNS and kept at 25 ˚C for 12 months. Two units for each time point (0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11 and 12 months) were stored at 25 ˚C and transferred to 18 ˚C (reference temperature) after completion of the test time. Three replicate samples were prepared from each unit and were analyzed by IRMS-EA under the repeatability conditions for $^{13}C/^{12}C$ isotope ratio delta values.

The data was first examined by single Grubbs test for both 95% and 99% confidence intervals to find out outliers. Number of detected outliers are given in the Table 6. Since no technical reason can be found to reject these data, all outliers were included in the LTS calculations. All data related with long term stability are given in Annex 5 and plots are given in Annex 6.

Values calculated for each time point were plotted against the time for the assessment of LTS. The relationship between variables were analyzed in order to determine if any significant change exists in $\delta^{13}C_{VPDB\text{-}LSVEC}$ values with the testing time (regression analysis). It was found that the slopes were not significantly different than zero for all CRMs in the 95% confidence interval. Uncertainity contribution of long term stability, u_{lts}, is calculated using equation (5) for 5 years of shelf life at 25 °C.

$$u_{lts} = \frac{RSD}{\sqrt{\sum (t_i - \bar{t})^2}} \times t \qquad (5)$$

where,

RSD : relative standard deviation obtained from all data in LTS

t_i : time point for each replicate

$\bar{t}$: mean of all time points

t : shelf life suggested at 25 °C

Shelf life for CRMs is set as 5 years after sales. Continuous post certification monitoring studies are going to be carried out in order to check the validity of the certified value over longer time.

Table 6. Long Term Stability Test Results

CRM	u_{lts} (‰)	Number of outliers in 95 % confidence interval	Number of outliers in 99 % confidence interval	Is there a significant trend in 95 % confidence interval?	Is there a significant trend in 99 % confidence interval?
UME CRM 1309	0.11	1	1	No	No
UME CRM 1310	0.15	1	1	No	No
UME CRM 1311	0.12	-	-	No	No
UME CRM 1312	0.12	1	-	No	No
UME CRM 1313	0.20	-	-	No	No

CHARACTERIZATION

Characterization and value assignment in accordance with the ISO 17034 can be made in various ways [1]. In this project, characterization was performed for a specifically defined measurand traceable to a specifically defined VPDB-LSVEC scale using a network of competent laboratories. Selection criteria for participating laboratories were:

- expertise in $\delta^{13}C_{VPDB\text{-}LSVEC}$ analysis
- having accreditation for the measurements of same or similar sample matrices
- successful participation in proficiency testing schemes

All CRM candidates (UME CRM 1309, 1310, 1311, 1312 and 1313) were sent to 6 different national laboratories. In addition, UME CRM 1312 (unadulterated honey) was sent to national metrology institutes participated in the CCQM-K140 key comparison exercise. The information about this comparison study is given in “Additional Information” section of this report. List of laboratories participated in characterisation and key comparison excersise are given in Table 1.

Laboratories were used validated methods in their measurements. 2 units were measured by each laboratory, and the selection of these units were made by TRaNS to represent all produced batch. From each unit of candidate reference material, 3 independent measurement results were reported by each laboratory. Overall, for each candidate reference material, each laboratory reported 6 independent measurement results for two units on two different days with measurement uncertainties and details of their methods they used. Details of the reference materials used for calibration/correction was reported as the source of measurement traceability. All participating laboratories used EA-IRMS method in the characterization study. Results obtained from characterization study are given in Annex 7, and characterisation plots are given in Annex 8.

Data obtained from characterization study revealed normal distribution and measurement uncertainities were calculated according to the “Guide to the Expression of Uncertainty in Measurements (GUM)” and “EURACHEM/CITAC Guide Quantifying Uncertainty in Analytical Measurement” documents. The uncertainty of characterization study (u_{char}) was calculated by

combining the results obtained by the participating laboratories and reported according to the ISO 33405.

Characterization standard uncertainty (u_{char}) is calculated using equation (6) by taking into account the uncertainties and the standard deviation of the results reported by the participating laboratories.

$$u_{char} = \sqrt{\overline{u}^2_{labs} + \left(\frac{SS}{\sqrt{n}}\right)^2} \quad (6)$$

where,

u_{char} : Standard uncertainty arising from characterization study

$\overline{u}_{labs}$: Mean of standard uncertainties reported by the participating laboratories

SS : Standard deviation of accepted means of participating laboratories

n : Number of laboratories with accepted results

PROPERTY VALUE AND UNCERTAINTY ASSIGNMENT

Assigned values and uncertainities of the CRMs were evaluated by applying combination approach in the characterization and uncertainty data that contribute to the homogeneity and stability assessments.

Data obtained in the characterization study were checked for normal distribution and outliers. Distributions were found to be normal and no outlier was detected.

Mean value of all accepted characterisation results is assigned as the property value of the reference materials. Formula (7) is used to calculate the combined expanded uncertainty of CRMs [6]:

$$\mathrm{U_{CRM}} = k\sqrt{\mathrm{u^2_{char}} + \mathrm{u^2_{bb}} + \mathrm{u^2_{sts}} + \mathrm{u^2_{lts}}} \quad (7)$$

Uncertainity value on CRM certificate includes uncertainity contribution from characterization (u_{char}), homogeneity (u_{bb}), short term stability (u_{sts}) and long term stability (u_{lts}). Expansion of uncertainity value of CRMs were done with a coverage factor (k=2) representing confidence level of approximately 95%. Certificate values and uncertainities are given in Table 7 and relative percent contribution of each component on uncertainity is given in Table 8.

Table 7. Certificate values and uncertainity components

CRM	Certificate value, $\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰)	U_{CRM}, (‰) $k=2$	u_{char} (‰)	u_{bb} (‰)	u_{sts} (‰)	u_{lts} (‰)
UME CRM 1309	-26.78	0.81	0.37	0.06	0.09	0.11
UME CRM 1310	-11.19	0.68	0.29	0.03	0.05	0.16
UME CRM 1311	-10.97	0.67	0.30	0.02	0.08	0.13
UME CRM 1312	-24.02	0.68	0.31	0.03	0.04	0.12
UME CRM 1313	-11.73	0.70	0.27	0.03	0.04	0.21

Table 8. Percent contribution of each parameter to u_{CRM}

CRM	$u_{char, rel}$ (%)	$u_{bb, rel}$ (%)	$u_{sts, rel}$ (%)	$u_{lts, rel}$ (%)
UME CRM 1309	85.0	2.0	5.5	7.5
UME CRM 1310	73.8	0.9	2.1	23.2
UME CRM 1311	79.6	0.5	5.5	14.4
UME CRM 1312	85.0	0.7	1.1	13.2
UME CRM 1313	62.3	0.8	1.2	35.7

ADDITIONAL INFORMATION

The $\delta^{13}C_{VPDB-LSVEC}$ certificate value of UME CRM 1312 (honey, unadulterated) was also confirmed by CCQM-K140 interlaboratory comparison study participated by national metrology institutes which has been organised by LGC. When the $\delta^{13}C_{VPDB-LSVEC}$ values obtained by the characterisation study and the CCQM-K140 international comparison study are compared, $\delta^{13}C_{VPDB-LSVEC,}$ ‰ mean results are found as -24.02 and -24.09, respectively. Results are found to be in good agreement within the stated uncertainty. The CCQM K-140 Intercomparison study plot is given in Annex 9.

TRACEABILITY

Validated methods were used in homogeneity, stability and characterisation studies. Information about the reference materials used for the correction of the measurement results are given in Table 9.

Table 9. Reference Materials Used in Measurements for Traceability to VPDB-LSVEC scale

Lab No	Reference Material	Material Code	Traceability
1	Sucrose	IAEA-CH-6	IAEA
	L-glutamic acid	USGS 40	RSIL
2	Sucrose	IAEA-CH-6	IAEA
	Cellulose	IAEA-CH-3	IAEA
3	Caffeine	IAEA-600	IAEA
4	Cellulose	IAEA-CH-3	IAEA
5	Sucrose	IAEA-CH-6	IAEA
6	Cellulose	IAEA-CH-3	IAEA
7	Sucrose	IAEA-CH-6	IAEA
	Polyethylene	IAEA-CH-7	IAEA

INSTRUCTIONS FOR USE

Storage conditions

All CRMs should be stored at (+20 ± 5) °C.

Minimum sample intake

Minimum sample intake amount is 0.2 mg, which is the minimum amount used in the homogeneity study.

Safety precautions

Normal laboratory precautions are applied. Use and dispose of materials according to existing local rules is strongly recommended. The use of powder-free gloves for the handling of the materials is recommended. It is recommended to read the Safety Data Sheet before using the UME CRM 1309, UME CRM 1310 and UME CRM 1311.

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REVISION HISTORY

Date	Remarks
25.08.2016	First issue.
21.09.2016	The uncertainty values are revised and the certificates are updated.
21.09.2026	VPDB scale is revised as VPDB-LSVEC.

Annex 1. Homogeneity Data for UME CRM 1309-1313

Table A1. Homogeneity Data for UME CRM 1309, Sucrose

Unit Numbers				Analytical Sequence			Measurement Results, $\delta^{13}C_{VPDB-LSVEC}$ (‰)		
No	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**
1	8	8	8	11	6	16	-26,81	-26,86	-26,84
2	31	31	31	1	21	26	-26,74	-26,53	-26,86
3	49	49	49	17	27	22	-26,64	-26,75	-26,72
4	72	72	72	12	2	7	-26,79	-26,50	-26,85
5	89	89	89	8	18	28	-26,88	-26,81	-26,87
6	107	107	107	23	3	13	-26,91	-26,73	-26,69
7	129	129	129	29	9	4	-26,79	-26,81	-26,61
8	147	147	147	14	24	19	-26,65	-26,76	-26,67
9	167	167	167	10	5	25	-26,81	-26,84	-27,04
10	193	193	193	30	15	20	-26,75	-26,47	-26,62

Table A2. Homogeneity Data for UME CRM 1310, Glucose

Unit Numbers				Analytical Sequence			Measurement Results, $\delta^{13}C_{VPDB-LSVEC}$ (‰)		
No	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**
1	6	6	6	2	8	17	-11.13	-11.11	-10.97
2	28	28	28	21	25	6	-11.00	-11.03	-11.00
3	49	49	49	7	13	24	-10.98	-11.07	-11.00
4	69	69	69	26	4	11	-10.94	-11.11	-11.10
5	92	92	92	14	20	30	-11.16	-11.02	-10.94
6	110	110	110	1	10	15	-11.06	-11.07	-11.09
7	130	130	130	23	29	3	-10.99	-10.80	-11.06
8	158	158	158	9	16	22	-11.01	-11.21	-11.06
9	173	173	173	27	5	12	-11.31	-10.93	-11.18
10	191	191	191	18	19	22	-11.09	-11.11	-11.02

Table A3. Homogeneity Data for UME CRM 1311, Fructose

Unit Numbers				Analytical Sequence			Measurement Results, $\delta^{13}C_{VPDB-LSVEC}$ (‰)		
No	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**
1	3	3	3	5	8	13	-10.65	-10.81	-10.75
2	24	24	24	23	29	6	-10.65	-10.79	-10.79
3	43	43	43	12	16	21	-10.72	-10.72	-10.69
4	65	65	65	27	3	9	-10.77	-10.68	-10.76
5	86	86	86	15	22	26	-10.86	-10.72	-10.74
6	104	104	104	1	10	14	-10.67	-10.77	-10.77
7	126	126	126	19	25	4	-10.85	-10.65	-10.65
8	145	145	145	11	18	24	-10.81	-10.76	-10.84
9	165	165	165	28	2	7	-10.65	-10.54	-10.75
10	185	185	185	17	20	30	-10.74	-10.78	-10.73

Table A4. Homogeneity Data for UME CRM 1312, Honey (Unadulterated)

Unit Numbers				Analytical Sequence			Measurement Results. $\delta^{13}C_{VPDB-LSVEC}$ (‰)		
NO	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**
1	10	10	10	4	12	15	-24.12	-24.04	-24.20
2	29	29	29	20	28	3	-24.20	-24.27	-24.22
3	46	46	46	9	17	23	-24.14	-24.08	-24.18
4	65	65	65	27	5	7	-24.17	-24.15	-24.13
5	88	88	88	13	24	26	-24.24	-24.23	-24.16
6	109	109	109	6	10	18	-24.25	-24.22	-24.13
7	128	128	128	21	25	1	-24.08	-24.20	-24.06
8	148	148	148	8	16	19	-24.19	-24.28	-24.18
9	169	169	169	30	2	11	-24.25	-24.18	-24.25
10	189	189	189	14	22	29	-24.03	-24.22	-24.22

Table A5. Homogeneity Data for UME CRM 1313, Honey (Adulterated)

Unit Numbers				Analytical Sequence			Measurement Results, $\delta^{13}C_{VPDB-LSVEC}$ (‰)		
No	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**	**Rep. 1**	**Rep. 2**	**Rep. 3**
1	7	7	7	1	12	18	-11.68	-11.54	-11.65
2	28	28	28	21	26	6	-11.55	-11.70	-11.64
3	45	45	45	10	14	24	-11.53	-11.56	-11.55
4	64	64	64	27	2	7	-11.58	-11.68	-11.44
5	85	85	85	13	22	25	-11.64	-11.51	-11.64
6	108	108	108	4	11	17	-11.49	-11.53	-11.55
7	123	123	123	20	30	5	-11.61	-11.72	-11.64
8	151	151	151	9	15	19	-11.50	-11.52	-11.57
9	166	166	166	28	8	3	-11.59	-11.49	-11.50
10	187	187	187	16	23	29	-11.64	-11.63	-11.52

Annex 2. Plots for Homogeneity Studies

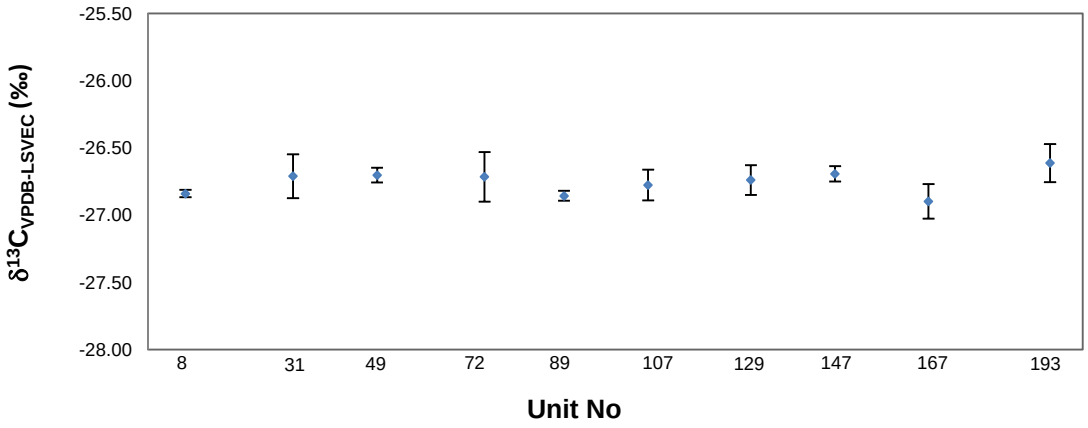


Figure 1. UME CRM 1309 Sucrose, Homogeneity plot

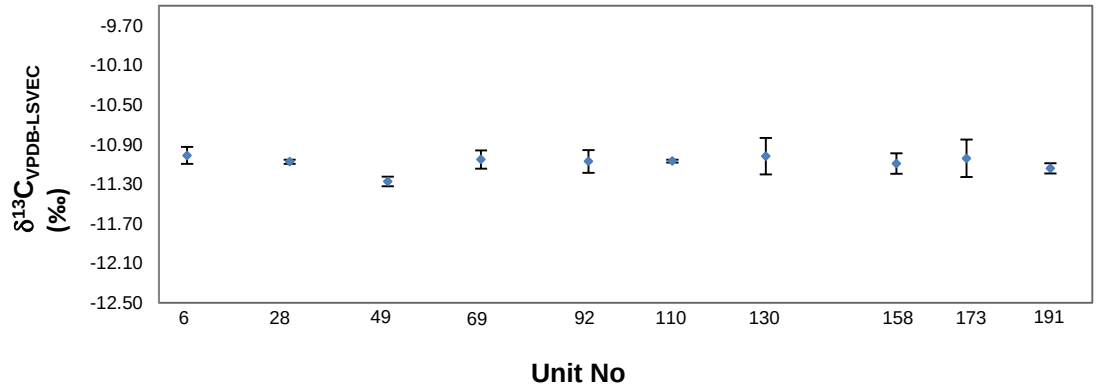


Figure 2. UME CRM 1310 Glucose, Homogeneity plot

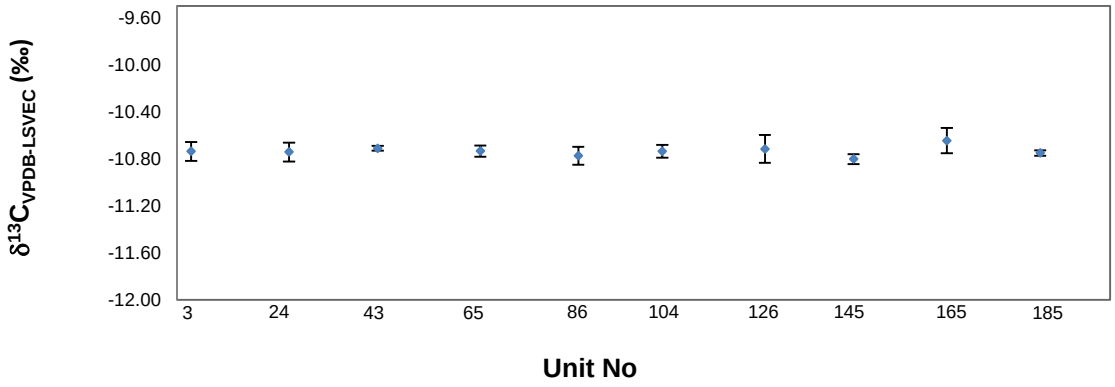


Figure 3. UME CRM 1311 Fructose, Homogeneity plot

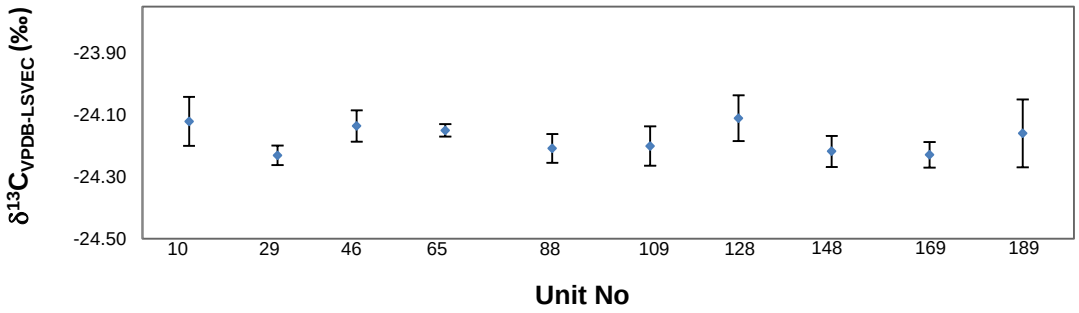


Figure 4. UME CRM 1312 Honey (Unadultrated), Homogeneity plot

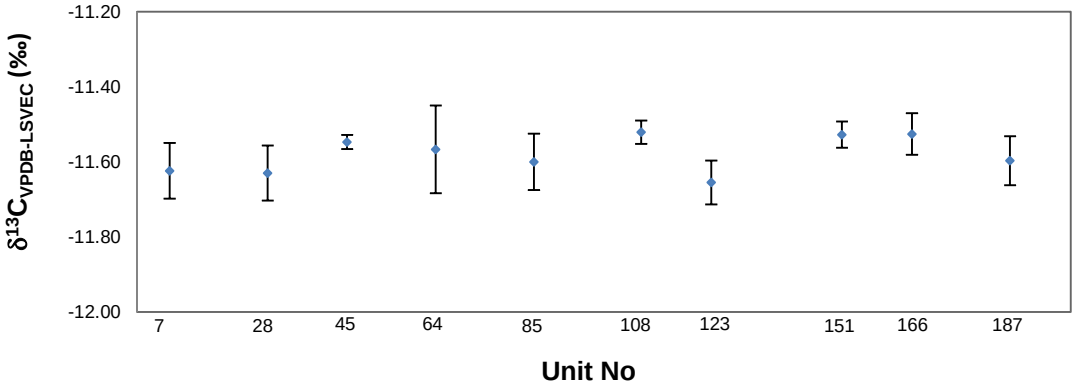


Figure 5. UME CRM 1313 Honey (Adultrated), Homogeneity plot

Annex 3. Short Term Stability (STS) Data for UME CRM 1309-1313

Table A6. STS Data for UME CRM 1309, Sucrose at 4 °C and 60 °C

Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰)	Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰)
52	34-1	0	18	-26.15	52	34-1	0	18	-26.15
54	34-2	0	18	-26.36	54	34-2	0	18	-26.36
51	34-3	0	18	-26.28	51	34-3	0	18	-26.28
49	109-1	0	18	-26.22	49	109-1	0	18	-26.22
50	109-2	0	18	-26.31	50	109-2	0	18	-26.31
53	109-3	0	18	-26.37	53	109-3	0	18	-26.37
4	1-1	1	4	-26.73	28	17-1	1	60	-26.56
11	1-2	1	4	-26.62	35	17-2	1	60	-26.37
17	1-3	1	4	-26.74	41	17-3	1	60	-26.43
9	188-1	1	4	-26.69	33	148-1	1	60	-26.41
16	188-2	1	4	-26.69	40	148-2	1	60	-26.44
23	188-3	1	4	-26.56	47	148-3	1	60	-26.53
12	97-1	2	4	-26.64	36	40-1	2	60	-26.34
13	97-2	2	4	-26.72	37	40-2	2	60	-25.78
20	97-3	2	4	-26.61	44	40-3	2	60	-26.54
3	151-1	2	4	-26.55	27	90-1	2	60	-26.60
7	151-2	2	4	-26.66	31	90-2	2	60	-26.60
14	151-3	2	4	-26.69	38	90-3	2	60	-26.47
8	113-1	3	4	-26.66	32	57-1	3	60	-26.66
1	113-2	3	4	-26.57	25	57-2	3	60	-26.56
10	113-3	3	4	-26.67	34	57-3	3	60	-26.42
18	127-1	3	4	-26.67	42	80-1	3	60	-26.64
22	127-2	3	4	-26.55	46	80-2	3	60	-26.48
19	127-3	3	4	-26.62	43	80-3	3	60	-26.51
21	46-1	4	4	-26.59	45	25-1	4	60	-26.71
15	46-2	4	4	-26.66	39	25-2	4	60	-26.48
2	46-3	4	4	-26.66	26	25-3	4	60	-26.37
24	173-1	4	4	-24.99	48	142-1	4	60	-26.29
6	173-2	4	4	-26.59	30	142-2	4	60	-26.90
5	173-3	4	4	-26.58	29	142-3	4	60	-26.49

Table A7. STS Data for UME CRM 1310, Glucose at 4 and 60 °C

Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰)	Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰)
51	52-1	0	18	-10.47	51	52-1	0	18	-10.47
49	52-2	0	18	-10.53	49	52-2	0	18	-10.53
53	52-3	0	18	-10.51	53	52-3	0	18	-10.51
54	150-1	0	18	-10.52	54	150-1	0	18	-10.52
50	150-2	0	18	-10.71	50	150-2	0	18	-10.71
52	150-3	0	18	-10.64	52	150-3	0	18	-10.64
4	3-1	1	4	-10.68	28	8-1	1	60	-10.50
11	3-2	1	4	-10.56	35	8-2	1	60	-10.52
17	3-3	1	4	-10.66	41	8-3	1	60	-10.37
9	175-1	1	4	-10.43	33	186-1	1	60	-10.54
16	175-2	1	4	-10.58	40	186-2	1	60	-10.52
23	175-3	1	4	-10.41	47	186-3	1	60	-10.33
21	25-1	2	4	-10.48	45	37-1	2	60	-10.43
15	25-2	2	4	-10.58	39	37-2	2	60	-10.40
2	25-3	2	4	-10.55	26	37-3	2	60	-10.39
24	153-1	2	4	-10.43	48	159-1	2	60	-10.56
6	153-2	2	4	-10.61	30	159-2	2	60	-10.42
5	153-3	2	4	-10.45	29	159-3	2	60	-10.51
12	54-1	3	4	-10.45	36	60-1	3	60	-10.56
13	54-2	3	4	-10.46	37	60-2	3	60	-10.53
20	54-3	3	4	-10.27	44	60-3	3	60	-10.31
3	125-1	3	4	-10.59	27	137-1	3	60	-10.39
7	125-2	3	4	-10.51	31	137-2	3	60	-10.42
14	125-3	3	4	-10.33	38	137-3	3	60	-10.46
8	79-1	4	4	-10.56	32	87-1	4	60	-10.58
1	79-2	4	4	-10.57	25	87-2	4	60	-10.42
10	79-3	4	4	-10.49	34	87-3	4	60	-10.45
18	105-1	4	4	-10.50	42	107-1	4	60	-10.63
22	105-2	4	4	-10.61	46	107-2	4	60	-10.54
19	105-3	4	4	-10.76	43	107-3	4	60	-10.59

Table A8. STS Data for UME CRM 1311, Fructose at 4 °C and 60 °C

Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB-LSVEC}$ (‰)	Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB-LSVEC}$ (‰)
50	34-1	0	18	-10.71	50	34-1	0	18	-10.71
52	34-2	0	18	-10.60	52	34-2	0	18	-10.60
54	34-3	0	18	-10.57	54	34-3	0	18	-10.57
49	163-1	0	18	-10.82	49	163-1	0	18	-10.69
51	163-2	0	18	-10.73	51	163-2	0	18	-10.76
53	163-3	0	18	-11.26	53	163-3	0	18	-10.73
4	6-1	1	4	-10.94	4	2-1	1	60	-10.62
11	6-2	1	4	-10.75	11	2-2	1	60	-10.68
17	6-3	1	4	-10.67	17	2-3	1	60	-10.57
9	181-1	1	4	-10.81	9	180-1	1	60	-10.72
16	181-2	1	4	-10.63	16	180-2	1	60	-10.69
23	181-3	1	4	-10.74	23	180-3	1	60	-10.68
21	36-1	2	4	-10.95	21	26-1	2	60	-10.47
15	36-2	2	4	-10.72	15	26-2	2	60	-10.99
2	36-3	2	4	-11.02	2	26-3	2	60	-10.51
24	158-1	2	4	-10.68	24	150-1	2	60	-10.77
6	158-2	2	4	-10.84	6	150-2	2	60	-10.55
5	158-3	2	4	-10.78	5	150-3	2	60	-10.82
12	61-1	3	4	-10.63	12	50-1	3	60	-11.04
13	61-2	3	4	-10.99	13	50-2	3	60	-10.88
20	61-3	3	4	-10.36	20	50-3	3	60	-10.52
3	131-1	3	4	-10.70	3	128-1	3	60	-10.65
7	131-2	3	4	-10.74	7	128-2	3	60	-10.77
14	131-3	3	4	-10.67	14	128-3	3	60	-10.52
8	84-1	4	4	-10.75	8	76-1	4	60	-10.73
1	84-2	4	4	-10.85	1	76-2	4	60	-10.63
10	84-3	4	4	-10.61	10	76-3	4	60	-12.24
18	106-1	4	4	-10.67	18	101-1	4	60	-10.61
22	106-2	4	4	-10.46	22	101-2	4	60	-10.63
19	106-3	4	4	-10.81	19	101-3	4	60	-10.44

Table A9. STS Data for UME CRM 1312, Honey (unadulterated) in 4 °C and 60 °C

Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB-LSVEC}$ (‰)	Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB-LSVEC}$ (‰)
49	41-1	0	18	-24.06	49	41-1	0	18	-24.06
52	41-2	0	18	-23.87	52	41-2	0	18	-23.87
54	41-3	0	18	-23.87	54	41-3	0	18	-23.87
50	112-1	0	18	-23.78	50	112-1	0	18	-23.78
53	112-2	0	18	-23.92	53	112-2	0	18	-23.92
51	112-3	0	18	-23.98	51	112-3	0	18	-23.98
4	5-1	1	4	-24.05	4	8-1	1	60	-23.93
11	5-2	1	4	-23.96	11	8-2	1	60	-23.89
17	5-3	1	4	-23.87	17	8-3	1	60	-23.85
9	178-1	1	4	-24.21	9	182-1	1	60	-23.87
16	178-2	1	4	-23.85	16	182-2	1	60	-23.80
23	178-3	1	4	-23.94	19	182-3	1	60	-23.92
21	28-1	2	4	-23.88	21	32-1	2	60	-23.86
15	28-2	2	4	-23.84	15	32-2	2	60	-23.89
2	28-3	2	4	-24.09	2	32-3	2	60	-23.97
24	152-1	2	4	-23.78	24	156-1	2	60	-23.89
6	152-2	2	4	-23.94	6	156-2	2	60	-23.82
5	152-3	2	4	-23.97	5	156-3	2	60	-24.02
12	55-1	3	4	-23.98	12	59-1	3	60	-23.92
13	55-2	3	4	-23.87	13	59-2	3	60	-23.81
20	55-3	3	4	-23.94	20	59-3	3	60	-23.78
3	125-1	3	4	-23.89	3	137-1	3	60	-23.92
7	125-2	3	4	-23.96	7	137-2	3	60	-23.87
14	125-3	3	4	-23.84	14	137-3	3	60	-23.82
8	79-1	4	4	-24.03	8	82-1	4	60	-23.85
1	79-2	4	4	-23.80	1	82-2	4	60	-23.85
10	79-3	4	4	-23.85	10	82-3	4	60	-23.85
18	103-1	4	4	-23.80	18	108-1	4	60	-23.89
22	103-2	4	4	-23.98	22	108-2	4	60	-23.75
19	103-3	4	4	-23.92	19	108-3	4	60	-23.88

Table A10. STS Data for UME CRM 1313, Honey (adulterated) in 4 °C and 60 °C

Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB-LSVEC}$ (‰)	Injection No	Unit No	Time (week)	Temp. (°C)	$\delta^{13}C_{VPDB-LSVEC}$ (‰)
50	6-1	0	18	-11.49	50	6-1	0	18	-11.49
54	6-2	0	18	-11.59	54	6-2	0	18	-11.59
52	6-3	0	18	-11.61	52	6-3	0	18	-11.61
49	149-1	0	18	-11.63	49	149-1	0	18	-11.63
53	149-2	0	18	-11.54	53	149-2	0	18	-11.54
51	149-3	0	18	-11.60	51	149-3	0	18	-11.60
4	10-1	1	4	-11.70	4	4-1	1	60	-11.50
11	10-2	1	4	-11.51	11	4-2	1	60	-11.60
17	10-3	1	4	-11.72	17	4-3	1	60	-11.57
9	183-1	1	4	-11.48	9	177-1	1	60	-11.55
16	183-2	1	4	-11.65	16	177-2	1	60	-11.59
23	183-3	1	4	-11.56	23	177-3	1	60	-11.56
21	36-1	2	4	-11.55	21	26-1	2	60	-11.66
15	36-2	2	4	-11.41	15	26-2	2	60	-11.68
2	36-3	2	4	-11.48	2	26-3	2	60	-11.59
24	160-1	2	4	-11.71	24	154-1	2	60	-11.51
6	160-2	2	4	-11.71	6	154-2	2	60	-11.51
5	160-3	2	4	-11.46	5	154-3	2	60	-11.54
12	60-1	3	4	-11.63	12	54-1	3	60	-11.51
13	60-2	3	4	-11.47	13	54-2	3	60	-11.54
20	60-3	3	4	-11.51	20	54-3	3	60	-11.37
3	137-1	3	4	-11.57	3	125-1	3	60	-11.41
7	137-2	3	4	-11.47	7	125-2	3	60	-11.53
14	137-3	3	4	-11.56	14	125-3	3	60	-11.54
8	81-1	4	4	-11.46	8	76-1	4	60	-11.51
1	81-2	4	4	-11.43	1	76-2	4	60	-11.57
10	81-3	4	4	-11.56	10	76-3	4	60	-11.57
18	107-1	4	4	-11.48	18	102-1	4	60	-11.59
22	107-2	4	4	-11.48	22	102-2	4	60	-11.43
19	107-3	4	4	-11.63	19	102-3	4	60	-11.69

Annex 4. Short Term Stability (STS) Plots for UME CRM 1309-1313 (4 ºC and 60 ºC)

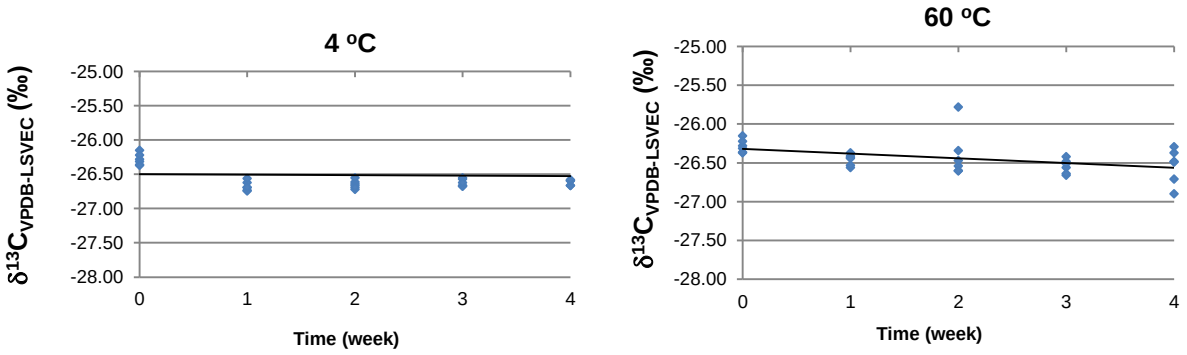


Figure 6. UME CRM 1309 Sucrose Short Term Stability Plots

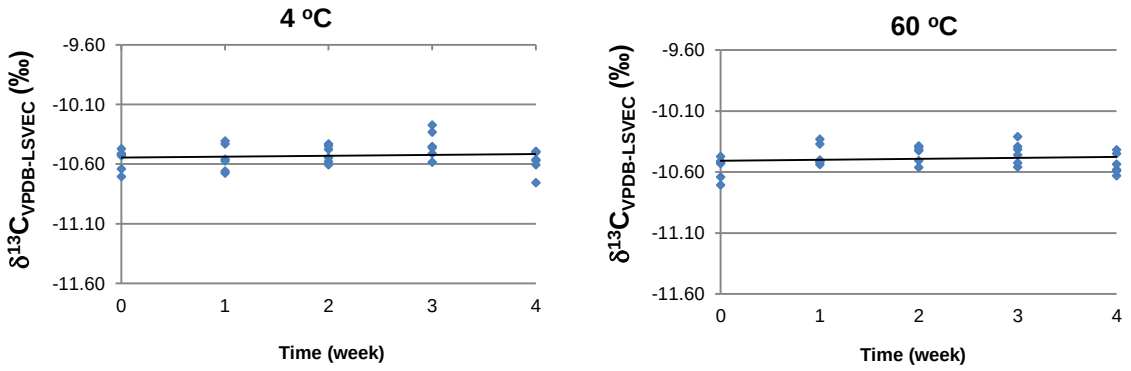


Figure 7. UME CRM 1310 Glucose Short Term Stability Plots

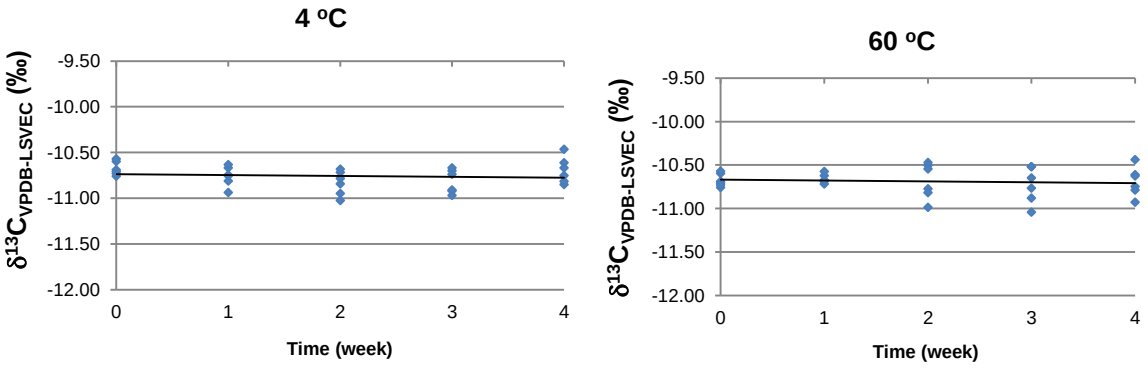


Figure 8. UME CRM 1311 Fructose Short Term Stability Plots

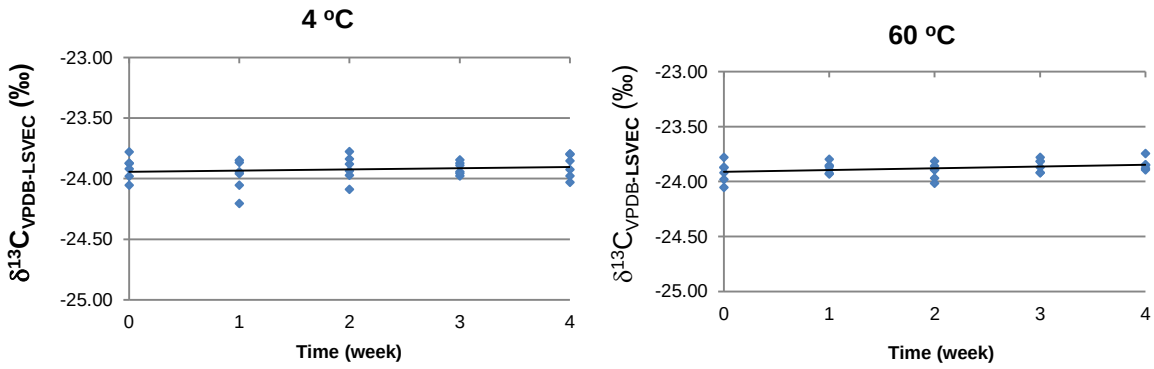


Figure 9. UME CRM 1312 Honey (Unadulterated) Short Term Stability Plots

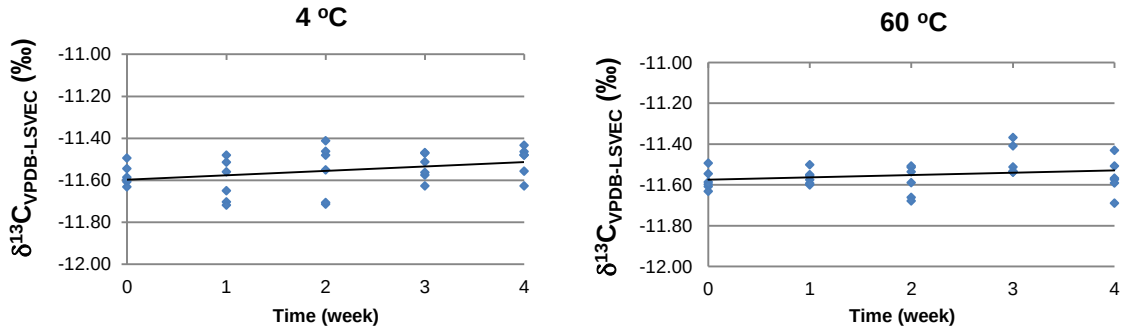


Figure 10. UME CRM 1313 Honey (Adulterated) Short Term Stability Plots

Annex 5. Long Term Stability (LTS) Data for UME CRM 1309-1313

Table A11. LTS Data for UME CRM 1309-1310

UME CRM 1309 Sucrose					UME CRM 1310 Glucose				
Injection No	Unit No	Time (month)	Temperature	$\delta^{13}C_{VPDB-LSVEC}$ (‰)	Injection No	Unit No	Time (month)	Temperature(°C)	$\delta^{13}C_{VPDB-LSVEC}$
82	34-1	0	18	-26.84	76	52-1	0	18	-10.94
84	34-2	0	18	-26.91	78	52-2	0	18	-11.24
86	34-3	0	18	-26.86	74	52-3	0	18	-10.96
88	109-1	0	18	-26.91	75	150-1	0	18	-11.03
83	109-2	0	18	-26.94	73	150-2	0	18	-11.02
85	109-3	0	18	-26.90	77	150-3	0	18	-11.06
1	3-1	1	25	-26.85	1	1-1	1	25	-11.02
25	3-2	1	25	-26.87	25	1-2	1	25	-11.06
49	3-3	1	25	-26.88	49	1-3	1	25	-11.02
37	195-1	1	25	-26.87	37	195-1	1	25	-10.98
13	195-2	1	25	-26.86	13	195-2	1	25	-11.02
61	195-3	1	25	-26.85	61	195-3	1	25	-11.02
2	20-1	2	25	-26.79	2	11-1	2	25	-11.00
26	20-2	2	25	-26.86	26	11-2	2	25	-11.18
50	20-3	2	25	-26.87	50	11-3	2	25	-11.01
38	178-1	2	25	-26.82	38	185-1	2	25	-11.07
14	178-2	2	25	-26.92	14	185-2	2	25	-10.99
62	178-3	2	25	-26.84	62	185-3	2	25	-10.98
3	25-1	3	25	-26.69	3	18-1	3	25	-11.02
27	35-2	3	25	-26.65	27	18-2	3	25	-11.00
51	35-3	3	25	-26.94	51	18-3	3	25	-11.03
39	158-1	3	25	-26.75	39	177-1	3	25	-11.01
15	158-2	3	25	-26.87	15	177-2	3	25	-11.07
63	158-3	3	25	-26.77	63	177-3	3	25	-11.07
4	51-1	4	25	-26.86	4	27-1	4	25	-11.02
28	51-2	4	25	-26.88	28	27-2	4	25	-10.99
52	51-3	4	25	-26.94	52	27-3	4	25	-10.97
40	145-1	4	25	-26.87	40	170-1	4	25	-11.04
16	145-2	4	25	-26.85	16	170-2	4	25	-11.03
64	145-3	4	25	-26.85	64	170-3	4	25	-11.07
5	68-1	5	25	-26.80	5	33-1	5	25	-10.98
29	68-2	5	25	-26.89	29	33-2	5	25	-11.00
53	68-3	5	25	-26.90	53	33-3	5	25	-11.01
41	125-1	5	25	-26.84	41	160-1	5	25	-11.01
17	125-2	5	25	-26.92	17	160-2	5	25	-11.00
65	125-3	5	25	-26.86	65	160-3	5	25	-10.96
6	85-1	6	25	-26.83	6	44-1	6	25	-10.93
30	85-2	6	25	-26.97	30	44-2	6	25	-10.98
54	85-3	6	25	-26.81	54	44-3	6	25	-10.97
42	108-1	6	25	-26.95	42	152-1	6	25	-10.97
18	108-2	6	25	-26.91	18	152-2	6	25	-10.92
66	108-3	6	25	-26.62	66	152-3	6	25	-10.97
7	92-1	7	25	-26.78	7	51-1	7	25	-10.97
31	92-2	7	25	-26.86	31	51-2	7	25	-11.02
55	92-3	7	25	-26.88	55	51-3	7	25	-11.01
43	102-1	7	25	-26.81	43	145-1	7	25	-11.01
19	102-2	7	25	-26.88	19	145-2	7	25	-11.00
67	102-3	7	25	-26.83	67	145-3	7	25	-10.99
8	76-1	8	25	-26.87	8	59-1	8	25	-11.05
32	76-2	8	25	-26.95	32	59-2	8	25	-11.02
56	76-3	8	25	-26.85	56	59-3	8	25	-11.03
44	120-1	8	25	-26.84	44	136-1	8	25	-11.00
20	120-2	8	25	-26.90	20	136-2	8	25	-10.96
68	120-3	8	25	-26.87	68	136-3	8	25	-11.01
9	60-1	9	25	-26.94	9	68-1	9	25	-11.04
33	60-2	9	25	-26.82	33	68-2	9	25	-10.99
57	60-3	9	25	-26.85	57	68-3	9	25	-10.97
45	134-1	9	25	-26.86	45	128-1	9	25	-10.95
21	134-2	9	25	-26.85	21	128-2	9	25	-10.95
69	134-3	9	25	-26.86	69	128-3	9	25	-10.84
10	43-1	10	25	-26.83	10	76-1	10	25	-10.98
34	43-2	10	25	-26.87	34	76-2	10	25	-11.01
58	43-3	10	25	-26.94	58	76-3	10	25	-11.02
46	152-1	10	25	-26.92	46	119-1	10	25	-10.96
22	152-2	10	25	-26.80	22	119-2	10	25	-10.98
70	152-3	10	25	-26.82	70	119-3	10	25	-11.00
11	26-1	11	25	-26.84	11	83-1	11	25	-11.03
35	26-2	11	25	-26.88	35	83-2	11	25	-10.99
59	26-3	11	25	-26.89	59	83-3	11	25	-10.98
47	170-1	11	25	-26.89	47	111-1	11	25	-11.16
23	170-2	11	25	-26.84	23	111-2	11	25	-10.96
71	170-3	11	25	-26.85	71	111-3	11	25	-11.06
12	11-1	12	25	-26.83	12	93-1	12	25	-10.95
36	11-2	12	25	-26.83	36	93-2	12	25	-11.01
60	11-3	12	25	-26.88	60	93-3	12	25	-10.98
48	187-1	12	25	-26.92	48	102-1	12	25	-11.00
24	187-2	12	25	-26.87	24	102-2	12	25	-10.95
72	187-3	12	25	-26.81	76	52-1	0	18	-10.94

Table A12. LTS Data for UME CRM 1311-1312

UME CRM 1311 Fructose					UME CRM 1312 Honey (Unadulterated)				
Injection No	Unit No	Time (month)	Temperature(°C)	$\delta^{13}C_{VPDB-LSVEC}$	Injection No	Unit No	Time (month)	Temperature	$\delta^{13}C_{VPDB-LSVEC}$
73	34-1	0	18	-10.86	73	41-1	0	18	-24.17
78	34-2	0	18	-10.70	76	41-2	0	18	-24.14
75	34-3	0	18	-10.65	78	41-3	0	18	-24.09
76	163-1	0	18	-10.77	75	112-1	0	18	-24.16
74	163-2	0	18	-10.79	77	112-2	0	18	-24.10
77	163-3	0	18	-10.79	74	112-3	0	18	-24.16
1	1-1	1	25	-10.76	1	3-1	1	25	-23.95
25	1-2	1	25	-10.90	25	3-2	1	25	-24.14
49	1-3	1	25	-10.84	49	3-3	1	25	-24.00
37	192-1	1	25	-10.69	13	192-1	1	25	-24.15
13	192-2	1	25	-10.86	37	192-2	1	25	-24.30
61	192-3	1	25	-10.85	61	192-3	1	25	-24.10
2	9-1	2	25	-10.93	26	9-1	2	25	-24.09
26	9-2	2	25	-10.94	2	9-2	2	25	-24.02
50	9-3	2	25	-10.86	50	9-3	2	25	-24.06
38	187-1	2	25	-10.85	38	183-1	2	25	-24.14
14	187-2	2	25	-10.87	14	183-2	2	25	-24.12
62	187-3	2	25	-10.72	62	183-3	2	25	-24.17
3	17-1	3	25	-10.89	3	17-1	3	25	-24.00
27	17-2	3	25	-10.87	27	17-2	3	25	-24.09
51	17-3	3	25	-10.90	51	17-3	3	25	-24.05
39	178-1	3	25	-10.78	15	175-1	3	25	-24.10
15	178-2	3	25	-10.84	39	175-2	3	25	-24.11
63	178-3	3	25	-10.82	63	175-3	3	25	-24.09
4	28-1	4	25	-10.71	28	26-1	4	25	-24.03
28	28-2	4	25	-10.94	4	26-2	4	25	-23.87
52	28-3	4	25	-10.85	52	26-3	4	25	-23.91
40	168-1	4	25	-10.84	40	167-1	4	25	-24.12
16	168-2	4	25	-10.86	16	167-2	4	25	-24.09
64	168-3	4	25	-10.86	64	167-3	4	25	-24.13
5	35-1	5	25	-10.78	5	37-1	5	25	-23.93
29	35-2	5	25	-10.78	29	37-2	5	25	-24.19
53	35-3	5	25	-10.81	53	37-3	5	25	-24.05
41	159-1	5	25	-10.93	17	161-1	5	25	-24.09
17	159-2	5	25	-10.88	41	161-2	5	25	-24.17
65	159-3	5	25	-10.88	65	161-3	5	25	-24.10
6	45-1	6	25	-10.90	30	44-1	6	25	-24.09
30	45-2	6	25	-10.78	6	44-2	6	25	-24.09
54	45-3	6	25	-10.69	54	44-3	6	25	-24.12
42	151-1	6	25	-10.84	42	151-1	6	25	-24.10
18	151-2	6	25	-10.94	18	151-2	6	25	-24.12
66	151-3	6	25	-10.77	66	151-3	6	25	-24.12
7	53-1	7	25	-10.80	7	53-1	7	25	-24.07
31	53-2	7	25	-10.78	31	53-2	7	25	-24.08
55	53-3	7	25	-10.87	55	53-3	7	25	-24.08
43	143-1	7	25	-10.82	19	144-1	7	25	-24.13
19	143-2	7	25	-10.82	43	144-2	7	25	-24.12
67	143-3	7	25	-10.78	67	144-3	7	25	-24.12
8	58-1	8	25	-10.81	32	58-1	8	25	-24.09
32	58-2	8	25	-10.85	8	58-2	8	25	-24.05
56	58-3	8	25	-10.91	56	58-3	8	25	-24.07
44	133-1	8	25	-10.72	44	134-1	8	25	-24.11
20	133-2	8	25	-10.79	20	134-2	8	25	-24.09
68	133-3	8	25	-10.85	68	134-3	8	25	-24.17
9	69-1	9	25	-10.82	9	67-1	9	25	-24.17
33	69-2	9	25	-10.82	33	67-2	9	25	-24.07
57	69-3	9	25	-10.85	57	67-3	9	25	-24.14
45	125-1	9	25	-10.89	21	126-1	9	25	-24.13
21	125-2	9	25	-10.80	45	126-2	9	25	-24.13
69	125-3	9	25	-10.79	69	126-3	9	25	-24.10
10	77-1	10	25	-10.69	34	76-1	10	25	-24.15
34	77-2	10	25	-10.85	10	76-2	10	25	-24.07
58	77-3	10	25	-10.83	58	76-3	10	25	-24.13
46	118-1	10	25	-10.94	46	120-1	10	25	-24.13
22	118-2	10	25	-10.80	22	120-2	10	25	-24.28
70	118-3	10	25	-10.81	70	120-3	10	25	-24.02
11	85-1	11	25	-10.84	11	86-1	11	25	-24.12
35	85-2	11	25	-10.87	35	86-2	11	25	-24.16
59	85-3	11	25	-10.78	59	86-3	11	25	-24.13
47	112-1	11	25	-10.79	23	111-1	11	25	-24.09
23	112-2	11	25	-10.74	47	111-2	11	25	-24.10
71	112-3	11	25	-10.90	71	111-3	11	25	-24.10
12	92-1	12	25	-10.80	36	95-1	12	25	-24.04
36	92-2	12	25	-10.62	12	95-2	12	25	-24.10
60	92-3	12	25	-10.79	60	95-3	12	25	-24.16
48	100-1	12	25	-10.83	48	102-1	12	25	-24.09
24	100-2	12	25	-10.83	24	102-2	12	25	-24.22
72	100-3	12	25	-10.85	72	102-3	12	25	-24.11

Table A13. LTS Data for UME CRM 1313

UME CRM 1313 Honey (Adulterated)				
Injection No	Unit No	Time (month)	Temperature (°C)	$\delta^{13}C_{VPDB\text{-}LSVEC}$ (‰)
73	6-1	0	18	-11.52
77	6-2	0	18	-11.58
75	6-3	0	18	-11.60
76	149-1	0	18	-11.60
74	149-2	0	18	-11.75
78	149-3	0	18	-11.49
1	3-1	1	25	-11.62
25	3-2	1	25	-11.59
49	3-3	1	25	-11.65
13	193-1	1	25	-11.59
37	193-2	1	25	-11.55
61	193-3	1	25	-11.67
50	11-1	2	25	-11.45
2	11-2	2	25	-11.52
26	11-3	2	25	-11.49
62	186-1	2	25	-11.61
14	186-2	2	25	-11.58
38	186-3	2	25	-11.53
27	18-1	3	25	-11.54
51	18-2	3	25	-11.70
3	18-3	3	25	-11.32
39	175-1	3	25	-11.52
63	175-2	3	25	-11.57
15	175-3	3	25	-11.47
4	27-1	4	25	-11.22
28	27-2	4	25	-11.48
52	27-3	4	25	-11.49
16	167-1	4	25	-11.52
40	167-2	4	25	-11.62
64	167-3	4	25	-11.59
53	34-1	5	25	-11.40
5	34-2	5	25	-11.82
29	34-3	5	25	-11.54
65	161-1	5	25	-11.47
17	161-2	5	25	-11.51
41	161-3	5	25	-11.52
30	43-1	6	25	-11.75
54	43-2	6	25	-11.54
6	43-3	6	25	-11.27
42	153-1	6	25	-11.54
66	153-2	6	25	-11.56
18	153-3	6	25	-11.53
7	51-1	7	25	-11.44
31	51-2	7	25	-11.55
55	51-3	7	25	-11.58
19	143-1	7	25	-11.77
43	143-2	7	25	-11.63
67	143-3	7	25	-11.59
56	62-1	8	25	-11.41
8	62-2	8	25	-11.59
32	62-3	8	25	-11.63
68	133-1	8	25	-11.54
20	133-2	8	25	-11.51
44	133-3	8	25	-11.50
33	67-1	9	25	-11.57
57	67-2	9	25	-11.46
9	67-3	9	25	-11.50
45	127-1	9	25	-11.61
69	127-2	9	25	-11.57
21	127-3	9	25	-11.51
10	75-1	10	25	-11.49
34	75-2	10	25	-11.43
58	75-3	10	25	-11.73
22	118-1	10	25	-11.48
46	118-2	10	25	-11.54
70	118-3	10	25	-11.63
59	86-1	11	25	-11.57
11	86-2	11	25	-11.51
35	86-3	11	25	-11.61
71	110-1	11	25	-11.80
23	110-2	11	25	-11.56
47	110-3	11	25	-11.80
36	92-1	12	25	-11.88
60	92-2	12	25	-11.55
12	92-3	12	25	-11.53
48	100-1	12	25	-11.51
72	100-2	12	25	-11.58
24	100-3	12	25	-11.50

Annex 6. Long Term Stability (LTS) Plots for UME CRM 1309-1313

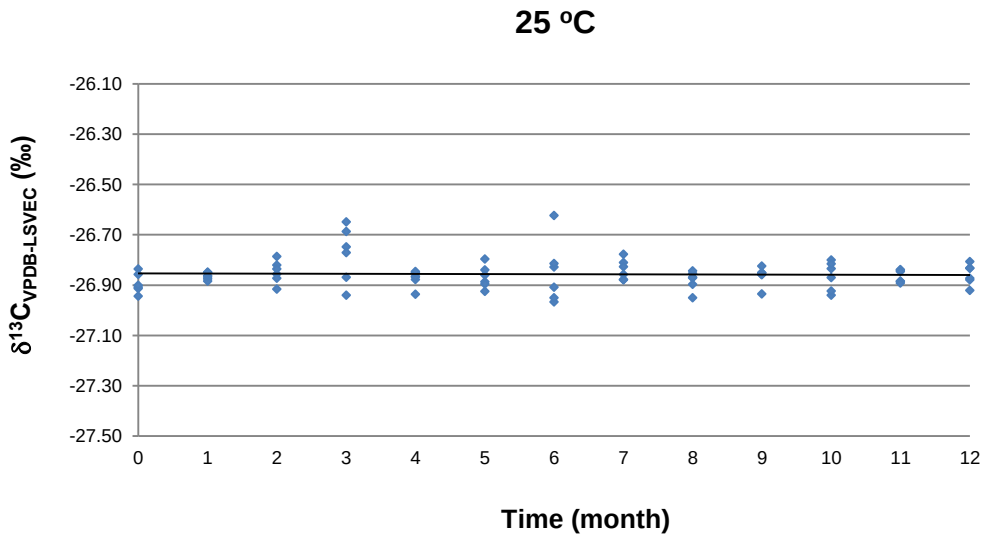


Figure 11. UME CRM 1309 Sucrose Long Term Stability Plot

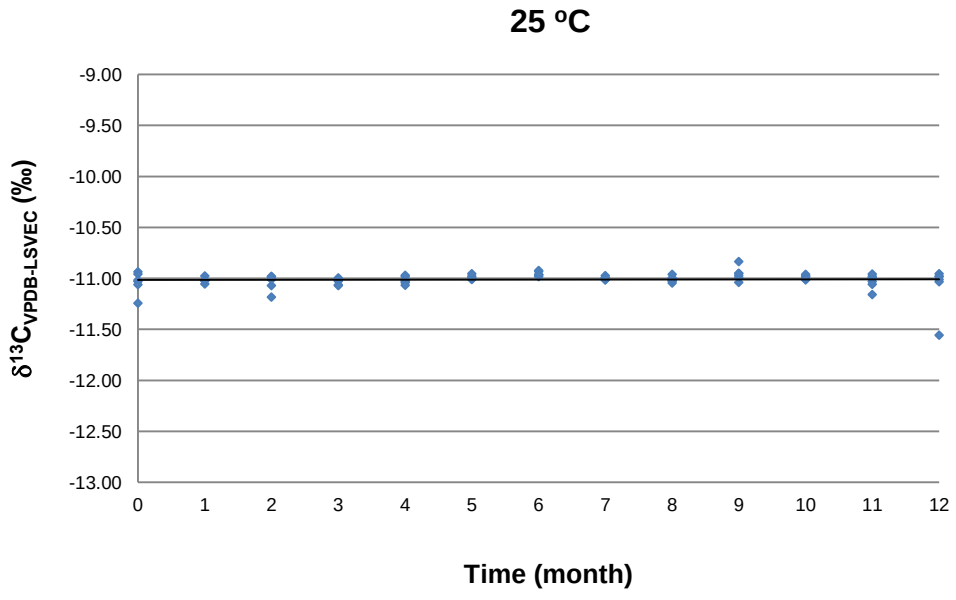


Figure 12. UME CRM 1310 Glucose Long Term Stability Plot

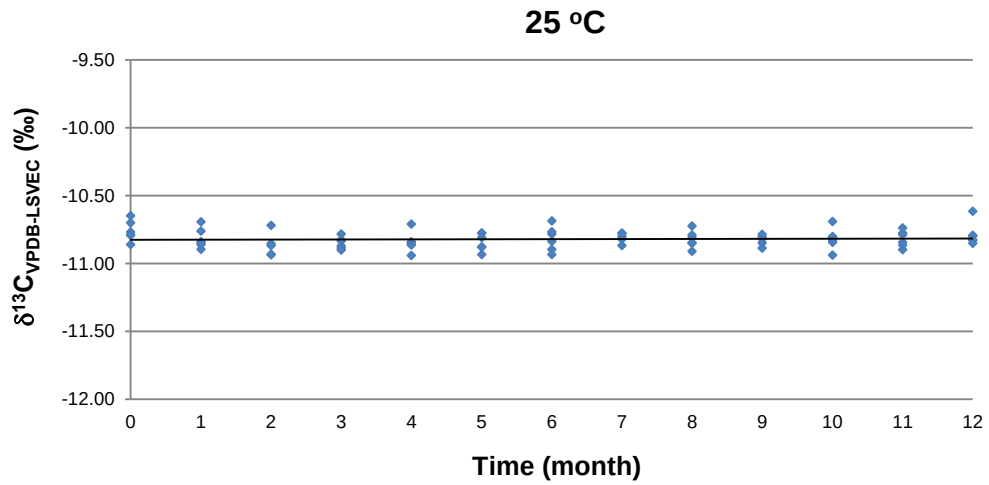


Figure 13. UME CRM 1311 Fructose Long Term Stability Plot

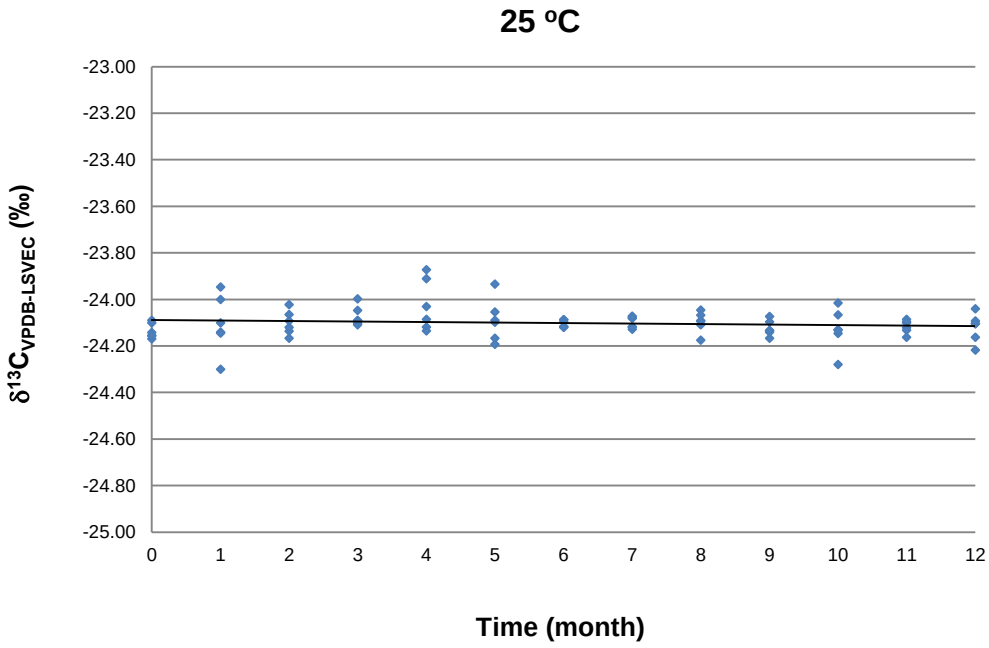


Figure 14. UME CRM 1312 Honey (Unadulterated) Long Term Stability Plot

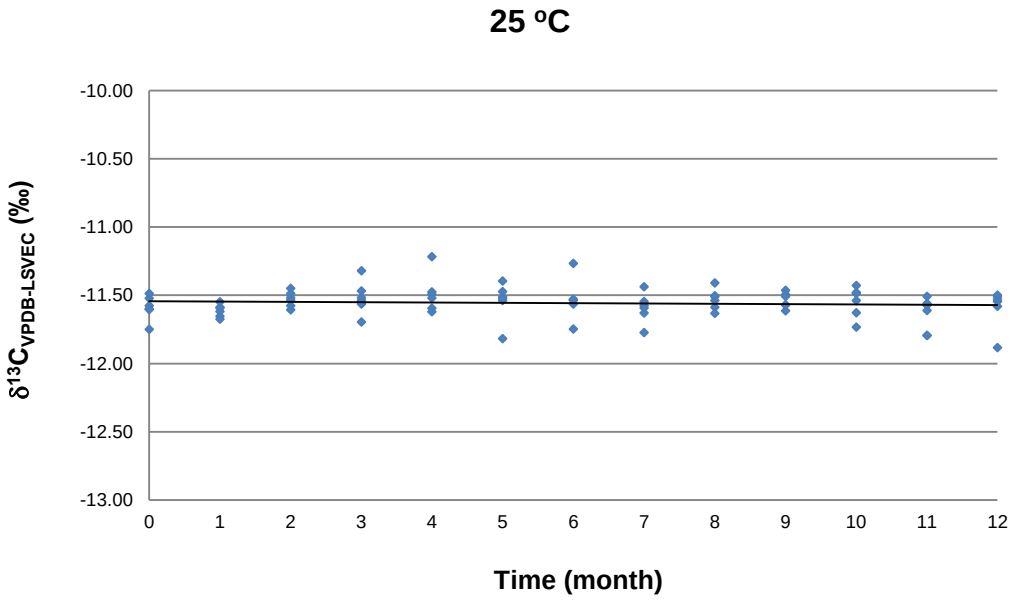


Figure 15. UME CRM 1313 Honey (Adulterated) Long Term Stability Plot

Annex 7. Characterisation Data for UME CRM 1309-1313

Table A14. Characterisation Results for UME CRM 1309-1313

		Mean $\delta^{13}C_{VPDB-LSVEC}$ (‰)				
Lab. No	**Technique**	**UME CRM 1309**	**UME CRM 1310**	**UME CRM 1311**	**UME CRM 1312**	**UME CRM 1313**
1	EA-IRMS	-26.70	-11.98	-11.72	-24.00	-12.37
2	EA-IRMS	-26.84	-11.55	-11.28	-24.12	-12.22
3	EA-IRMS	-26.90	-11.12	-10.80	-24.22	-11.70
4	EA-IRMS	-27.19	-11.18	-11.30	-24.31	-11.67
5	EA-IRMS	-26.64	-11.03	-10.84	-23.83	-11.52
6	EA-IRMS	-26.38	-10.45	-10.18	-23.86	-11.13
7	EA-IRMS	-26.79	-11.03	-10.69	-23,83	-11,53

Annex 8. Characterisation Plots for UME CRM 1309-1313

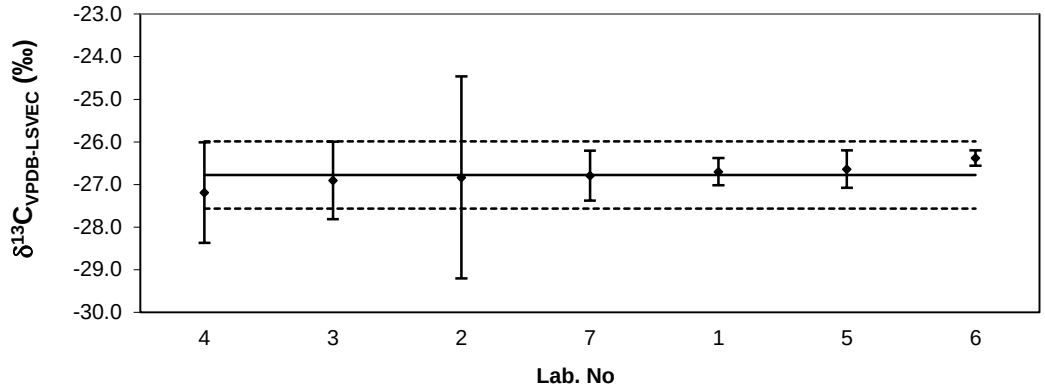


Figure 16. UME CRM 1309 Sucrose Characterisation Plot

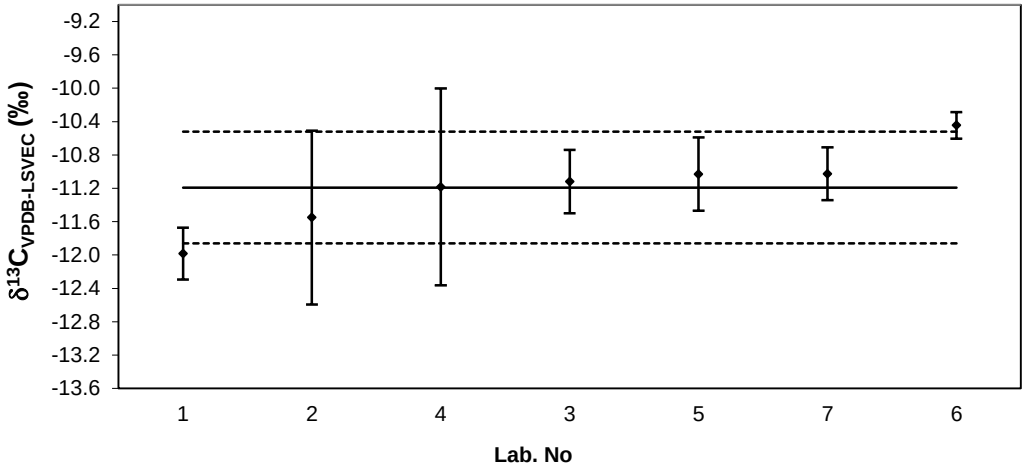


Figure 17. UME CRM 1310 Glucose Characterisation Plot

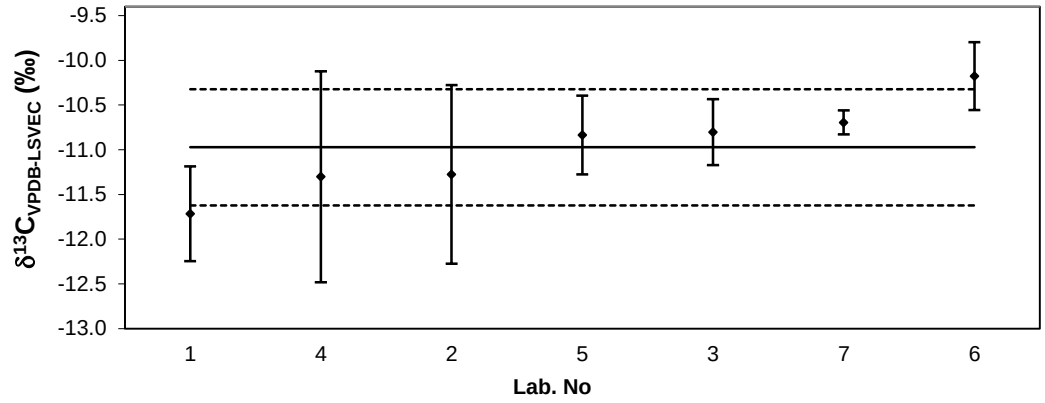


Figure 18. UME CRM 1311 Fructose Characterisation Plot

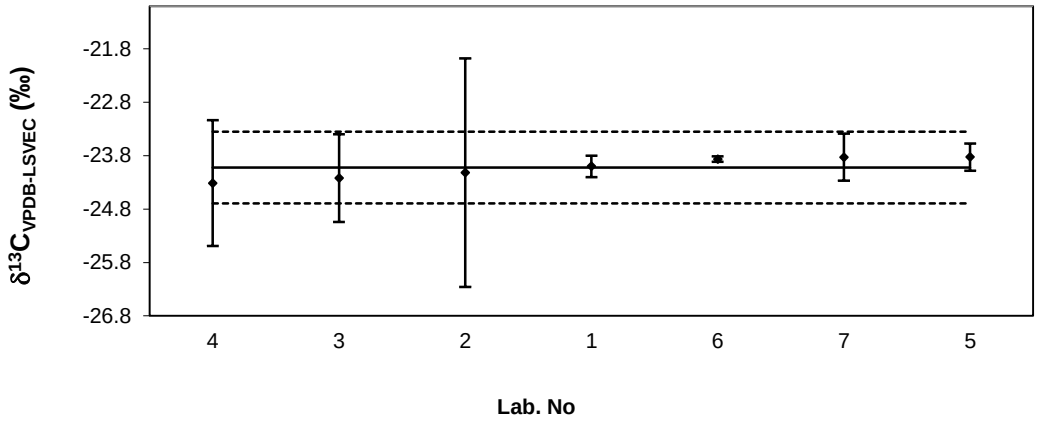


Figure 19. UME CRM 1312 Honey (Unadulterated) Characterisation Plot

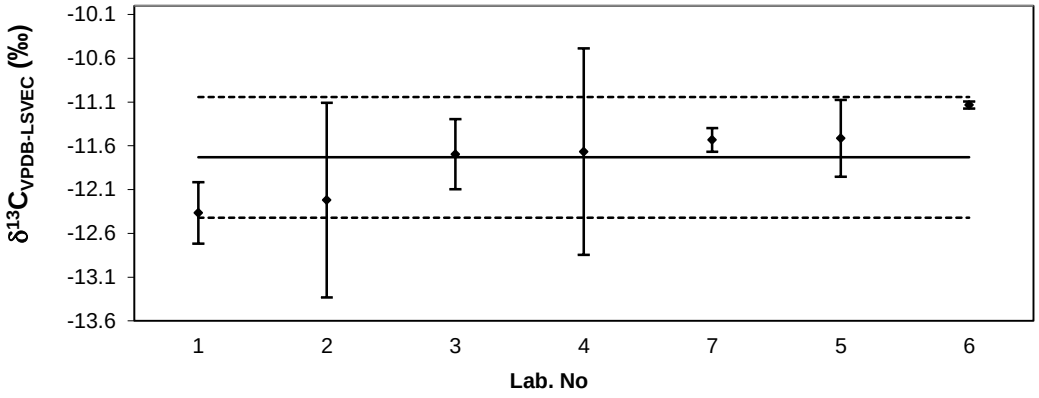


Figure 20. UME CRM 1313 Honey (Adulterated) Characterisation Plot

Annex 9. CCQM K-140 Intercomparison Study Results for UME CRM 1312

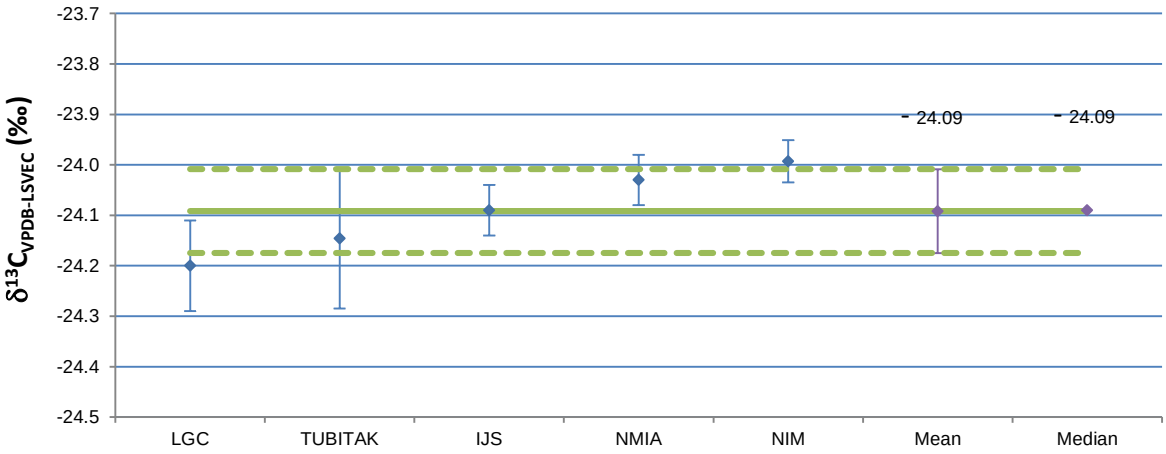


Figure 21. UME CRM 1312 Honey (Unadulterated) Intercomparison Study Plot