CERTIFICATE OF PARTICIPATION

This certificate confirms that:

Chemisches Labor Dr. Mang

took part in:
Food Chemistry Proficiency Test **3304**
Start Date: 24/10/2023
Cannabidiol in Oil

and were allocated laboratory number **12**.

The performance of the laboratory is shown in the relevant report, which is available from the secure pages at fapas.com

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Fapas® – Food Chemistry Proficiency Test Report 3304

Cannabidiol in Oil

October-December 2023

PARTICIPANT LABORATORY NUMBER

Participants can log in to Fapas® SecureWeb at any time to obtain their laboratory number for this proficiency test.

Laboratory numbers are displayed in SecureWeb next to the download link for this report.

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QUALITY SYSTEMS

This proficiency test has been conducted according to the principles of ISO/IEC 17043:2010 [3] but is not within the scope of Fapas® accreditation.

Fera is an ISO 9001 certified organisation.

SUMMARY

1. The test materials for Fapas® – Food Chemistry proficiency test 3304 were dispatched in October 2023. Each participant received an oil test material to be analysed for cannabidiol, with the option to also report results for cannabidivarin.
2. An assigned value (x_a) was determined for each analyte and in conjunction with the standard deviation for proficiency (σ_p) was used to calculate a z-score for each result. However, those for cannabidivarin in 3304A are given *for information only.*
3. Results for this proficiency test are summarised as follows:

analyte	assigned value, x_a	units	number of scores, $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
Cannabidiol in 3304A	0.465	% w/w	16	16	100
Cannabidivarin in 3304A	*7.78*	mg/kg	*4*	7	*57*
Cannabidiol in 3304B	0.952	% w/w	15	16	94
Cannabidivarin in 3304B	39.0	mg/kg	6	8	75

italics indicate for information only

CONTENTS

1. INTRODUCTION

1.1. Proficiency Testing

Proficiency testing aims to provide an independent assessment of the competence of participating laboratories. Together with the use of validated methods, proficiency testing is an essential element of laboratory quality assurance.

Further details of the Fapas® – Food Chemistry proficiency testing scheme are available in our protocols [4, 5].

2. TEST MATERIAL

2.1. Preparation

Preparation of the samples for this proficiency test was sub-contracted to a laboratory meeting the quality requirements of the scheme.

The test materials were prepared from olive oil spiked using cannabidiol isolate.

Samples were stored at -20°C until dispatch.

2.2. Screening and Homogeneity

To test for homogeneity, randomly selected test materials were analysed in duplicate. Testing was sub-contracted to a laboratory meeting the quality requirements of the scheme.

These data showed sufficient homogeneity and were not included in the subsequent calculation of the assigned values.

Screening was carried out before test material dispatch to ensure the samples contained less than 1 mg of the controlled cannabinoids.

2.3. Dispatch

The start date was 24 October 2023. Test materials were sent to 20 participants.

3. RESULTS

The instructions for reporting results were as follows:

Determine the level of analyte(s) present in the test material as follows:

analyte	units	comment
Cannabidiol	% w/w	as received
Cannabidivarin	mg/kg	please note that the issuing of performance assessments for cannabidivarin will be dependent on the quality of the results received

PLEASE NOTE: It is important that you report the results in this way so that we can include as many results as possible in the statistical analysis.

Results were submitted by 16 participants (80%) before the closing date for this test, 08 December 2023.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1 to Table 2.

Participants' comments are given in Table 3.

The analytical methods used by each participant are summarised in APPENDIX I.

4. STATISTICAL EVALUATION OF RESULTS

The results submitted by participants were statistically analysed in order to provide an assigned value for each analyte. The assigned values were then used in combination with the standard deviation for proficiency, σ_p, to calculate a z-score [6] for each result. The procedure is detailed in the relevant protocols [4, 5].

Further background on the procedure followed can be found in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [7].

4.1. Calculation of the Assigned Value, x_a

The assigned value, x_a, for each analyte was derived from the consensus of the results submitted by participants.

The procedure used to derive this consensus involved:

i) exclusion, if present, of any non numerical results i.e. qualitative or semi-quantitative results,

ii) exclusion, if present, of any results that were approximately 10, 100 or 1000 × greater or smaller than the majority of submitted results (as these were considered to be reporting errors).

For all analytes, the median was chosen as the assigned value because of the low number of results received. The assigned value for cannabidivarin in 3304A is given *for information only* due to the high associated uncertainty.

The assigned values for all analytes are shown in Table 4.

4.2. Standard Deviation for Proficiency, σ_p

The standard deviation for proficiency, σ_p, was set at a value that reflects best practice for the analyses in question.

For all analytes, σ_p was calculated using fitness-for-purpose criteria based on an expansion of the value calculated by the Horwitz equation. The Horwitz value on its own was not appropriate [4] for the observed sets of data.

The values for σ_p used to calculate z-scores from the reported results of this test are given in Table 4.

4.3. Individual z-Scores

Participants' z-scores were calculated as:

$$z = \frac{(x - x_a)}{\sigma_p}$$

where x = the participant's reported result,
x_a = the assigned value, see Table 4,
and σ_p = the standard deviation for proficiency, see Table 4.

Participants' z-scores for all analytes are given in Table 1 to Table 2 and shown as histograms in Figures 1–4. Those for cannabidivarin in 3304A are given *for information only*. It is possible for the z-scores published in this report to differ slightly from the z-score that can be calculated using the formula given above. These differences arise from the necessary rounding of the actual assigned values and standard deviations for proficiency prior to their publication in Table 4.

The number and percentage of z-scores in the range -2 ≤ z ≤ 2 for all analytes are given in Table 5.

5. INTERPRETATION OF SCORES

In normal circumstances, over time, about 95% of z-scores will lie in the range -2 ≤ z ≤ 2. Occasional scores in the range 2 <|z| <3 are to be expected, at a rate of 1 in 20. Whether or not such scores are of importance can only be decided by considering them in the context of the other scores obtained by that laboratory.

Scores where |z| >3 are to be expected at a rate of about 1 in 300. Given this rarity, such z-scores very strongly indicate that the result is not fit-for-purpose and almost certainly requires investigation.

The consideration of a set or sequence of z-scores over time provides more useful information than a single z-score. Examples of suitable methods of comparison are provided in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [7].

6. REFERENCES

1 Adobe Approved Trust List, https://helpx.adobe.com/acrobat/kb/approved-trust-list2.html#Whatisit accessed 24/03/2022.

2 GlobalSign AATL Document Signing FAQs, https://support.globalsign.com/aatl-document/aatl-document-signing-faqs accessed 24/03/2022.

3 ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing.

4 Fapas®, 2023, Protocol for Proficiency Testing Schemes, Version 8, January 2023, Part 1 – Common Principles.

5 Fapas®, 2023, Protocol for Proficiency Testing Schemes, Version 6, January 2023, Part 2 – Fapas® Food Chemistry scheme (FAPAS).

6 AMC Tech Brief No. 74, z-Scores and other scores in chemical proficiency testing – their meanings, and some common misconceptions, *Anal. Methods*, 2016, **8**, 5553.

7 Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem.*, **78**, No. 1, 145–196.

Table 1: Results and z-Scores for 3304A

laboratory number	analyte			
	Cannabidiol in 3304A assigned value: 0.465 % w/w		Cannabidivarin in 3304A assigned value: *7.78* mg/kg	
	result	z-score	result	z-score
001	0.4697	0.1	Below LOQ	
002	0.47	0.1	7.55	*-0.4*
003	0.4607	-0.1		
004	0.435	-0.9		
005	0.48	0.5		
006	0.41	-1.7	<500	
007	0.46	-0.2		
008	0.45	-0.5		
009	0.42	-1.4	8	*0.4*
010	0.510	1.4	<LOQ	
011	0.4559	-0.3	0	*-14.3*
012	0.5	1.1	8.54	*1.4*
013	0.4224	-1.3	7.48	*-0.5*
014	0.485	0.6		
015	0.49	0.8	6.4	*-2.5*
016	0.513	1.5	13.4	*10.3*

z-scores outside |z| >2 are shown in **bold,** see Section 5
italics indicate for information only

Table 2: Results and z-Scores for 3304B

laboratory number	analyte			
	Cannabidiol in 3304B assigned value: 0.952 % w/w		Cannabidivarin in 3304B assigned value: 39.0 mg/kg	
	result	z-score	result	z-score
001	0.9633	0.2	40.8	0.7
002	1.01	0.9	37.44	-0.6
003	0.9548	0.0		
004	0.880	-1.1		
005	0.96	0.1		
006	0.85	-1.5	<500	
007	0.95	0.0		
008	0.93	-0.3		
009	0.80	**-2.3**	36	-1.1
010	1.058	1.6	<LOQ	
011	0.9641	0.2	0	**-14.3**
012	0.98	0.4	42.77	1.4
013	0.8683	-1.3	38.04	-0.4
014	0.924	-0.4		
015	0.94	-0.2	39	0.0
016	1.01	0.9	68.2	**10.7**

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 3: Participants' Comments

laboratory number	comments
009	"733-2023-10174726 (3304A) 733-2023-10174727 (3304B)" CON-RV 23-157

comments are as submitted by participants but some may have been edited to maintain participant anonymity

Table 4: Assigned Values and Standard Deviations for Proficiency

analyte	data points, *n*	assigned value, x_a	units	uncertainty, *u*		standard deviation for proficiency, σ_p
Cannabidiol TMA	16	0.465	% w/w	0.008	ffp	0.0326
Cannabidivarin TMA	6	*7.78*	mg/kg	*0.32*	ffp	*0.544*
Cannabidiol TMB	16	0.952	% w/w	0.010	ffp	0.0667
Cannabidivarin TMB	7	39.0	mg/kg	1.0	ffp	2.73

ffp = fitness-for-purpose criteria
italics indicate for information only

Table 5: Number and Percentage of z-Scores where |z| ≤2

analyte	number of scores where |z| ≤2	total number of scores	% |z|≤2
Cannabidiol TMA	16	16	100
Cannabidivarin TMA	*4*	7	*57*
Cannabidiol TMB	15	16	94
Cannabidivarin TMB	6	8	75

italics indicate for information only

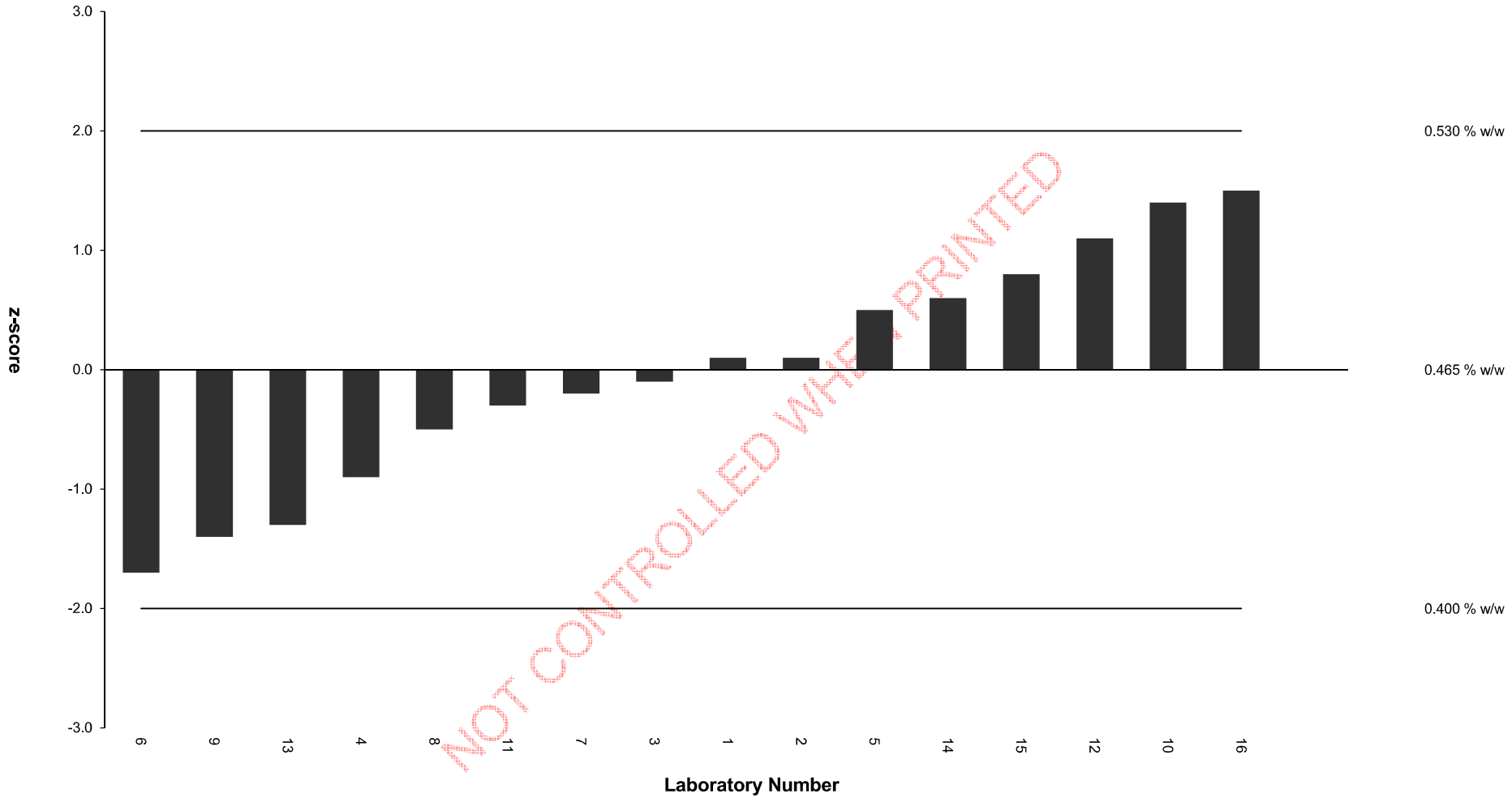

Figure 1: z-Scores for Cannabidiol in 3304A

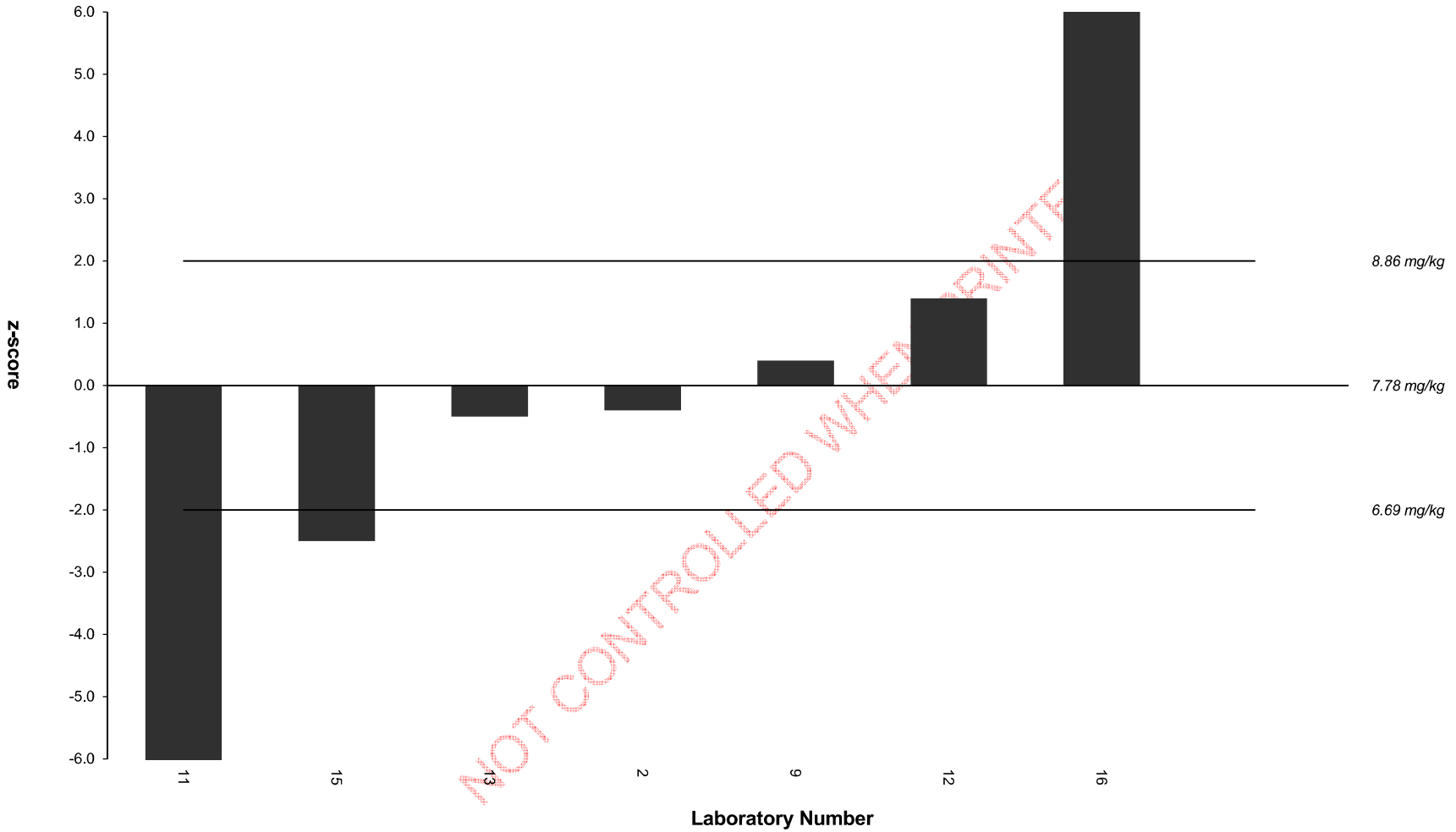

Figure 2: z-Scores for Cannabidivarin in 3304A

this histogram is given for information only

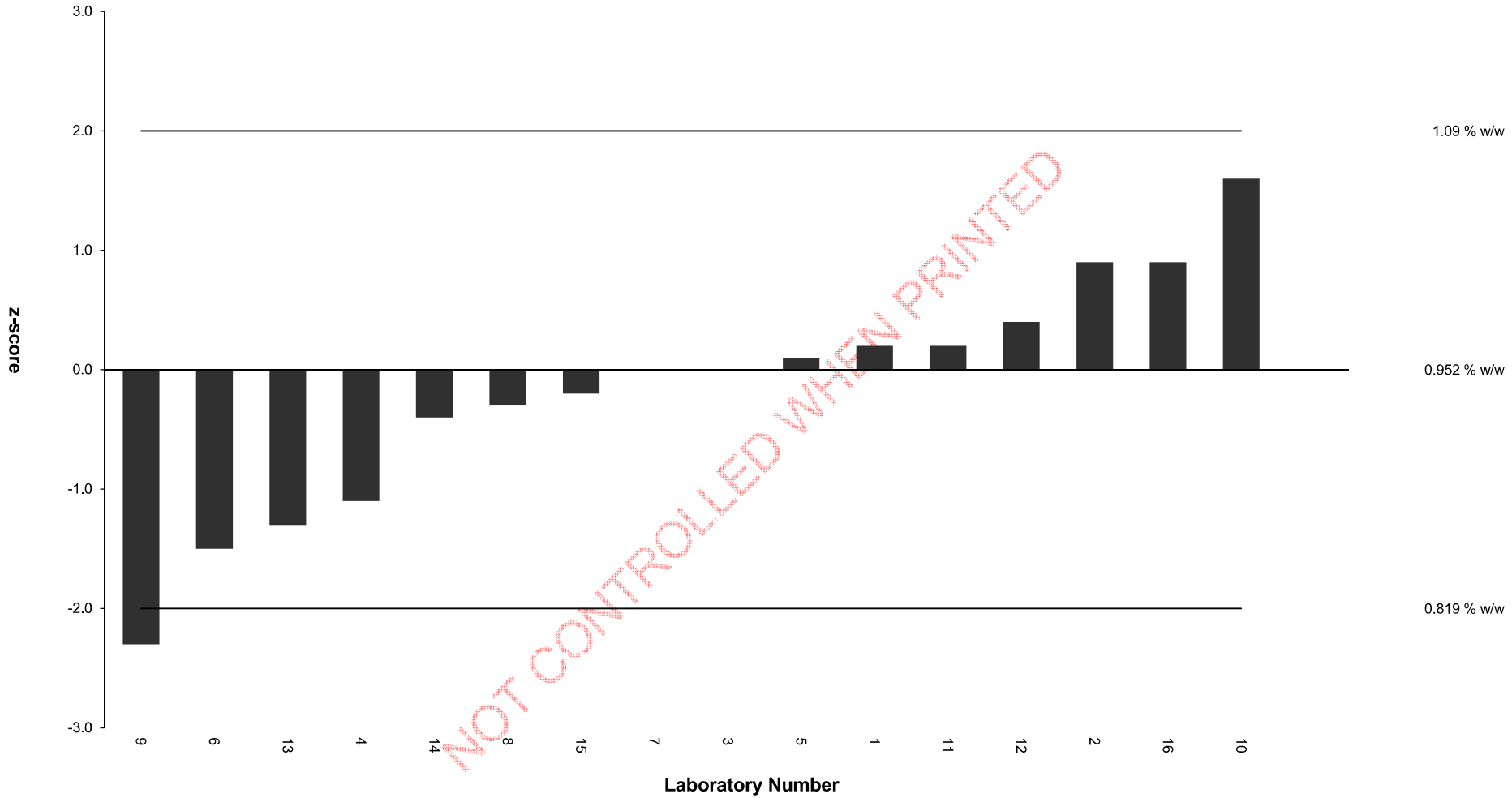

Figure 3: z-Scores for Cannabidiol in 3304B

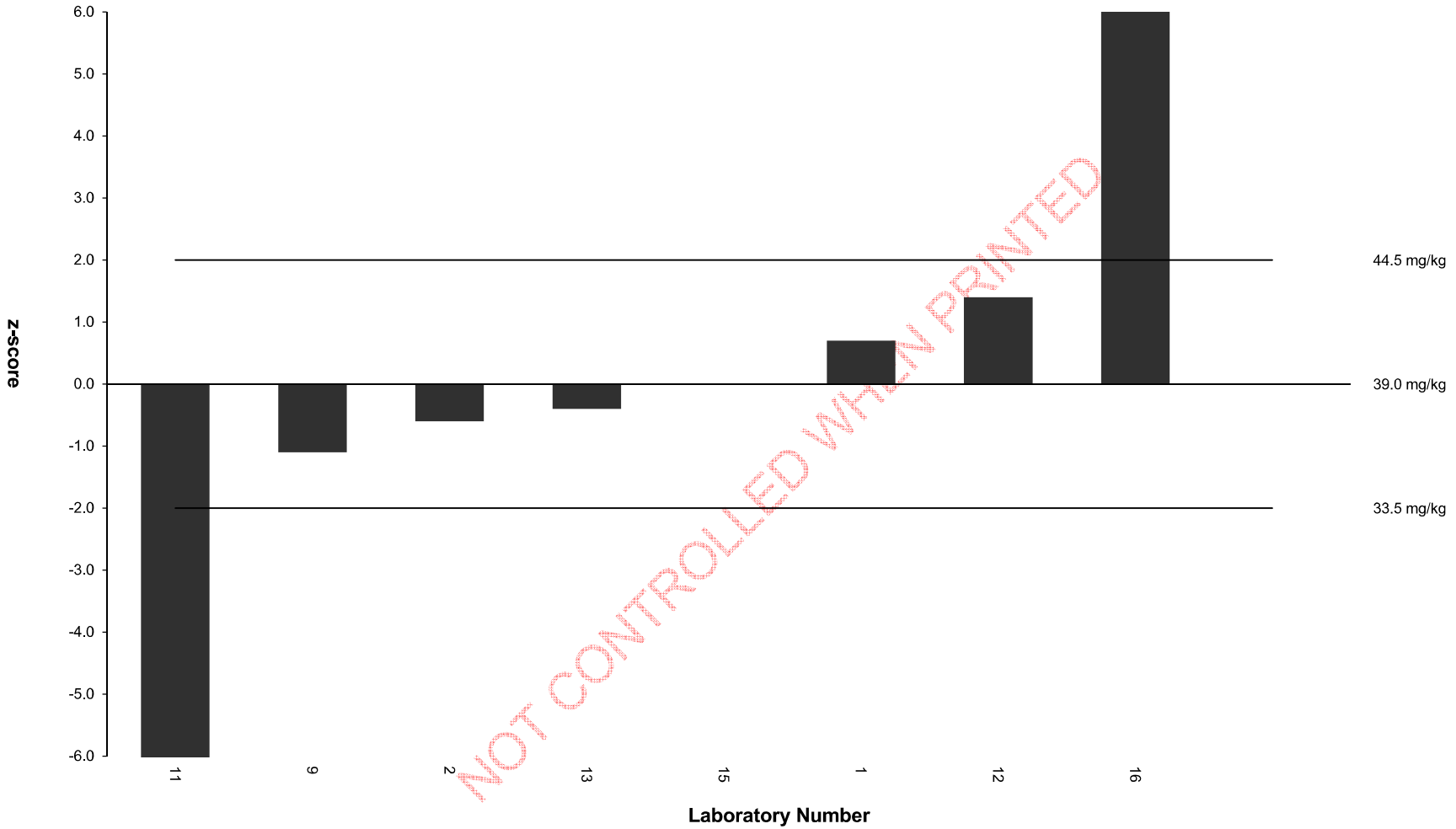

Figure 4: z-Scores for Cannabidivarin in 3304B

APPENDIX I: Analytical Methods Used by Participants

Methods are tabulated according to the information supplied by participants, but some responses may have been combined or edited for clarity. Text that appears as unreadable symbols are derived from entries made using non-Western characters.

Is the Method Used Accredited?	laboratory number
no	003 012 015
yes	001 002 004 005 006 008 010 011 013 014 016

What is Your Method Based On?	laboratory number
In house method	001 002 004 005 008 010 011 012 013 016
International Standard	003 006 015

Sample Weight (g) (please specify)	laboratory number
0,1	015
0.1	012 013
0.1-0.2	011
0.1g	002
0.2	008 010 016
0.20	003
0.25	005
0.25 g	004
0.5	001
Approx 0.5g	006

Extraction solvent components	laboratory number
Acetone / Methanol	012
Acetonirile	016
Acetonitril	015
Ethanol	001 006 010
Hexane	004
IPA	002 005
Isopropyl alcohol	011
MeOH:CHCl3 (95:5, v/v)	013
Methanol	003
methanol/water/formic acid	008

Sample Preparation & Extraction	laboratory number
Dilution	012
Ethanolic extraction of the sample followed by dilution with methanol	006
Hexane extraction	004
liquid:liquid	001
Liquid-liquid extraction	011
shaking, ultrasonic	008
Sonicated	003
Sonication to dissolve before dilution and filtering	005
Soxhlet	016
ultrasound extraction	013
vortex	002
weight of sample, weight of ethanol, vortex, sonication, filtration	010

Determination method	laboratory number
DAD UPLC	006
Diode Array Detector	001
HPLC/DAD; multiple point calibration	010
HPLC-DAD	003 004 011
HPLC-UV	005
LC/MSMS	015
LCMS	002
LC-MS	013
LC-MS/MS	016
LC-MSMS	012
PDA	008

Calibration range used	laboratory number
0.01 - 0.1 ng/µl	012
0.01mg/l - 1.0mg/L	002
0.1 to 10%	005
0.25 µg/ml - 50 µg/ml	008
0.3 -100 ng/mL	016
0.5 to 20 ppm	004

Calibration range used	laboratory number
0.5-10 ug/mL	006
1-50 ug/mL	003
20-100 ppm	011
CBD 0.4-6.4ug/mL CBDV 0.016-0.256 ug/mL	013
CBD: 0,013 - 0.70 mg/g ~ 10 - 550 mg/L	010
From 0.5ug/ml to 10ug/ml	001

Source of Standards	laboratory number
Dr Ehrenstorfer	010 013
Lipomed	003
Merck	001 004 005 012
Restek	002
Sigma/Aldrich	016
Thames Restek	011

APPENDIX II: Fapas® SecureWeb, Protocol and Contact Details

1. Fapas® SECUREWEB

Access to the secure area of our website is only available to participants in our proficiency tests. Please contact us if you require a UserID and Password. Fapas® SecureWeb allows participants to:

- Obtain their laboratory numbers for the proficiency tests in which they have participated.
- View the results they submitted in past and current proficiency tests.
- Submit their results and methods for current tests.
- Review future tests they have ordered.
- Order proficiency tests, reference materials and quality control materials.
- Freely download copies of reports (PDF file), of proficiency tests in which they have participated.
- View charts of their z-scores obtained in previous Fapas® – Food Chemistry proficiency tests.

2. PROTOCOL

The Protocols [4, 5] set out how Fapas® – Food Chemistry is organised. Copies can be downloaded from our website.

3. CONTACT DETAILS

This report was prepared and authorised on behalf of Fapas® by Claire Williamson (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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