

Report of the Inter-laboratory Comparison on Manganese and Permanganate Index in Water II (2020)

THE SECOND ROUND OF AN INTERNATIONAL STUDY

2020



Research Center for Eco-Environmental Sciences
Chinese Academy of Sciences

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Water Quality Analysis Laboratory, RCEES, CAS
Centre of Excellence for Water and Environment (CEWE), CAS-TWAS

Title

Report of the Inter-laboratory Comparison on Manganese and Permanganate Index in Water II (2020)

Authors

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Standards

ISO 13528: 2015

Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparison

GB/T 27043-2012

Conformity Assessment-General Requirements for Proficiency Testing

GB/T 28043-2019

Statistical methods for use in proficiency testing by interlaboratory comparison

JF 1117.1-2012

Measurement Comparison of Chemical Quantity

JF 1343-2012

General and Statistical Principles for Characterization of Reference Materials

CNAS-GL002: 2018

Guidance on Statistic Treatment of Proficiency Testing Results and Performance Evaluation

CNAS-GL003: 2018

Guidance on Evaluating the Homogeneity and Stability of Samples Used for Proficiency Testing

CNAS-GL032: 2018

Guidance on the Selection: Review and Use of Proficiency Testing

Key words

Inter-laboratory Comparison, Manganese, Permanganate Index, Water

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Summary

This work was the second part of the Inter-laboratory Comparison on Manganese and Permanganate Index in Water (2020), which was jointly conducted by Water Quality Analysis Laboratory, RCEES, CAS and Centre of Excellence for Water and Environment (CEWE), CAS-TWAS. And as part II of this activity, we sincerely appreciated the support from both Certification and Accreditation Administration of People's Republic of China, CNCA and RusAccreditation.

This study also included the determination of manganese and permanganate index in two different water items, which were both distributed to the participating laboratories with two testing samples at the same concentration respectively. Based on the close cooperation between China and Russia in the field of the inspection and testing industry, 9 representative laboratories were recommended by RusAccreditation to participate this project.

Totally 13 sets of testing samples were sent to the 9 different laboratories. This report II presents the returned results for 9 sets of manganese samples and 4 sets of permanganate index samples.

The standard value for each analyte in the testing samples were determined by National Institute of Metrology, China. All values exceeding $\pm 50\%$ of the assigned concentrations were removed from the calculation. The consensus mean and the standard deviation (SD) were calculated from the remaining data, while this SD and the consensus mean were used to subsequently calculate z-scores.

For the samples of manganese(-a and -b), z-scores within ± 2 were obtained by 66.7% of the reporting participants (corresponding to 6 of the total 9 participants).

For the samples of permanganate index(-a and -b), within ± 2 were obtained by 75% of the reporting participants (corresponding to 3 of the total 4 participants).

Introduction

Inter-laboratory Comparison is an effective way to improve the quality control system for the analytical laboratories through external measures, which is particularly becoming of increasing importance in the context of globalization of world economy.

The main objective of the activity is to assess the between laboratory reproducibility on water quality analysis, and to provide a QA/QC tool for each participating laboratory to improve their performance.

The second part of this activity started from November 2020. And because of the Customs inspection when delivering samples, 8 sets of samples were distributed to the 5 laboratories again in January 2021. Therefore, The work of this part was ended in March 2021 when all the result reports were received. And the report was drafted on April 2021.

From the end of 2019, the worldwide SARS-CoV-2 epidemic had brought great challenges to the implementation of this inter-laboratory comparison work. We sincerely appreciate all the participants and individual analysts for their professional contribution to to this activity.

Design and practical implementation

Study design and reporting of results

The analysis should be performed using the laboratories' own testing methods, their own quantification standards and quantification procedures. Table 1 showed the testing methods from the result reports of participants.

Table 1 Testing methods from the participants in the Inter-laboratory Comparison on Manganese and Permanganate Index in Water II (2020)

Items	Testing Methods	Amounts
Manganese	Inductively Coupled Plasma Optical Emission Spectrometer(ICP)	3
	Atomic Absorption Spectroscopy (AAS)	6
Permanganate Index	Acid Potassium Permanganate Titration Method	4

Samples design, sample distribution, confidentiality and statistical analysis were all consistent with those in the previous report completed on March 2021. Table 2 showed the assigned value of the testing sampls and the acceptable range of results.

Z-score was adopted to evaluate the results in the inter-laboratory comparison. Z-score was calculated according to the equation (1):

$$Z = \frac{x - \bar{X}}{\hat{\sigma}} \dots\dots\dots(1)$$

Where x =reported value; \bar{X} =assigned value; $\hat{\sigma}$ =SD. $|z| \leq 2.0$ means a satisfied result; $2.0 < |z| < 3.0$ means a problematic result; $|z| \geq 3.0$ means an unsatisfied result.

There were three kinds of evaluation results: satisfactory, problematic and unsatisfactory. For each laboratory, only when both samples meet the conditions of " $|z| \leq 2.0$ ", a satisfied result will be achieved. Otherwise, the result will be evaluated as unsatisfied.

Table 2 Acceptable range of results in the Inter-laboratory Comparison on Manganese and Permanganate Index in Water II (2020)

Items	Assigned value(mg/L)	$ z \leq 2.0$	Minimum Concentration (mg/L)	Maximum Concentration (mg/L)	Lab to Lab Accuracy(%)
Manganese	23.9	Satisfied	21.31	26.5	≤ 11
Permanganate Index	107	Satisfied	78	136	≤ 27

The final report and certificate

The final report of the Inter-laboratory Comparison on Manganese and Permanganate Index in Water II (2020) was drafted by the coordinators and published on April 2021.

A certificate of participation was sent to each laboratory who has contributed to the results by the end of April 2021.

Coordination

This activity was initiated by CNCA and RCEES, and jointly carried out by the Water Quality Analysis Laboratory and CAS-TWAS Centre of Excellence for Water and Environment (CEWE), RCEES. Members of the coordination committee were:

- Dr. Hongyan LI, Senior engineer
- Prof. Min YANG, Director
- szfxsys@126.com; cas_twas@rcees.ac.cn

Results

Manganese

For the testing samples of manganese, results from 9 laboratories were received. The assigned value of manganese is 23.9 mg/L, the uncertainties is 1% and SD was 1.31.

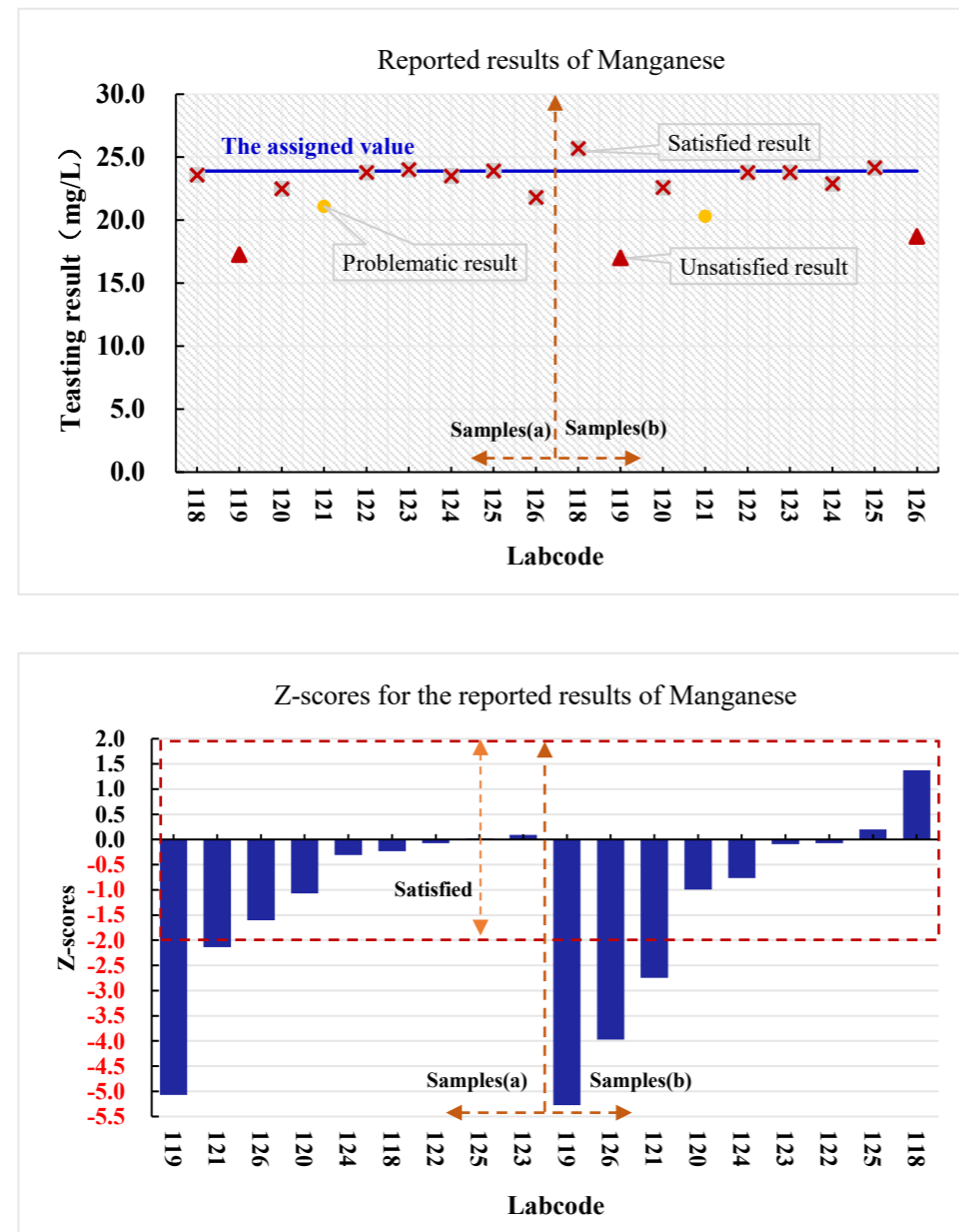


Figure 1 Study results of Manganese

Figure 1 showed the study results of manganese. It could be seen that of the 9 participating laboratories, 6 achieved z-scores within ± 2 as satisfied results, and 1 obtained z-scores over ± 3 as unsatisfied results. In addition, one laboratory reported both testing results with Z-score of 2-3 as problematic results, and one laboratory submitted the testing results with z-score of -1.6 for manganese-a as satisfied result while z-score of -4.0 for manganese-b as unsatisfied result. Result of each participant were presented in Appendix C.

Permanganate Index

For the samples of permanganate index, results from 4 laboratories were received. The assigned concentration of permanganate index is 107 mg/L, the uncertainties is 1.7% and SD is 14.72.

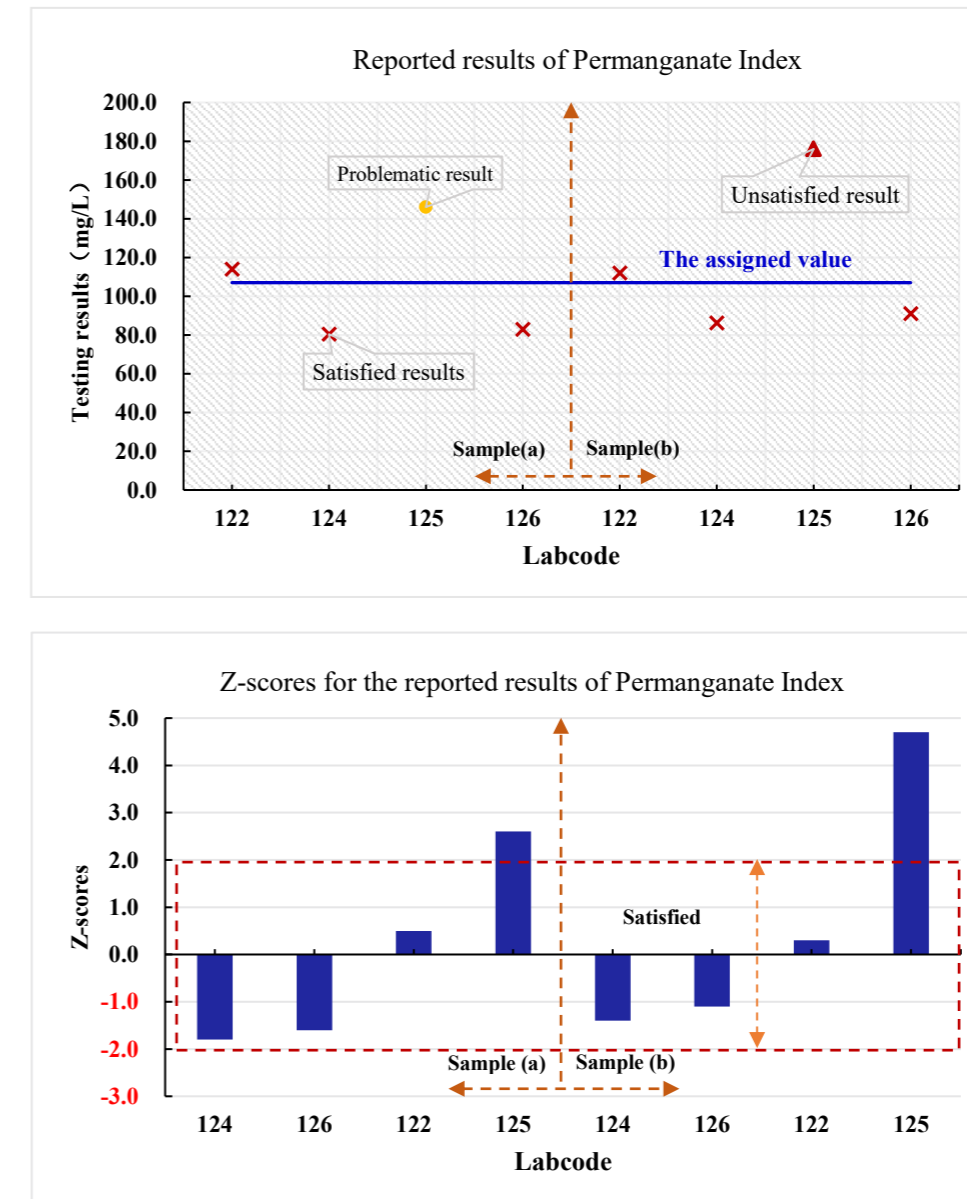


Figure 2 Study results of Permanganate Index

Figure 2 showed the study results of permanganate index. It could be seen that of the 4 participating laboratories, 3 achieved z-scores of ± 2 as satisfied results, and one laboratory submitted the testing results with z-score of 2.6 for permanganate index-a as problematic result and z-score of 4.7 for permanganate index-b as unsatisfied result. Result of each participant were presented in Appendix C.

Technical analysis

Through analyzing the original records and testing methods, the reasons for the unsatisfactory evaluation results would help to identify the potential risk of data and improve the testing ability of laboratories. As the previous report, the z-score of intra-laboratory (ZW) in the “Guidance on Statistic Treatment of Proficiency Testing Results and Performance Evaluation CNAS-GL002:2018” was also introduced to evaluate the risk of all returned results.

$|ZW| \leq 2.0$ means low risk of inaccuracy; $|ZW| > 2.0$ means moderate risk of inaccuracy; $|ZW| \geq 3.0$ means high risk of inaccuracy, or the results are not even inaccurate.

ZW of Manganese

Figure 3 showed the data security risk identification status of 9 laboratories that returned the results of manganese. Two of the laboratories with satisfactory results, Lab 118 and Lab 124 were at high and medium risk of inaccuracy respectively. Among the 3 non-satisfied laboratories, 2 laboratories (Lab 121, Lab 126), which presented results in the medium and high risk of inaccuracy respectively, provided some original records. Unfortunately, this part of original records was insufficient to make technical traceability.

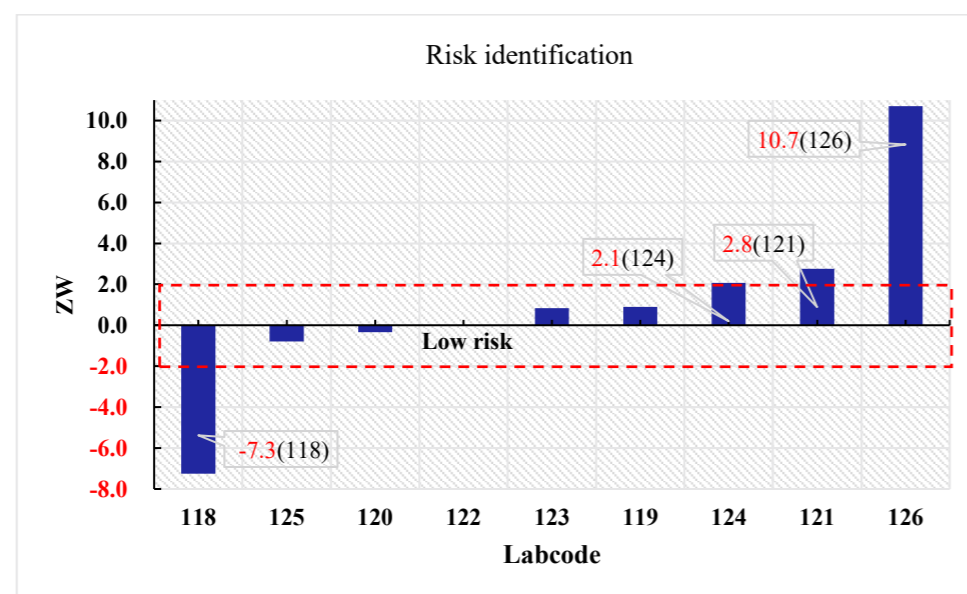


Figure 3 Study results (ZW) of Manganese

ZW of Permanganate Index

Figure 4 showed the data security risk identification status of 4 laboratories that returned the results of permanganate index. All the 3 laboratories with satisfied results showed low risk of inaccuracy. And the laboratory with unsatisfied results was high risk of inaccuracy as well. However, the laboratory (lab125) has not provided original records, so it is difficult to make technical traceability.

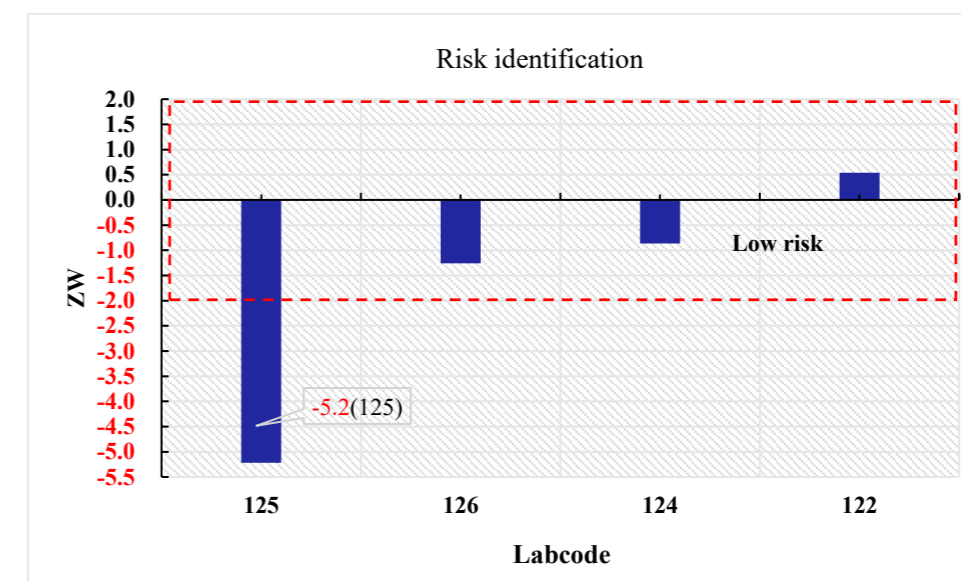


Figure 4 Study results (ZW) of Permanganate Index

Analysis of the testing methods

For the manganese testing, the laboratories with unsatisfied results or high risk of inaccuracy mainly used the atomic absorption method. For this method, more attention should be paid to the optical path regulation and atomization temperature.

For the determination of permanganate index, all the 4 laboratories used the titration method in the standard of "Water quality - Determination of permanganate index (ISO 8467:1993)". For this method, manual titration processes, such as blank treatment, water bath and identification of the titration end, should be noted.

Acknowledgement

Thanks support from Certification and Accreditation Administration (Grant No.[2020]24) and support from the Alliance of International Science Organizations(Grant No.ANSO-CR-KP-2020-05); Special thanks to Director Dong GUO from CNCA and Mr. Egor from RusAccreditation for the support to this project.

Appendix A-1 Operation Instruction

“Manganese in water” Operation Instruction

Participating laboratories:

The Inter-laboratory Comparison on Manganese and Permanganate Index in Water 2020 is organized and implemented by the CAS-TWAS Center of Excellence for Water and Environment Research Center for Eco-environmental Sciences Chinese Academy of Sciences. In this project, your laboratory code is XXX. The relevant information of the project is as follows:

To ensure the smooth implementation of the capacity verification, please read the following instructions carefully before testing:

1. Test sample description

1.1 This operation instruction is for the determination of **manganese in water**, and the assessment samples will be provided randomly according to the registration information.

1.2 **Two** samples provided for this test are packaged in ampoules with volume about 20 mL, numbered **MXXXa** and **MXXXb**. The matrix is 2% HNO₃. The reference concentration of the manganese in samples is between 2.50 mg/L~37.50 mg/L (before the dilution).

1.3 The samples will be delivered from CAS-TWAS Center of Excellence for Water and Environment Research Center for Eco-environmental Sciences Chinese Academy of Sciences.

1.4 Upon receipt, please confirm that the sample is in good condition. Please fill in the **Sample Receiving Status Confirmation Form** within 7 days after receipt and send the scanned copy of the form to szfxsys@126.com. If the sample received is damaged, please contact with szfxsys@126.com in time and apply for replacement (Note: The replacement is only for damage caused by transportation, but not that caused by experimental operations).

1.5 Store in dark with room temperature, and test as soon as possible.

2. Test description

2.1 Dilution method: Use a clean and dry pipette to accurately remove 10 mL of the sample from the ampoule, transfer it to a 250 mL volumetric flask, dilute to volume with ultrapure water or as required by the test method, and test immediately after mixing. Each sample must be tested in duplicate.

2.2 The actual testing methods of each laboratory should be consistent with that in the registration form. If there is any change, instructions for the change should be submitted and the registration form should be resubmitted.

3. Result report

3.1 The results of "Manganese in water" should be reported in mg/L with **the concentration before dilution** in the 《Capability Verification Results Report Form》. At the same time, the average results should be calculated and retained 3-digit valid numbers. If the laboratory can evaluate the uncertainty of the results, please give the extended uncertainty (U) (k=2) in the report.

3.2 Each laboratory should send the 《Capability Verification Results Report Form》 and the detailed original records to cas_twas@rcees.ac.cn within 30 natural days (including weekends and national holidays) since the receipt of the samples. It will not be count and evaluated if the results are not sent in time.

3.3 During the implementation of this proficiency testing program, each laboratory should pay attention to confidentiality, independently complete the experiment and submit the report.

Note: The original records should include quality control samples, standard samples, parallel samples and other quality control measures. Quality control measures should reflect the reliability of test results.

4. Contact information

If you have any question during the competency verification process, please contact with CAS-TWAS Center of Excellence for Water and Environment Research Center for Eco-environmental Sciences Chinese Academy of Sciences.

Contact: Si, Ludan

Contact number: +86-10-62849800

E-mail: cas_twas@rcees.ac.cn

Contact address: CAS-TWAS Center of Excellence for Water and Environment Research Center for Eco-environmental Sciences Chinese Academy of Sciences, Beijing 100085, CHINA

“Permanganate index in water” Operation Instruction

Participating laboratories:

The Inter-laboratory Comparison on Manganese and Permanganate Index in Water 2020 is organized and implemented by the CAS-TWAS Center of Excellence for Water and Environment Research Center for Eco-environmental Sciences Chinese Academy of Sciences. In this project, your laboratory code is XXX. The relevant information of the project is as follows:

To ensure the smooth implementation of the capacity verification, please read the following instructions carefully before testing:

1. Test sample description

1.1 This operation instruction is for the determination of **permanganate index in water**, and the assessment samples will be provided randomly according to the registration information.

1.2 **Two** samples provided for this test are packaged in ampoules with volume about 20 mL, numbered **PXXXa** and **PXXXb**. The matrix is water. The reference concentration of the permanganate index in samples is between 50.00 mg/L~250.00 mg/L (before the dilution).

1.3 The samples will be delivered from CAS-TWAS Center of Excellence for Water and Environment Research Center for Eco-environmental Sciences Chinese Academy of Sciences.

1.4 Upon receipt, please confirm that the sample is in good condition. Please fill in the **Sample Receiving Status Confirmation Form** within 7 days after receipt and send the scanned copy of the form to szfxsys@126.com. If the sample received is damaged, please contact with szfxsys@126.com in time and apply for replacement (Note: The replacement is only for damage caused by transportation, but not that caused by experimental operations).

1.5 Store in dark with room temperature, and test as soon as possible.

2. Test description

2.1 Dilution method: Use a clean and dry pipette to accurately remove 10 mL of the sample from the ampoule, transfer it to a 500 mL volumetric flask, dilute to volume with ultrapure water or as required by the test method, and test immediately after mixing. Each sample must be tested in duplicate.

2.2 The actual testing methods of each laboratory should be consistent with that in the registration form. If there is any change, instructions for the change should be submitted and the registration form should be resubmitted.

3. Result report

3.1 The results of "Permanganate index in water" should be reported in mg/L with **the concentration before dilution** in the 《Capability Verification Results Report Form》. At the same time, the average results should be calculated and retained 3-digit valid numbers. If the laboratory can evaluate the uncertainty of the results, please give the extended uncertainty (U) (k=2) in the report.

3.2 Each laboratory should send the 《Capability Verification Results Report Form》 and the detailed original records to cas_twas@rcees.ac.cn within 30 natural days (including weekends and national holidays) since the receipt of the samples. It will not be count and evaluated if the results are not sent in time.

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Contact: Si, Ludan

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E-mail: cas_twas@rcees.ac.cn

Contact address: CAS-TWAS Center of Excellence for Water and Environment Research Center for Eco-environmental Sciences Chinese Academy of Sciences, Beijing 100085, CHINA

Appendix A-2 Sample Receiving Status Confirmation Form

Sample Receiving Status Confirmation Form

Laboratory Name		
Laboratory Code		
Accepted Date		
Accepted Sample	Sample Amount	<input type="checkbox"/> 2 <input type="checkbox"/> 4
	Sample Number	
	Sample Status	<input type="checkbox"/> in good condition <input type="checkbox"/> broken Note: If the samples are broken, please attach sample photos when returning this form.
Recipient	Name	
	E-Mail	

Appendix A-3 Capability Verification Results Report Form

Capability Verification Results Report Form

“Manganese in water” sample test results report

Laboratory name: _____ Laboratory code: _____

Report date: _____

Sample number	Test results (mg/L)			Extended uncertainty (k=2)	Name and number of the test method	Ambient temperature	Instrument name and model	Date of inspection	Inspector signature	Signature of the certifier
	1	2	Average							

Problems or anomalies that occur during the experiment: _____

(Not enough, please attach a page)

Person in charge (signature): _____

Official seal: _____

Capability Verification Results Report Form

“Permanganate index in water” sample test results report

Laboratory name: _____ Laboratory code: _____

Report date: _____

Sample number	Test results (mg/L)			Extended uncertainty (k=2)	Name and number of the test method	Ambient temperature	Instrument name and model	Date of inspection	Inspector signature	Signature of the certifier
	1	2	Average							

Problems or anomalies that occur during the experiment: _____

(Not enough, please attach a page)

Person in charge (signature): _____

Official seal: _____

国家认证认可监督管理委员会

认秘函〔2020〕24号

认监委秘书处关于开展食品、水质、矿产 检验检测能力验证活动的通知

中国检验检疫科学研究院测试评价中心、中国科学院生态环境研究中心、北京中实国金国际实验室能力验证研究中心、各有关检验检测机构：

为贯彻落实党中央、国务院关于做好“六稳”工作、落实“六保”任务的决策部署，保障粮食安全和外贸产业链畅通运转，充分发挥检验检测对“一带一路”建设的技术支撑作用，经研究，决定在食品、水质和矿产检验检测领域组织开展能力验证活动，组织国内外相关检验检测机构共同参与，推动检验检测数据、结果的采信和互认，促进与相关国家在检验检测领域开展更大范围、更深层次的务实合作。现将有关事项通知如下：

一、能力验证项目和参加要求

本次能力验证活动，委托中国检验检疫科学研究院测试评价中心承担“鸡肉中硝基呋喃类兽药残留定量分析”项目实施，委托中国科学院生态环境研究中心承担“水中锰和高锰酸盐指数检测”项目实施，委托北京中实国金国际实验室能力验证研究中心承担“铜精矿中铜、硫、金、银的测定”项目实施。

取得国家认监委颁发的资质认定证书，且具备相关检测项目技术能力的国家产品质检中心应当报名参加相关能力验证项目；因故不能参加的，须向项目承担单位提交书面情况说明。国家认监委将从国内报名单位中选取部分检验检测机构参与本次能力验证活动。

项目承担单位负责联系和邀请“一带一路”沿线国家和其他国家的检验检测机构参加本次能力验证活动。

二、检测标准和样品信息

“鸡肉中硝基呋喃类兽药残留定量分析”可采用 GB/T 21311-2007《动物源性食品中硝基呋喃类药物代谢物残留量检测方法 高效液相色谱/串联质谱法》、SN/T 3380-2012《出口动物源食品中硝基呋喃代谢物残留量的测定 酶联免疫吸附法》、DB34/T 1838-2013《动物源性组织中硝基呋喃类药物代谢物残留量检测方法 高效液相色谱荧光法》等标准方法或实验室内部方法。测试样品为鸡肉冻干粉，满足常温运输要求，样品规格 10 克/包，每个检验检测机构发放 1 种规格 1 包样品。

“水中锰和高锰酸盐指数检测”可采用 ISO 8467:1993《Water quality - Determination of permanganate index》，GB/T 5750.7-2006《生活饮用水标准检验方法 有机物综合指标》，ISO 6333-1986《Water quality - Determination of manganese- Form aldoxime spectrometric method》，GB/T 5750.6-2006《生活饮用水标准检验方法 金属指标》等标准方法。测试样品为水溶液，

洁净安瓿瓶包装，样品规格 20 毫升/瓶，每个检验检测机构发放 1 个浓度水平样品 2 瓶。

“铜精矿中铜、硫、金、银的测定”可采用 GB/T3884 系列铜精矿化学分析方法，ISO 10258:2018 《Copper sulfide concentrates — Determination of copper content — Titrimetric methods》，ISO10378:2016 《Copper, lead and zinc sulfide concentrates — Determination of gold and silver — Fire assay gravimetric and flame atomic absorption spectrometric method》等标准方法。测试样品为 2 种规格的铜精矿样品，用玻璃瓶包装，60 克/瓶，每个检验检测机构发放 1 种规格 1 瓶样品。

三、时间安排

- (一) 机构报名：2020 年 7 月—8 月；
- (二) 样品发放：2020 年 10 月前；
- (三) 检测结果反馈：2020 年 10 月底前；
- (四) 初步技术分析报告：2020 年 11 月底前；
- (五) 结果发布：2020 年 12 月底前。

四、其他事宜

- (一) 本次能力验证活动不收取费用。
- (二) 报名参加的检验检测机构应填写报名表（见附件），通过发送电子邮件方式进行报名。

(三) 联系方式

1. “鸡肉中硝基呋喃类兽药残留定量分析”能力验证项目
中国检验检疫科学研究院测试评价中心

— 3 —

黎焯昕，+86+010-53897814， liyx@acas.com.cn

地址：北京市亦庄经济技术开发区荣华南路 11 号，100176

2. “水中锰和高锰酸盐指数检测”能力验证项目

中国科学院生态环境研究中心

李红岩，郑蓓：+86+010-62849135， szfxsys@126.com

地址：北京市海淀区双清路 18 号，100085

3. “铜精矿中铜、硫、金、银的测定”能力验证项目

朱生慧：+86+010-62185713， zsh@analysis.org.cn

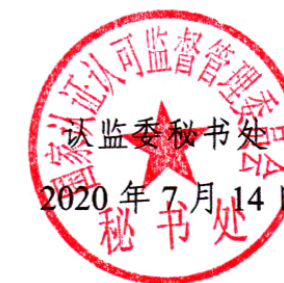
唐凌天：+86+010-62182652， tanglt@analysis.org.cn

地址：北京市海淀区高粱桥斜街 13 号新材料大楼十层

附件：1. 鸡肉中硝基呋喃类兽药残留定量分析能力验证报名表

2. 水中锰和高锰酸盐指数检测能力验证报名表

3. 铜精矿中铜、硫、金、银的测定能力验证报名表



(此件不公开)

— 4 —

Appendix C Presentation of results for manganese and permanganate index

Lab code	country	Item	comprehensive assessment Conclusion	Testing method	Sample code	Conc 1 (mg/L)	Conc 2 (mg/L)	Mean values (mg/L)	z-score	Conclusion	Sample code	Conc 1 (mg/L)	Conc 2 (mg/L)	Mean values (mg/L)	z-score	Conclusion
118	Russian Federation	Manganese	satisfied	AAS	M118a	23.5	23.7	23.6	-0.2	satisfied	M118b	25.9	25.5	25.7	1.4	satisfied
119			unsatisfied		M119a	17.328	17.181	17.255	-5.1§	unsatisfied	M119b	16.827	17.16	16.994	-5.3§	unsatisfied
120			satisfied	ICP	M120a	22.8	22.3	22.5	-1.1	satisfied	M120b	22.4	22.8	22.6	-1.0	satisfied
121			problematic	AAS	M121a	21.7	20.4	21.1	-2.1*	problematic	M121b	20.5	20.0	20.3	2.8*	problematic
122			satisfied		M122a	23.8	23.8	23.8	-0.1	satisfied	M122b	23.8	23.7	23.8	-0.1	satisfied
123			satisfied	ICP	M123a	24.15	23.89	24.02	0.1	satisfied	M123b	23.74	23.82	23.78	-0.1	satisfied
124			satisfied		M124a	23.7	23.3	23.5	-0.3	satisfied	M124b	22.8	22.9	22.9	-0.8	satisfied
125			satisfied	AAS	M125a	23.65	24.21	23.93	0.0	satisfied	M125b	24.11	24.20	24.16	0.2	satisfied
126			unsatisfied		M126a	21.9, 21.5	22.1	21.8	-1.6	satisfied	M126b	18.6	18.7, 18.7	18.7	-4.0§	unsatisfied
122			Permanganate Index	satisfied	Acid potassium permanganate titration method	P122a	115	113.5	114	0.5	satisfied	P122b	111.5	113.5	112	0.30
124	satisfied	P124a		80.4		80.4	80.4	-1.8	satisfied	P124b	86.2	86.2	86.2	-1.4	satisfied	
125	unsatisfied	P125a		144		148	146	2.6*	problematic	P125b	176	176	176	4.7§	unsatisfied	
126	satisfied	P126a		83.0		83.0	83.0	-1.6	satisfied	P126 b	91.0	91.0	91.0	-1.1	satisfied	
Notes	Manganese Testing: assigned vale =23.9, standard deviation =1.31.Permanganate Index Testing: assigned vale =107, standard deviation =14.72.									Z ≤ 2.0 means a satisfied result; 2.0 < Z < 3.0 means a problematic result, which is marked with * in the table; Z ≥ 3.0 means an unsatisfied result, which is marked with § in the table. The evaluation is "unsatisfactory", when any results in the two samples get a z >2.0.						