The Proficiency Testing of Determination of Veterinary Drug Multi-Residues in Chicken

M CTI 03/12A

September 2014, Beijing China

Chinese Academy of Inspection and Quarantine (CAIQ)

Overview

- This sub-project is under the Multi Year Project, entitled ***Building Convergence in Food Safety Standards and Regulatory Systems*** (CTI 03 12A) funded by APEC Technical Assistance and Training Facility (**TATF**)

- APEC funding:124,000 ;China co-**funding 137,928** USD.

- This project responds to the newly revised and agreed Food Safety Capacity Building Priority Areas 2011-2015 of APEC Food Safety Cooperation Forum (FSCF).

Overview

- This project is carried out by Chinese Academy of Inspection and Quarantine (under AQSIQ) with great assistance from APLAC, ASEAN, FSANZ, NMIA, US FDA, and USDA, etc.

China National Accreditation Service for Conformity Assessment

LABORATORY ACCREDITATION CERTIFICATE

(Registration No. CNAS L0424)

Allied Inspection Center of

Chinese Academy of Inspection and Quarantine

No.A3, Gaobeidian North Road, Chaoyang District, Beijing, China

is accredited to ISO/IEC 17025:2005 General Requirements for the Competence of Testing and Calibration Laboratories(CNAS-CL01 Accreditation Criteria for the Competence of Testing and Calibration Laboratories) for the competence of testing

The scope of accreditation is detailed in the attached appendices bearing the same registration number as above. The appendices form an integral part of this certificate.

Date of Issue: 2011-07-07

Date of Expiry: 2014-07-06

Date of Initial Accreditation: 2003-05-12

Date of Update: 2011-07-07

Signed on behalf of China National Accreditation Service for Conformity Assessment

No.CNAS AL 2

00C1641

China National Accreditation Service for Conformity Assessment

PROFICIENCY TESTING PROVIDER ACCREDITATION CERTIFICATE

(Registration No. CNAS PT0026)

Comprehensive Inspection Center,

Chinese Academy of Inspection & Quarantine

No.A3, Gaobeidian North Road, Chaoyang District, Beijing, China

is accredited to ISO/IEC 17043:2010 General Assessment-General Requirements for Proficiency Testing (CNAS-CL03 Accreditation Criteria for Proficiency Testing Providers) for the competence to provide proficiency testing schemes.

The scope of accreditation is detailed in the attached appendices bearing the same registration number as above. The appendices form an integral part of this certificate.

Date of Issue: 2012-12-27

Date of Expiry: 2015-12-26

Date of Initial Accreditation: 2012-12-27

Date of Update: 2012-12-27

Signed on behalf of China National Accreditation Service for Conformity Assessment

China National Accreditation Service for Conformity Assessment (CNAS) is authorized by Certification and Accreditation Administration of the People's Republic of China (CNCA) to operate the national accreditation schemes for conformity assessment. CNAS is the full member of International Laboratory Accreditation Cooperation (ILAC) and Asia Pacific Laboratory Accreditation Cooperation (APLAC).

No.CNAS APT 2

0000051

Objective

- This project is an inter-laboratory proficiency testing (PT) program, to determine veterinary drug multi-residues in animal origin products, which is important to develop laboratory capabilities within APEC economies, to improve the acceptability of test results so as to facilitate animal origin products trade among APEC economies.

Timetable

Timelines	Activities	Key Deliverables
Dec. 2012 – July 2013	1.1 Expert Working Group Establishment	List of Member
	1.2 Develop and confirm PT scheme	Confirmed PT scheme
	1.3 Laboratories Recruitment	List of Participant Laboratories
May- Oct. 2013	2.1 Testing Items Preparation	Prepared Items; Homogeneity and Stability of Items; Assigned Value and Acceptable Range
Nov. 2013 --- Feb. 2014	2.2 Testing Items Distribution and Analysis	Testing Result

Timetable

Timelines	Activities	Key Deliverables
Dec. 2013 --- March 2014	2.3 Results collection and Data Analysis	Data form
April – August 2014	3.1 Data Analysis	Develop Interium Report and draft final report
September 10-11, 2014	3.2 Workshop in China	PT Report; Improvement Proposal
Sep.-Oct. 2014	3.3 Evaluation and Completion	final Report

Progress

- PT scheme development
- Invitation and nomination/Sample preparation
- Samples deliver and analysis
- Data collection and analysis
- PT workshop

PT Scheme Development

- Following requirement of ISO/IEC 17043
- Testing items choosing
- Agreement of Testing Methods
- Sample preparation method confirmation
- Statistical Assessments method selecting
- Potential participants defining
- Budget development

Invitation and Nomination

Dear FSCF members,

Veterinary Drug Multi-residues in Chicken Proficiency Testing Program (Briefing as PT Program) is one of laboratory capacity building events under the MYP *Building Convergence in Food Safety Standards and Regulatory Systems* (M CTI 02 12A) hosted by FSCF and led by China. The PT Program is developed and now circulated for your comments.

Please respond by March 30 with any comments to this approach.

Veterinary Drug Multi-residues in Chicken Proficiency Testing Program

Objective

The objective of this proficiency testing program is to evaluate the competence of laboratories for quantitative testing of veterinary drug multi-residues including 3-amino-2-oxazolidone (AOZ), 5-morpholinomethyl-3-amino-2-oxazolidone (AMOZ), sulfamethoxazole, sulfadimidine, sulfaquinoxaline, ciprofloxacin in chicken samples.

Organization

The program will be coordinated by Chinese Academy of Inspection and Quarantine, AQSIQ, China.

Coordinator

Dr. LIU Hanxia 8610 85781069, liuhanxia_cn@163.com.

Participants

Food testing laboratories of APEC economies are encouraged to participate the PT program. Each economy is suggested to nominate not more than 2 participants.

Samples

Two vacuum freeze dried chicken samples will be supplied to each participant.

Homogeneity and Stability Evaluation

Ten and twelve samples will be tested for homogeneity and stability evaluation respectively. The samples were prepared and tested by Chinese Academy of Inspection and Quarantine Comprehensive Test Center (CAIQTEST). CAIQTEST is accredited according to ISO/IEC 17025 and ISO 17043.

Tests

The participating laboratories may choose one or more of the following analysts: AOZ, items of AOZ, AMOZ, sulfamethoxazole, sulfadimidine, sulfaquinoxaline and ciprofloxacin in the samples to take part in this proficiency testing program.

Methodology

For testing each item, routine methods should be preferably used, such as LC/MS or LC/MSn method. Other methods could also be used, however, the limit of quantitation of AOZ and AMOZ should be higher than 0.5

APLAC

Asia Pacific Laboratory Accreditation Cooperation

Invitation Letter to APEC PT on Veterinary Drug Residue

22 May 2013

APLAC PT Committee Chair

Koichi NARA

Dear PT Committee members and APLAC PT Contacts,

APEC FSCF will be running a PT of "Veterinary Drug Multi-residues in Chicken ". You are invited to nominate up to 2 laboratories without participation fee. The information is given as the following.

Objective

The objective of this proficiency testing program is to evaluate the competence of laboratories for quantitative testing of veterinary drug multi-residues including 3-amino-2-oxazolidone (AOZ), 5-morpholinomethyl-3-amino-2-oxazolidone (AMOZ), sulfamethoxazole, sulfadimidine, sulfaquinoxaline, ciprofloxacin in chicken samples.

Coordinating Organization

The program will be coordinated by Chinese Academy of Inspection and Quarantine, AQSIQ, China.

Coordinator

Dr. LIU Hanxia 8610 85781069, liuhanxia_cn@163.com.

Test materials and analytes

Two vacuum freeze dried chicken samples will be supplied to each participant. Each sample will be packaged in vial. The participating laboratories may choose one or more of the following analysts: AOZ, AMOZ, sulfamethoxazole, sulfadimidine, sulfaquinoxaline and ciprofloxacin in the samples to take part in this proficiency testing program.

Methodology

For testing each item, routine methods should be preferably used, such as LC/MS or LC/MSn method. Other methods could also be used, however, the limit of quantitation of AOZ and AMOZ

1 / 2

Invitation and Nomination

- 30 labs from 13 economies have participated in the PT Program.

Economy	Number of Labs
Australia	1
Canada	4
Chile	2
Hong Kong, China	2
Indonesia	1
Malaysia	2
New Zealand	1
Peru	1
People's Republic of China	2
Singapore	2
Chinese Taipei	5
Thailand	5
The United States	1

Sample Preparation

Sample Preparation

Sample Preparation

------Homogeneity (ISO 13528)

Table C.1-1 Homogeneity Testing Results of AOZ in Sample B

The number of samples:10 Each sample test duplicated. unit: ug/kg Test items: AOZ Test method: GB/T 21311-2007

Number	Result 1	Result 2	Average result	Within groups bias 1	Within groups bias 2	Between groups bias	Within groups range
j	x_{1j}	x_{2j}	$\bar{x}_j$	$(x_{1j}-\bar{x}_j)^2$	$(x_{2j}-\bar{x}_j)^2$	$n_j(\bar{x}_j-\bar{\bar{x}})^2$	$(x_{1j}-x_{2j})^2$
1	4.51	3.99	4.250	0.0676	0.0676	0.4238	0.2704
2	3.53	4.28	3.907	0.1391	0.1391	0.0275	0.5565
3	3.80	3.69	3.745	0.0030	0.0030	0.0041	0.0119
4	4.28	3.75	4.015	0.0702	0.0702	0.1015	0.2809
5	2.54	3.16	2.850	0.0961	0.0961	1.7660	0.3844
6	4.78	4.08	4.430	0.1225	0.1225	0.8200	0.4900
7	3.87	3.31	3.590	0.0784	0.0784	0.0797	0.3136
8	3.00	3.97	3.485	0.2352	0.2352	0.1857	0.9409
9	4.18	4.46	4.320	0.0196	0.0196	0.5625	0.0784
10	3.75	2.86	3.305	0.1982	0.1982	0.4693	0.7930
			$\bar{\bar{x}}$	SUM1	SUM2	SUMs	SUMw
		$\bar{x}_i$	3.790	1.029997	1.029997	4.440182	4.119987

One-Way ANOVA

	SS	f	MS	F	F critical value	Confidence probability	STD
Between groups	4.4402	9	0.4934	2.39	3.02	0.95	0.379
Within groups	2.0600	10	0.2060				

Sample Preparation

------Homogeneity (ISO 13528)

Table C.2-5 Homogeneity Testing Results of Sulfaquinoxaline in Sample A

Number of samples:10 Each sample was tested duplicated. unit: ug/kg Test items: Sulfaquinoxaline Test method: GB/T 21311-2007

Number	Result 1	Result 2	Average result	Within groups bias 1	Within groups bias 2	Between groups bias	Within groups range
j	x_{1j}	x_{2j}	$\bar{x}_j$	$(x_{1j}-\bar{x}_j)^2$	$(x_{2j}-\bar{x}_j)^2$	$n_j(\bar{x}_j-\bar{\bar{x}})^2$	$(x_{1j}-x_{2j})^2$
1	968	1061	1014.500	2162.2500	2162.2500	3698.0000	8649.0000
2	892	1040	966.000	5476.0000	5476.0000	60.5000	21904.0000
3	984	957	970.500	182.2500	182.2500	2.0000	729.0000
4	903	998	950.500	2256.2500	2256.2500	882.0000	9025.0000
5	1040	1020	1030.000	100.0000	100.0000	6844.5000	400.0000
6	874	930	902.000	784.0000	784.0000	9660.5000	3136.0000
7	959	989	974.000	225.0000	225.0000	12.5000	900.0000
8	1024	990	1007.000	289.0000	289.0000	2520.5000	1156.0000
9	1021	971	996.000	625.0000	625.0000	1200.5000	2500.0000
10	958	851	904.500	2862.2500	2862.2500	8978.0000	11449.0000
			$\bar{\bar{x}}$	SUM1	SUM2	SUMb	SUMw
		$\bar{\bar{x}}$	971.500	14962.0000	14962.0000	33859.0000	59848.0000

One-Way ANOVA

	SS	f	MS	F	F critical value	Confidence probability	STD
Between groups	33859.00	9	3762.1111	1.26	3.02	0.95	19.618
Within groups	29924.00	10	2992.4000				

Conclusion: The homogeneity of Sulfaquinoxaline in Sample A is acceptable at a 95% Confidence Interval level.

Test:Zhao Xin Date: 2013.10.23	Statistical analysis: Lu Xingan Date: 2013.10.24	Check: Wang Xiujun Date: 2013.10.25

Sample Preparation

------Stability (ISO 13528)

The stability check of ciprofloxacin in sample A

item: ciprofloxacin			σ	17.2	Sample	A		$\bar{\bar{x}}$	1.95	LOG (μg/kg)
DATE	TEMP.	t (day)	x_{1i}	x_{2i}	x_{1i}	x_{2i}	$\bar{x}_i$	$\bar{\bar{x}}_i$	$\delta = \lvert\bar{\bar{x}}_i - \bar{\bar{x}}\rvert$	δ / s *
2013-10-23	25	1	90.5	79.0	1.957	1.898	1.927	1.925	0.025	0.001
			91.9	89.0	1.963	1.949	1.956			
			72.6	83.6	1.861	1.922	1.892			
2013-11-1	25	10	86.2	92.1	1.936	1.964	1.950	1.939	0.011	0.001
			85.2	79.6	1.930	1.901	1.916			
			92.3	86.5	1.965	1.937	1.951			
2013-11-10	25	20	78.3	82.6	1.894	1.917	1.905	1.932	0.018	0.001
			92.1	89.1	1.964	1.950	1.957			
			89.2	82.4	1.950	1.916	1.933			
2013-11-19	25	30	81.2	89.1	1.910	1.950	1.930	1.927	0.023	0.001
			92.1	79.8	1.964	1.902	1.933			
			83.6	82.4	1.922	1.916	1.919			

Test: Zhao Xin Statistical analysis: Lu Xingan Check: Wang Xiujun

Date 11/11/13 Date 11/11/13 Date 12/11/13

Samples Delivery and Analysis

Instructions for Participating Laboratories

Dear <lab name>:

Welcome to participate in *Veterinary Drug Multi-residues in Chicken Proficiency Testing Program (Briefing as PT Program)* is one of the laboratory capacity building event among APEC's MYP *Building Convergence in Food Safety Standards and Regulatory Systems* (M CTI 02 12A) under APEC FSCF and lead by China. The confidentiality is ensured to use the lab code throughout the program. Your lab code is APEC FSCF-MYP-XXX.

To ensure that results from this program can be analyzed properly, participants are asked to adhere carefully to the following instructions.

1. Samples
Two freeze dried chicken samples with weight about 16 gram and different veterinary drug levels sealed in foil bags respectively are sent to each participant.

When receiving the artifacts, please check the packaging and the artifact in the receiving day, and send the "**Sample Receipt Form**" electronically to the coordinator of the PT program linhaoxia_cn@163.com cc hxx@sina.com.

2. Testing Period and Storage Instruction
Testing may commence as soon as samples are received.

Store your samples in the original packaging in room temperature between 15℃ and 25℃.

3. Analytes
The analytes among the following items will be tested according to your nomination in this PT program.

a) 3-amino-2-oxazolidone (AOZ)
b) 5-morpholinomethyl-3-amino-2-oxazolidone (AMOZ)
c) Sulfamethoxazole
d) Sulfadimidine
e) Sulfaquinoxaline
f) ciprofloxacin

4. Testing Procedure
The freeze dried sample should be recovered by adding three times weight of distilled water and stirring

Sample Receipt Form

Institute/ Laboratory: _____

Assigned lab code: _____

Postal address: _____

Contact person: _____ _____ _____
Title Given name Surname

Tel/Fax: _____

E-mail: _____

Print name / Signature: _____

Date: _____

Confirmation of Package Contents

I hereby acknowledge the receipt of the sealed shipping box for the *Veterinary Drug Multi-residues in Chicken Proficiency Testing Program* among MYP *Building Convergence in Food Safety Standards and Regulatory Systems* (M CTI 02 12A). The box contains:

☐ Two samples of freeze dried chicken powder with bags number _____ and _____.

☐ The sample is *Intact & Sealed / Broken / Missing** and should be *Suitable / Not Suitable** for analysis (*Please delete as appropriate).

☐ DECLARATION TO CUSTOMS OFFICIALS AND SHIPPING AGENTS

Other comments: _____

Data Collection

- 26 results reports have been received by deadline.

Results Report Form

Institute/ Laboratory: _____

Assigned lab code: _____

Postal address: _____

Contact person: _____ _____ _____
Title Given name Surname

Tel/Fax: _____

E-mail: _____

Print name / Signature: _____

Date: _____

1. Analytical results

Analytes	Sample No.	Testing results in duplicate (µg/kg) 1	2	Mean value (µg/kg)	Recovery (%)
3-amino-2-oxazolidone (AOZ)	13-C___				
	13-D___				
5-morpholinomethyl-3-amino-2-oxazolidone (AMOZ)	13-C___				
	13-D___				
sulfamethoxazole	13-C___				
	13-D___				

2. Methods of analysis

Analyte: 3-amino-2-oxazolidone (AOZ)

1. * Derivatization: YES / NO
   If yes, please specify the derivatization reagent and the derivatization duration: .....
2. Extraction: Solvent(s): .....
   Technique: .....
   Duration: .....
3. Cleaning up procedure: .....
4. Source(s) of calibration standard(s): .....
5. *Use of internal standard(s): YES (please specify): ..... / NO
6. *Analytical instrument(s): LC-MS / LC-MSMS / LC
   Others (please specify): .....
7. *Column: Normal phase / Reversed phase
   LC column / LC MS column
   Please specify the description, ID (mm), length (mm) and particle size (µm): .....
8. *Correction for recovery: YES (please specify recovery (%)): ..... / NO
9. *Method accreditation: YES / NO
10. Additional information: .....

* Please delete as appropriate

Data Analysis

- The participants' performance were assessed using z-scores calculated by robust statistical method. Median and normalized interquartile range (NIQR) intended to be used as the statistics for z-score calculation.
- The z-scores were calculated by the following formulae. Z-Score = (Result-Median) / NIQR
- As a general rule, any z-score outside the range of -2 to 2 indicates a questionable result/pair of results, while an outlier is any result/pair of results with a z-score outside the range of -3 to 3.

Data Analysis

Table 1 Reported Results of Residue of AOZ

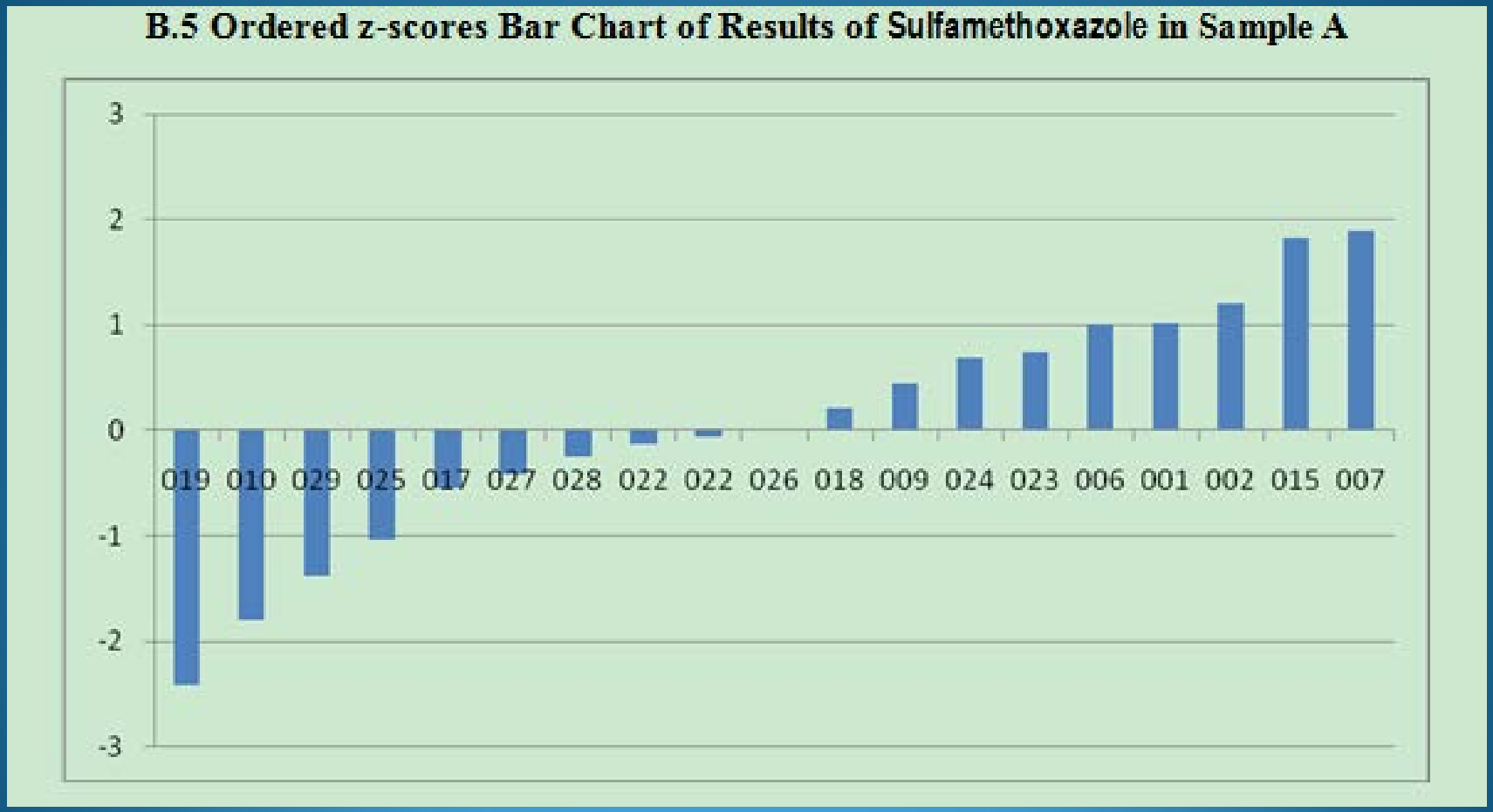
lab code	Sample A					Sample B					Method or instrument
	Testing results in duplicate (μg/kg)		Mean value (μg/kg)	Recovery (%)	Z-Score	Testing results in duplicate (μg/kg)		Mean value (μg/kg)	Recovery (%)	Z-score	
	1	2				1	2				
APEC FSCF-MYP-001	20.5	20.0	20.3	98.3	**6.20**	9.51	9.33	9.42	98.3	**5.42**	LC-MSMS
APEC FSCF-MYP-002	9.000	10.000	9.500	/	**1.61**	5.000	6.000	5.500	/	**1.74**	LC-MSMS
APEC FSCF-MYP-006	7.03	7.02	7.03	100.0	**0.56**	3.86	3.86	3.86	100.0	**0.20**	LC-MSMS
APEC FSCF-MYP-007	3.68	3.43	3.56	97.2	**-0.92**	2.18	2.17	2.18	97.2	**-1.37**	LC-MSMS
APEC FSCF-MYP-009	5.588	5.755	5.652	80.2	**-0.03**	3.207	3.229	3.218	90.0	**-0.40**	LC-MSMS
APEC FSCF-MYP-010	3.19	3.63	3.41	104.82	**-0.98**	2.02	2.40	2.21	104.82	**-1.34**	LC-MSMS
APEC FSCF-MYP-013	2.63	2.59	2.61	/	**-1.32**	1.71	1.81	1.76	/	**-1.77**	LC-MSMS
APEC FSCF-MYP-016	3.20	2.80	3.00	108	**-1.16**	1.80	1.70	1.75	126	**-1.78**	LC-MSMS
APEC FSCF-MYP-017	6.9	5.5	6.2	/	**0.20**	2.8	2.3	2.6	/	**-0.98**	LC-MSMS
APEC FSCF-MYP-018	7.70	7.80	7.80	112	**0.88**		4.70	4.70	112	**0.99**	LC-MSMS
APEC FSCF-MYP-019	2.04	2.01	2.03	40.4	**-1.57**	3.24	2.37	2.81	40.4	**-0.78**	LC-MSMS
APEC FSCF-MYP-021	3.96	3.77	3.87	74.4	**-0.79**	5.46	5.55	5.5	74.4	**1.74**	LC-MSMS
APEC FSCF-MYP-022	5.588	5.600	5.594	<90-110>	**-0.05**	3.544	3.524	3.534	<90-110>	**-0.10**	LC-MSMS
	5.79	5.792	5.791	<90-110>	**0.03**	3.605	3.992	3.799	<90-110>	**0.15**	LC-MSMS
APEC FSCF-MYP-025	6.59	6.57	6.58	99	**0.37**	3.76	3.92	3.84	99	**0.19**	LC-MSMS
APEC FSCF-MYP-026	5.4	5.2	5.3	88	**-0.18**	3.1	3.0	3.0	88	**-0.60**	LC-MSMS
APEC FSCF-MYP-027	7.52	7.29	7.40	107	**0.71**	4.21	4.14	4.18	107	**0.50**	LC-MSMS
APEC FSCF-MYP-028	0.80	0.74	0.77	7.8	**-2.11**	0.51	0.47	0.49	8.2	**-2.96**	LC-MSMS
APEC FSCF-MYP-029	6.013	5.999	6.006	91.1	**0.12**	3.799	3.702	3.7505	91.1	**0.10**	LC-MSMS
APEC FSCF-MYP-030	5.87	5.88	5.88	97.6	**0.07**	3.70	3.79	3.75	97.6	**0.10**	LC-MSMS

Data Analysis

Table 2 Reported Results of Residue of AMOZ

lab code	Sample A					Sample B					Method or instrument
	Testing results in duplicate (μg/kg)		Mean value (μg/kg)	Recovery (%)	Z-Score	Testing results in duplicate (μg/kg)		Mean value (μg/kg)	Recovery (%)	Z-score	
	1	2				1	2				
APEC FSCF-MYP-002	1.900	2.100	2.000	/	**1.02**	0.900	1.100	1.000	/	**0.98**	LC-MSMS
APEC FSCF-MYP-006	2.00	2.00	2.00	101.0	**1.02**	1.00	1.00	1.00	101.0	**0.98**	LC-MSMS
APEC FSCF-MYP-007	1.13	1.04	1.08	88.0	**-0.83**	0.46	0.51	0.485	88.0	**-0.94**	LC-MSMS
APEC FSCF-MYP-009	1.745		1.745	85.60	**0.51**	0.885	0.852	0.868	87.80	**0.49**	LC-MSMS
APEC FSCF-MYP-010	1.26	1.18	1.22	108.58	**-0.55**	0.53	0.73	0.63	108.58	**-0.40**	LC-MSMS
APEC FSCF-MYP-013	0.750	0.860	0.805	/	**-1.38**	0.385	0.409	0.397	/	**-1.27**	LC-MSMS
APEC FSCF-MYP-016	0.5	0.6	0.55	108	**-1.90**	<0.5	<0.5	<0.5	126	/	LC-MSMS
APEC FSCF-MYP-017	2.0	1.6	1.8	/	**0.62**	0.59	0.51	0.55	/	**-0.70**	LC-MSMS
APEC FSCF-MYP-018	2.10	2.00	2.10	99	**1.22**		0.99	0.99	99	**0.94**	LC-MSMS
APEC FSCF-MYP-019	1.02	0.895	0.956	45	**-1.08**	0.78	0.676	0.729	45	**-0.03**	LC-MSMS
APEC FSCF-MYP-021	<1	<1	<1	108.000	/	1.48	1.58	1.52	108	2.92	LC-MSMS
APEC FSCF-MYP-022	1.484	1.539	1.512	<90-110>	**0.04**	1.018	0.997	1.008	<90-110>	**1.01**	LC-MSMS
	1.508	1.486	1.497	<90-110>	**0.01**	1.017	1.078	1.048	<90-110>	**1.16**	LC-MSMS
APEC FSCF-MYP-025	1.50	1.48	1.49	80	**-0.01**	0.67	0.66	0.67	80	**-0.25**	LC-MSMS
APEC FSCF-MYP-026	1.8	2.0	1.9	100	**0.82**	0.7	0.8	0.8	100	**0.23**	LC-MSMS
APEC FSCF-MYP-027	1.63	1.64	1.64	104	**0.29**	0.637	0.669	0.653	104	**-0.31**	LC-MSMS
APEC FSCF-MYP-028	0.19	0.17	0.18	11.1	-2.64	0.14	0.14	0.14	0	-2.23	LC-MSMS
APEC FSCF-MYP-029	1.501	1.470	1.485	100.0	**-0.02**	0.743	0.748	0.7455	100.0	**0.03**	LC-MSMS
APEC FSCF-MYP-030	1.36	1.34	1.35	96.8	**-0.29**	0.68	0.74	0.71	96.8	**-0.10**	LC-MSMS

Data Analysis



Interim Report

CNAS
CNAS PT0026

APEC MYP (M CTI 02 12A)
Veterinary Drug Multi-residues in Chicken Proficiency Testing Program
INTERIM REPORT

Date of Issue : June 29, 2014

In this APEC proficiency testing program, veterinary drug multi-residues including 3-amino-2-oxazolidone (AOZ), 5-morpholinomethyl-3-amino-2-oxazolidone (AMOZ), sulfamethoxazole, sulfadimidine, sulfaquinoxaline, and ciprofloxacin in chicken muscle samples were tested.

Two gross samples A and B were prepared and divided respectively to testing samples A and B, then every two testing samples A and B were distributed to the participating laboratories.

The results for the tests on AOZ, AMOZ, sulfamethoxazole, sulfadimidine, sulfaquinoxaline, and ciprofloxacin residues reported from the participating laboratories are listed in Table 1.

The statistics given in Table were calculated by robust statistical methods based on the results listed submitted by participants. The z-scores can be calculated by the following formulae: Z-Score = (Result-Median) / NIQR.

As a general rule, any z-score outside the range of -2 to 2 indicates a questionable result/pair of results, while an outlier is any result/pair of results with a z-score outside the range of -3 to 3.

Please confirm this interim report NO LATER THAN JULY 15, 2014 with your lab's address, Lab Code No. in this program, email, phone and fax number, we will use them for sending the final report. Thanks for your cooperation.

If you have any question on any aspect of your test, or on this PT program, please feel free to contact the persons listed below.

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Table 1 Reported Results of Residue of AOZ

lab code	Sample A					Sample B					Method or instrument
	Testing results in duplicate (μg/kg)		Mean value (μg/kg)	Recovery (%)	Z-Score	Testing results in duplicate (μg/kg)		Mean value (μg/kg)	Recovery (%)	Z-score	
	1	2				1	2				
APECFSCF-MYP-001	20.5	20.0	20.3	98.3	6.20	9.51	9.33	9.42	98.3	5.42	LC-MSMS
APECFSCF-MYP-002	9.000	10.000	9.500	/	1.61	5.000	6.000	5.500	/	1.74	LC-MSMS
APECFSCF-MYP-006	7.03	7.02	7.03	100.0	0.56	3.86	3.86	3.86	100.0	0.20	LC-MSMS
APECFSCF-MYP-007	3.68	3.43	3.56	97.2	-0.92	2.18	2.17	2.18	97.2	-1.37	LC-MSMS
APECFSCF-MYP-009	5.588	5.755	5.652	80.2	-0.03	3.207	3.229	3.218	90.0	-0.40	LC-MSMS

Table 2 Reported Results of Residue of AMOZ

lab code	Sample A					Sample B					Method or instrument
	Testing results in duplicate (μg/kg)		Mean value (μg/kg)	Recovery (%)	Z-Score	Testing results in duplicate (μg/kg)		Mean value (μg/kg)	Recovery (%)	Z-score	
	1	2				1	2				
APECFSCF-MYP-002	1.900	2.100	2.000	/	1.02	0.900	1.100	1.000	/	0.98	LC-MSMS
APECFSCF-MYP-006	2.00	2.00	2.00	101.0	1.02	1.00	1.00	1.00	101.0	0.98	LC-MSMS
APECFSCF-MYP-007	1.13	1.04	1.08	88.0	-0.83	0.46	0.51	0.485	88.0	-0.94	LC-MSMS
APECFSCF-MYP-009	1.745		1.745	85.60	0.51	0.885	0.852	0.868	87.80	0.49	LC-MSMS
APECFSCF-MYP-010	1.26	1.18	1.22	108.58	-0.55	0.53	0.73	0.63	108.58	-0.40	LC-MSMS
APECFSCF-MYP-013	0.750	0.860	0.805	/	-1.38	0.385	0.409	0.397	/	-1.27	LC-MSMS
APECFSCF-MYP-016	0.5	0.6	0.55	108	-1.90	<0.5	<0.5	<0.5	126	/	LC-MSMS
APECFSCF-MYP-017	2.0	1.6	1.8	/	0.62	0.59	0.51	0.55	/	-0.70	LC-MSMS
APECFSCF-MYP-018	2.10	2.00	2.10	99	1.22		0.99	0.99	99	0.94	LC-MSMS
APECFSCF-MYP-019	1.02	0.895	0.956	45	-1.08	0.78	0.676	0.729	45	-0.03	LC-MSMS
APECFSCF-MYP-021	<1	<1	<1	108.000	/	1.48	1.58	1.52	108	2.92	LC-MSMS
APECFSCF-MYP-022	1.484	1.539	1.512	<90-110>	0.04	1.018	0.997	1.008	<90-110>	1.01	LC-MSMS
	1.508	1.486	1.497	<90-110>	0.01	1.017	1.078	1.048	<90-110>	1.16	LC-MSMS
APECFSCF-MYP-025	1.50	1.48	1.49	80	-0.01	0.67	0.66	0.67	80	-0.25	LC-MSMS
APECFSCF-MYP-026	1.8	2.0	1.9	100	0.82	0.7	0.8	0.8	100	0.23	LC-MSMS
APECFSCF-MYP-027	1.63	1.64	1.64	104	0.29	0.637	0.669	0.653	104	-0.31	LC-MSMS
APECFSCF-MYP-028	0.19	0.17	0.18	11.1	-2.64	0.14	0.14	0.14	0	-2.23	LC-MSMS
APECFSCF-MYP-029	1.501	1.470	1.485	100.0	-0.02	0.743	0.748	0.7455	100.0	0.03	LC-MSMS
APECFSCF-MYP-030	1.36	1.34	1.35	96.8	-0.29	0.68	0.74	0.71	96.8	-0.10	LC-MSMS

Item	Sample		Total Results	Satisfactory results	Questionable results	Unsatisfactory results
AOZ	Sample A	Number	20	18	1	1
		Percent(%)		95	5	5
	Sample B	Number	20	18	1	1
		Percent(%)		95	5	5
AMOZ	Sample A	Number	19	17	2	0
		Percent(%)		90	10	0
	Sample B	Number	19	17	2	0
		Percent(%)		90	10	0
Sulfamethoxazole	Sample A	Number	19	18	1	0
		Percent(%)		95	5	0
	Sample B	Number	19	18	0	1
		Percent(%)		95	0	5
Sulfadimidine	Sample A	Number	16	12	3	1
		Percent(%)		75	19	6
	Sample B	Number	16	13	1	2
		Percent(%)		82	6	12
Sulfaquinoxaline	Sample A	Number	19	15	4	0
		Percent(%)		79	21	0
	Sample B	Number	19	16	2	1
		Percent(%)		84	11	5
Ciprofloxacin	Sample A	Number	23	20	2	1
		Percent(%)		87	9	4
	Sample B	Number	23	16	3	4
		Percent(%)		70	13	17

Data Analysis Summary

Item	Sample	MEDIAN (mg/kg)	NIQR (mg/kg)	CV(%)
AOZ	A	5.72	2.35	41
	B	3.64	1.07	29
AMOZ	A	1.49	0.498	33
	B	0.737	0.268	36
sulfamethoxazole	A	28	5.78	21
	B	143	28.0	20
sulfadimidine	A	36.2	7.19	20
	B	123	27.9	23
sulfaquinoxaline	A	989	326	33
	B	216	57.4	27
ciprofloxacin	A	82.4	20.1	24
	B	430	53.8	13

Clarification and Conclusion

Item	Sample	MEDIAN' (mg/kg)	NIQR' (mg/kg)	CV'(%)	CV(%)
AOZ	A	6.15	1.76	29	41
	B	3.86	1.26	33	29
AMOZ	A	1.79	0.468	26	33
	B	0.99	0.261	26	36
sulfamethoxazole	A	30.6	5.60	18	21
	B	147	16.7	6	20
sulfadimidine	A	34.8	4.67	13	20
	B	128	16.7	13	23
sulfaquinoxaline	A	1024	278	27	33
	B	245	38.3	16	27
ciprofloxacin	A	95.2	17.2	18	24
	B	484	48.6	10	13

Technical Commentary

- Extracting method employed;
- Cleaning-up method employed;
- Recovery;
- Using internal standards;
- Following the INSTRUCTION for PARTICIPATING LABORATORY.

Thank you!

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