



**Asia-Pacific  
Economic Cooperation**

---

**2014/SCSC/WKSP2/007**

Session: 5.5

## **Experience Share for Veterinary Drug Multi-residues Analysis in Indonesia**

Submitted by: Indonesia



**Food Safety Cooperation Forum Partnership  
Training Institute Network Proficiency Testing  
Workshop  
Beijing, China  
10-11 September 2014**



# EXPERIENCE SHARE FOR VETERINARY DRUG MULTI-RESIDUES ANALYSIS

**Raphaella Widiastuti**

**Indonesian Research Center for Veterinary Science (ICRVS)  
Balai Besar Penelitian Veteriner**



Badan Penelitian dan Pengembangan Pertanian  
Kementerian Pertanian

SCIENCE. INNOVATION. NETWORKS  
[www.litbang.deptan.go.id](http://www.litbang.deptan.go.id)



# ICRVS in brief



Established : 1908

Location : Bogor (West Java Province),  $\pm$  60 km southern of Jakarta

5 departments : Toxicology, Bacteriology, Virology, Parasitology and Pathology

Accreditation : a. ISO/IEC 17025 : 2008

b. ISO 9001 : 2010

c. ISO 17043 : 2010 (has been accessed August 2014)



# Vision - BBALITVET

---

**“Becoming an international veterinary research institute in producing veterinary science and technologies by utilizing local resources to support animal and veterinary public health to be a sustainable industrial agriculture”**



# Mission

1. Conduct exploration, characterization, conservation and utilization of potential veterinary germ plasma resources for the development of vet. science and technologies
2. Produce veterinary science and innovations in line with the needs of users to be sustainable and superior industrial agriculture
3. Conduct diagnostic services for animal health, veterinary, public health and food safety of animal origin according to the national and international standard as a reference laboratory
4. Improve the network on veterinary research collaboration and science and technology development with other research institute, related to institutions and users both nationally and internationally
5. Improve the quality, capacity and capability of research resources to produce science and technologies based on national and international references.



# Toxicology Dept. in brief

Do research and public services for :

1. Veterinary drug residues (accredited for TCs)
2. Mycotoxins (accredited for aflatoxins)
3. Pesticides (accredited for lindane)
4. Heavy Metals (accredited for Pb, Cd, Cu, Zn, Ca & Mg)
5. Poisoning cases



## The involvement in PTs program

1. In 2005 involved PT from APLAC for enrofloxacin determination in 5 chicken powder.

The results showed 2 out of 5 samples were in layer (z score -0.39 and 1.35) but the rest were out layer (z score  $\geq 9.07$ )

2. In 2013 involved PT from APEC for ciprofloxacin determination in 2 chicken powder.

Samples received in 2 Dec 2013 and reports sent in 13 December 2013



# Cronology

- 19 April 2013 : received forwarded email from National Standardization Agency of Indonesia (BSN) to participate in Veterinary Drug Multi-residues in Chicken Proficiency Testing Program
- 25 April 2013 : replied of involvement and the Indonesian Food Safety Network (JKPN) asked for another possibility for other PT (nitrofurans metabolites, sulfonamide drugs)
- May 2013 : sought for extraction and detection method
- 25 May 2013 : received ciprofloxacin standard (Fluka)
- June 2013 : run validation series
- August 2013 : lab renovation
- October 2013 : lab moved





- 2 Dec 2013 : samples received in ICRVS
- 11 Dec 2013 : accident due to electricity shocked, Hitachi HPLC broken changed to Waters HPLC
- 13 Dec 2013 : sent the reports
- 16 Dec 2013 : notification of sample reports received
- 23 Dec 2013 : notification from Dr. Hanxia about workshop in Beijing in Sept 2014
- 22 June 2014 : asked for PT results to Dr. Hanxia
- 5 August 2014 : changed participant from Dr. Andriani to myself
- 10 August 2014 : acceptance from Dr. Hanxia



# The reason of choosing ciprofloxacin PT

1. Done researches and involved in PT of EFX in last 10 years
2. Could be detected both by UV or FL detector
3. Easy to find the standard
4. The analysis for multi drug residues has not been done yet.



# Operational conditions of ciprofloxacin PT

## 1. Extraction (duplo)

- a. Method : A-K. Wihlborg, O. Shimelis and A. Trinh. 2009.  
The Selective Extraction of Fluoroquinolones in Veterinary Samples using Molecularly Imprinted Polymer SPE. Sigma Aldrich Technical Report. T408177. Sigma-Aldrich Co.
- b. Solvents : 30 mL 50 mM NaH<sub>2</sub>PO<sub>4</sub>, pH 7.4
- c. Recovery : 68.90 %



## b. Cleaning up

Loaded the extracted sample into a Bond Elut C<sub>18</sub> SPE cartridge (Agilent Technologies) conditioned with methanol, DI water and phosphate buffer pH 7.4.

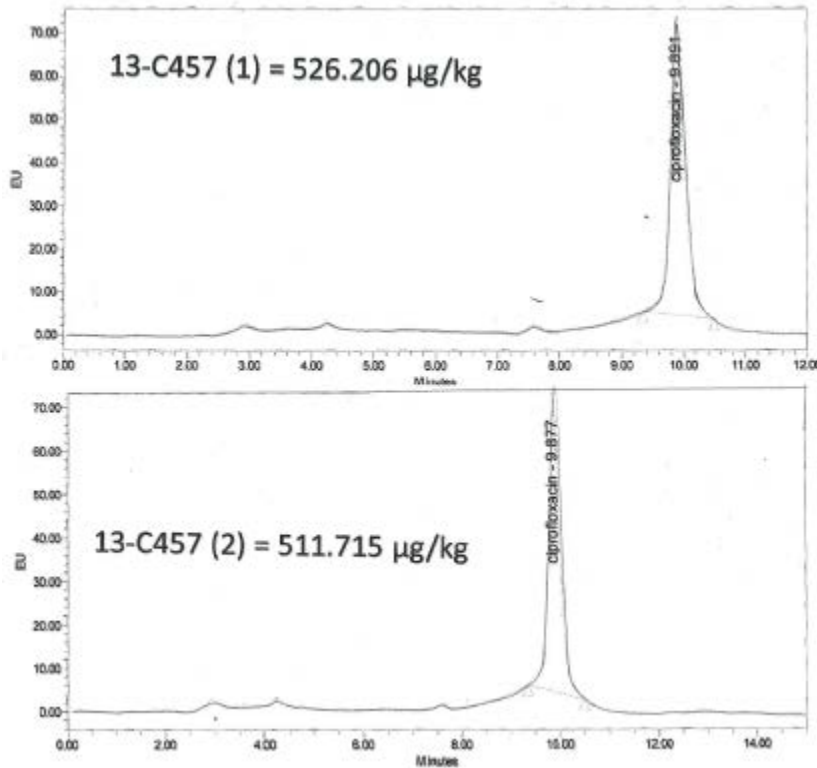
Cleaned-up the SPE C<sub>18</sub> with DI water, acetonitrile, 0.5% acetic acid, 0.1% ammonia in DI water and hexane. The sample was then eluted with 2% ammonia in methanol.



- Standard : Fluka, Vetranal (Sigma Aldrich)
- Instrumentation : Waters Alliance e2695 (Waters Corp.). Detected with a 2475 Multi Fluorescence (FLR) detector at excitation wavelength 300 nm and emission wavelength 458nm.
- Column : Reversed phase LC column : Shimpack VP-ODS C<sub>18</sub> (Shimadzu Corp.) 4.6 mm (ID) x 250 mm (L) and 5µm (particle size)
- Mobile phase : 0.02 M trichloroacetic acid, methanol and acetonirile (74:4:22) at flow rate of 1.0 ml /min



# Chromatogram of sample 13-C457



# Chromatogram of sample 13-D629

