ADVANCED REGIONAL TRAINING COURSE ON ISOTOPIC CONFIRMATORY TECHNIQUES FOR RESIDUES/CONTAMINANTS IN FOOD PRODUCTS

LOCATION: DOHA, QATAR
DATE: 27 TO 31 OCTOBER 2024

Presented By :

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19 Mac 2025

PENGENALAN KURSUS

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Ministry of Public Health
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IAEA
International Atomic Energy Agency

International Atomic Energy Agency (IAEA) menganjurkan acara di bawah projek kerjasama teknikal IAEA RAS5096, “Strengthening Multi-Stakeholder Food Safety Monitoring Programmes for Chemical Contaminants and Residues in Plant and Animal Products Using Nuclear/Isotopic Techniques” antara intipati kursus yang dijalankan ialah:

01 Radio-Receptor Assay Technique

02 Veterinary drug residue sample in screening and confirmatory analysis

03 Sulfonamides in chicken & honey (screening)

04 Chlorimphenicol in honey (screening)

05 Validation of veterinary drug residue screening test

05 Pesticide residue in fruits and vegetables

06 Pesticide residues analysis using mass spectrometry techniques (LC-MS/MS & GC-MS/MS)

07 Mycotoxins-M1 in milik (screening)

08 Pesticide residues national monitoring programs and regulatory framework

09 Method validation for testing laboratories & Analytical Quality Control (AQC) for pesticide residues analysis in food products

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MATLAMAT KURSUS

01

Pemantauan & Pengesanan

- Analisis sisa pencemaran bahan kimia dalam makanan dengan LC-MS/MS & GC-MS/MS.
- Radioreceptor Assay (RRA): Principles and practical applications for residue monitoring in food matrices. (CHARM II)

02

Penyediaan Sampel:

- Teknik untuk saringan dan pengesahan residu ubat veterinar.
- Analisis Residu Pestisid: Penggunaanmass spectrometry (LC-MS/MS & GC-MS/MS) untuk pengesanan yang tepat.

03

Pengurusan Kualiti Data dan Analisis:

- Teknik untuk memastikan kualiti data melalui penyediaan sampel yang betul, tafsiran data, dan pengesahan keputusan analisis.

Operator's Manual:

Charm® II Aflatoxin Test for Milk at 0.025 µg/kg

Quantitative Assay

CHARM SCIENCES INC

Kit Information

Introduction

The Charm II Aflatoxin Test is a rapid immunoreceptor assay. The Quantitative Assay is the most sensitive format detecting aflatoxin M1 at 0.025 µg/kg in raw commingled, pasteurized, homogenized, skim, and powdered cow milk. This test is designed for use by dairy, veterinary, laboratory, field and regulatory personnel.

Kit Contents and Materials Needed

Supplied with Kits	Disposables	Equipment
• CATM Tablet Reagents • Zero Control for Aflatoxin (Quantitative Assay) • Positive Control Standard • Solution AF • Typical Counts Sheet • Operator's Manual	• Pipet Tips • Glass Test Tubes • Corks (Caps) For Test Tubes • Cotton Swabs	• Pipets • Vortex Mixer • Charm II Inctronic Incubator • Centrifuge

Charm II equipment available only from Cha

For details and ordering information see Ord

Sensitivity and Selectivity

Aflatoxin-free raw milk yields negati

Table 1: Concentration of aflatoxin

AFLATOXIN
Aflatoxin M1

*µg/kg or ppb (parts per billion)

† Positive at least 90% of the time.

Interferences and Cross Re

There are no known interferences fr aminoglycosides, amphenicols, beta tetracyclines, sulfa drugs/sulfonami ivermectin, novobiocin, oxytocin, PA

There are no interferences from son

Storage

Store the tablet reagents at -15°C c or 0 to 4.5°C for US certified labs). **and Storage** for details.

Work instruction

Qualitative analysis of CAP in Honey using Charm (Rapid radio-receptor assay)

Preparation for reagents: should be given according to instructions mentioned on the bottles

1- **MSU-AMS:** reconstitute with 10ml DIW.
2- **Negative control (NC):** reconstitute with 30ml DIW.
3- **positive control (PC):** add 100 µl to 13.2ml NC to get 10µg/L CAP.

Sample preparation:

1- Weight 5gm sample (Blank, sample fortified with 1ml MSU-AMS) in 50 ml centrifuge tubes, dissolve the samples with 15ml DIW, Centrifuge if needed.

Test tubes preparation:

1- Prepare test tubes for NC, PC, Blank sample, samples fortified with the standard and routine samples.
2- Add an ion exchange resin tablet (grey) to each tube.
3- Add 5ml from each sample, centrifuge
4- Prepare new test tubes, add the binder (pink tablet) and dissolve in 300µL DIW.
5- Pour off the supernatant to new test tubes, vortex and incubate them at 50ċ for 6min.
6- Add green tablet, vortex and incubate at 50ċ for 3min. Then add black tablet and centrifuge for 5min at 5000rpm 0r 10min at 4000rpm.
7- Pour off supernatant to waste and dissolve residues in 300µL DIW.
8- Add 3ml Opti-flour (scintillation fluid) and vortex.
9- Count in the analyzer (count per minute, CPM).
10- If the sample result ≤ the control point (initial positive) and should be retested.

Calculations:

A- Control point determination:
1- Run 6 replicates of NC, determine the NC average (each measurement should be ±15% of the average).
2- Subtract 30% from the NC average to calculate the control point.
3- Control point= the NC average of fortified sample - 30%.
4- Run 3 replicates of PC, determine the PC average.

B- Verification of control point:
1- Test one NC and one PC.
2- Test blank sample (SDF) and positive sample.
3- Results (NC must be ±20% of NC average, PC must be < control point).

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Food Laboratories Section

Analysis of Chloramphenicol in Honey

Title: Determination of Chloramphenicol Residues in Honey samples by Quick and Easy Method (QuEChERS) using Liquid Chromatography / Tandem Mass Spectrometry (LC-MS/MS)

Name of Trainee: ____________________ **Group:** ____________

I. Materials Required

1) PTFE or polytetrafluoroethylene 15 ml and 50 ml screw cap tubes.
2) Centrifuge
3) LC-MS/MS Sciex triple quad 6500 with LC column (100 mm, 2.1 mm a
4) Analytical balance, capable of weighing to 0.01 g
5) Sonicator
6) Turbo vap , biotage
7) Disposable syringe
8) Syringe filters (PVDF, 0.2 µm)
9) Vortex mixer
10) Polypropylene centrifuge tubes with caps
11) Injection vials, 1.5 ml, suitable for LC, if necessary, with micro-inserts.
12) Screw capped glass vials, 20 ml, for storage of excessive amounts of fin
13) Methanol MS grade or equivalent (purity ≥ 99.9 %).
14) Certified reference standards
15) QuEChERS extraction kits
16) Dispersive solid phase lipid removal clean up tube

II. Procedure

Sample Preparation:

1) Weigh accurately__________ (2 ± 0.04 g) of the sample (Real/Spik (QuEChERS Extract tubes.
2) Add _____(8 ml) of Deionised water and shake for_____ (1 min).
3) Add 10 ml extraction solvent (1% acetic acid in acetonoitrile) shake min).
4) Add quenchers powder and shake well for 1 min
5) Centrifuge at _____(4700 RPM) for _____(10 min).
6) Transfer the upper clear layer to another centrifuge 50ml PTFE tube (2
7) Add another 10 ml extraction solvent (1% acetic acid in acetonoitril extraction and centrifugate at 4700 rpm for 10 min .
8) Transfer the upper clear layer to 2nd tube.
9) Transfer only 7 ml from extractant into dispersive solid phase extract sorbent .
10) Transfer 4 ml extractant from SPD to glass tube, evaporate at 40degre Turbovap.
11) The dry residue were dissolved in 800 µl (10% methanol, 0.1% formi

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Worksheet for Pesticide Residue analysis

Determination of Pesticides Residues in Fruits and Vegetables samples by Quick and Easy Method (QuEChERS) using Gas Chromatography / Tandem Mass Spectrometry (GC-MS/MS) and Liquid Chromatography / Tandem Mass Spectrometry (LC-MS/MS)

Name of Trainee: [illegible] **Group:** 3

pricky pear

I. Materials Required

1) PTFE or polytetrafluoroethylene 15 ml and 50 ml screw cap tubes.
2) Centrifuge
3) GC–MS/MS Agilent triple quad 7000 B with HP-5 MS capillary column (15 m,250 µm id, 0.25 µm film thickness)
4) LC-MS/MS Sciex triple quad 5500 with LC column (100 mm, 2.1 mm and 3µ pariclesize)
5) Analytical balance, capable of weighing to 0.01 g
6) Sonicator
7) Disposable syringe
8) Syringe filters (PVDF, 0.2 µm)
9) Polypropylene centrifuge tubes with caps
10) Injection vials, 1.5 ml, suitable for LC and GC autosampler, if necessary, with micro-inserts.
11) Screw capped glass vials, 20 ml, for storage of excessive amounts of final extracts, if necessary.
12) Acetonitrile MS grade or equivalent (purity ≥ 99.9 %).
13) Certified active ingredient standards or Readymade pesticide standard solutions from AccuStandard, Inc- USA or equivalent.
14) QuEChERS extraction kits containing (Sodium citrate 1 g, sodium Hydrogen citrate Sesquihydrate 0.5 g, Magnesium sulfate 4 g and Sodium Chloride 1 g), Agilent or equivalent.
15) Dispersive solid phase extraction tube containing sorbent (150 mg primary secondary amine (PSA) and 900 mg anhydrous magnesium sulphate), Agilent or equivalent.

II. Procedure

Sample Preparation:

500ul of 1ppm std.

1) Weigh accurately 9.9448 g (10 ± 0.1 g) of the sample (Real/Spiked) into 50 ml PTFE tube (QuEChERS Extract tubes.
2) Add 10 (10 ml) of Acetonitrile and shake for 1 (1 min).—using vortexer
3) Add QuEChERS mixture and shake vigorously for 1-2 (1-2 min).
4) Centrifuge at 4700 (4700 RPM) for 10 (10 min).
5) Transfer the upper clear layer 2.7ml (2 to 3ml) into dispersive solid phase extraction tube containing sorbent (Primary secondary amines and magnesium sulfate).—vortexer
6) Mix the extract with sorbent vigorously for 1 (1 min) and then centrifuge at 4700 (4700 RPM) for 5 (5 min).
7) Filter the extract through 0.2 µm nylon syringe filter and transfer to GC injection vial. Inject into GC-MS/MS.
8) For LC-MS/MS taken 200ul (200 µl) of filtrate to _____(800 µl) of LC diluent in 1 ml HPLC vial, mix and inject.

ISI KANDUNGAN KURSUS YANG MEMBERI FAEDAH KEPADA DVS

Selain latihan secara praktikal (hands-on), turut diadakan kelas teori mengenai:

01 Pesticide residues national monitoring programs and regulatory framework

02 Analytical Quality Control (AQC) for pesticide residues analysis in food products

03 Method validation for testing laboratories

IMPORTANCE OF PESTICIDE RESIDUE MONITORING

- To manage the Chemical Residues and Environment contaminants.
- To Strengthen consumers confidence.
- Facilitate access to domestic and international market.
- Assist Government to identify potential residue problems including failure to comply with Good Agricultural Practices.

Example of Available MRLs Database

Codex	Codex Alimentarius http://www.fao.org/fao-who-codexalimentarius/codex-texts/dbs/pestres/pesticidedetail/en/?p_i
European	European Union https://ec.europa.eu/food/plants/pesticides/maximum-residue-levels_fr
USA	United States http://www.mrldatabase.com/ https://www.fas.usda.gov/maximum-residue-limits-mrl-database
Canada	Canada canada.ca/en/health-canada/services/consumer-product-safety/pesticides-pest-management/public/protecting-your-health-environment/pesticides-food/maximumresidue-limits-pesticides
China	The National Food Safety Standards System, China https://www.fas.usda.gov https://cdn.ymaws.com/ifu-fruitjuice.com/resource/resmgr/pesticides/2020/China_Releases_Draft_Standar.pdf
Japan	Ministry of Health, Labour and Welfare (MHLW), Japan https://db.fcr.or.jp/front/pesticide_comp, the japan Food Chemical Research Foundation (English
India	Food Safety and Standards Authority of India (FSSAI) http://ppqs.gov.in/sites/default/fles/1._major_uses_of_pesticides_-_insecticide_as_ on_30.06.2021.pdf

Conclusions and Recommendations

Strengthen regional regulations (QSO, GSO etc.) with periodic rigorous review & Create a dynamic MRL database similar to CODEX & EU online database.

Collaborative risk assessment and knowledge sharing with respect to scientific studies dealing with impact of pesticides on public health. Notify CODEX about challenges related to MRLs.

Way Forward

Refer other available international legislations such as EU, US , India, Brazil, Australia MRLs

Improve sampling efficiency at Ports & Conduct retesting for suspected and / borderline rejections. Utilize measurement uncertainties effectively in decision rules.

CADANGAN PENAMBAHBAIKAN

Dari hasil kursus ke atas perkhidmatan jabatan:

01

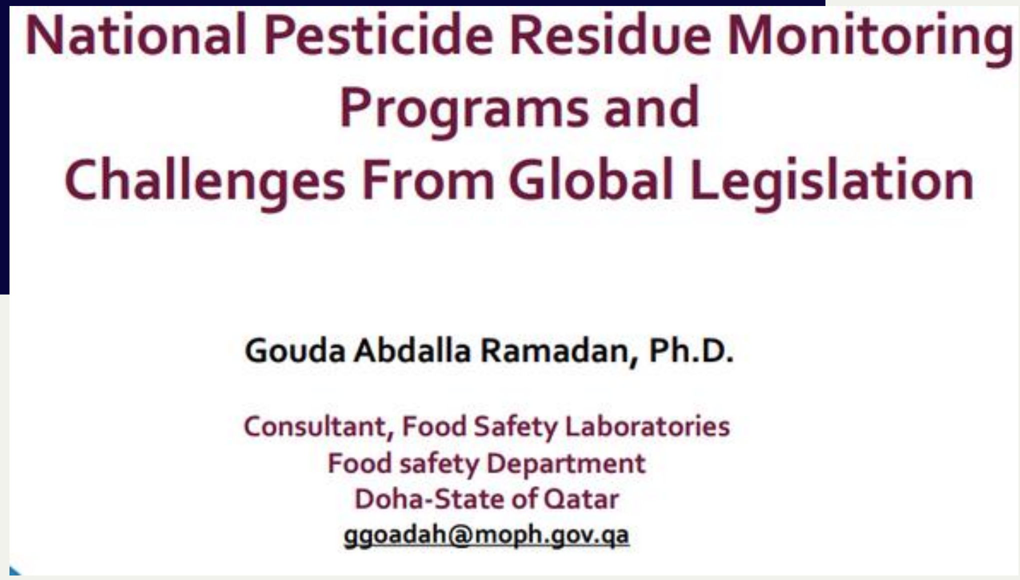
Meningkatkan keberkesanan penyelenggaraan peralatan makmal melalui peruntukan kos dan servis berkala.

02

Rekabentuk makmal

03

Mewujudkan jawatan konsultan bagi ujian analitikal kimia



PENUTUP

kebaikan yang di perolehi dari kursus ini:

Dapat mengeratkan hubungan dan kerjasama dengan negara Asia-Pasifik

Mendapat pendedahan tentang cara negara lain mengurus pematuhan peraturan, penilaian risiko, dan amalan terbaik di makmal

Jaringan Profesional yang Lebih Luas

Sekian,

TERIMA KASIH