



**Government Chemist**

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Conference 2023

**Safe food for tomorrow's  
world - food security in  
challenging global  
conditions**



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**Review of Referee  
Cases**

**20 June 2023**



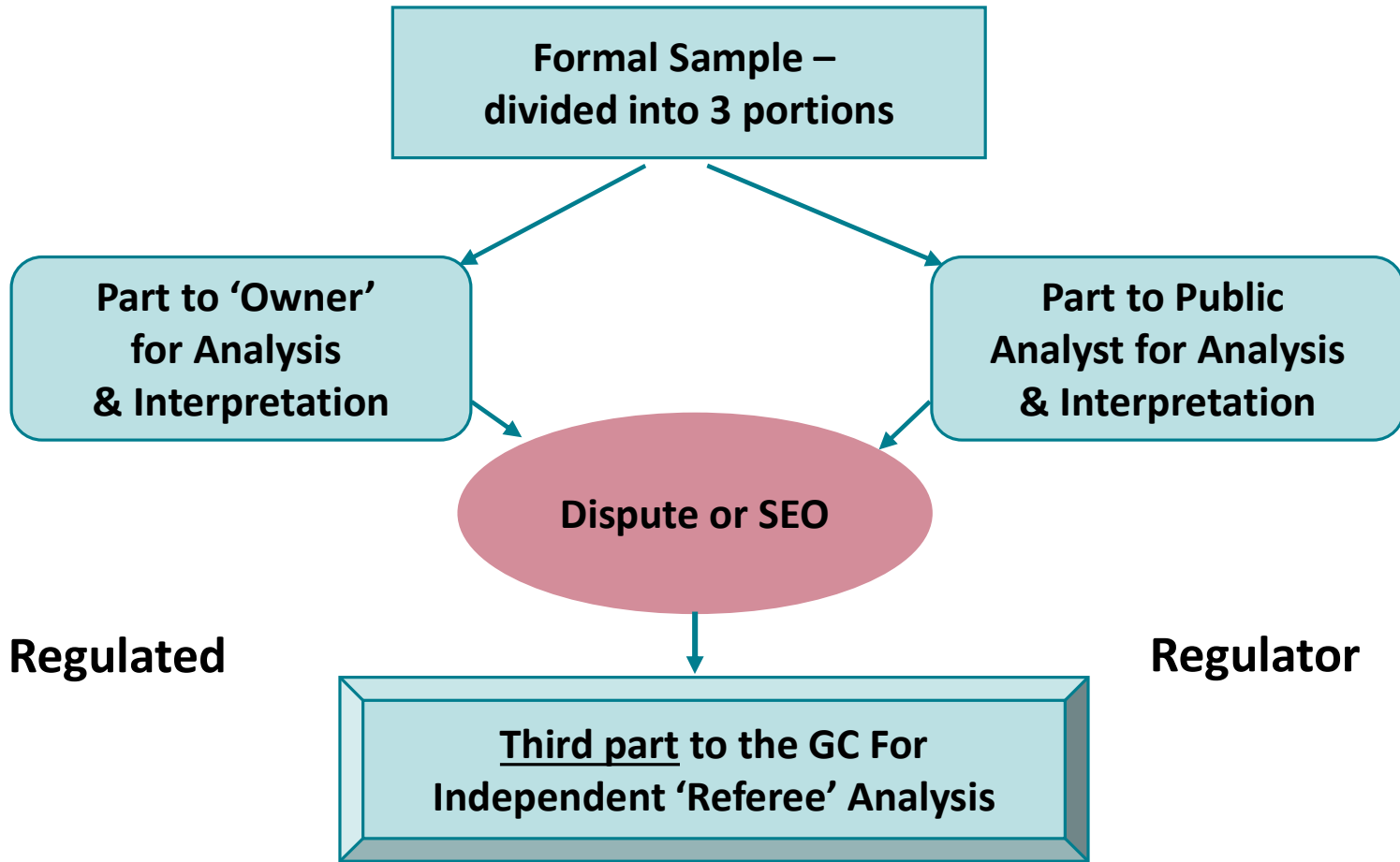
# Review of referee cases



- **Introduction**
  - How a case arises
  - Steps in a case
- **Cases**
- **Summary**



# How a case arises:



# Typical steps in a referee case



- **Decision to accept (is there a dispute?)**
- **Funding**
- **Schedule work**
- **Check legislation**
- **Identify appropriate methodology**
- **Method trialled**
- **Experimental design:**
  - Replicates 3 x 3
  - CRM's, RM's, spikes
  - Witnessed
- **More than one technique**
- **Transcriptions checked**
- **Results reviewed**
  - Interpretation
  - Statistical analysis
- **More analysis?**
- **Certificate**
  - Initial draft
  - Reviewed and independently checked
  - Issued to all parties

# Case resource



# Cases



- **Opinion on chemical form of a food supplement.**
- **GMO in rice products from China.**
- **PAA's in nylon kitchenware.**
- **Nitrofurans in prawns.**
- **Mycotoxins.**
- **Pesticides.**

# Food Supplement



- **Scientific opinion on chemical form of a food supplement.**
- **Requested by ASA following complaint regarding claims on supplement label.**
- **Critical assessment of evidence provided by both sides.**

# GMO's in rice products from China



- **China (Restriction on First Placing on the Market) (England) Regulations 2008**
  - Implement in England Commission Implementing Decision 2011/884/EU
- **Define specified rice products**
- **Permit the placing on the market such products only if they are compliant with EU law**
- **Non-compliant if a genetically modified element is detectable**
  - Target CaMV 35S, t-NOS and Cry 1Ab/Ac
- **Specified methods of analysis supported by EURL guidance**





# Typical analysis plan for a rice product

- **10 retail packs (250g each) received, each with 3 bundles of noodles**
- **Packs randomly divided into 3**
- **For each sub-sample all packs opened and bundles mixed.**
  - Air dry if necessary
- **2 bundles randomly selected (~160g) and homogenised**
- **2 x 100mg taken from each sub-sample**
- **DNA extracted on different days**
- **Subjected to PCR**
- **QC to include BT11 maize, MON 810 maize, LL rice (LL 62), and wild type rice**

# Summary outcome of GMO cases



Case	Product	PA result	GC result	Outcome
2023-8	Instant rice meal	Cry1Ab/Ac detected	GMO ND*	Compliant
2023-9	Rice noodles	Cry1Ab/Ac detected	Cry 1Ab/Ac detected	Non-compliant
2023-10	Rice noodles	Cry1Ab/Ac detected	GMO ND	Compliant
2023-11	Rice cakes	Cry1Ab/Ac detected	GMO ND	Compliant
2023-12	Short grain rice	CaMV 35S detected	T-NOS detected	Non-compliant

\*ND = CaMV 35S, t-NOS or Cry1Ab/Ac not detected

# Summary outcome of GMO cases



Case	Product	PA result	GC result	Outcome
2023-15	Vermicelli	Cry1Ab/Ac detected	Cry 1Ab/Ac detected	Non-compliant
2023-16	Round grain rice	t-NOS and CaMV 35S detected	t-NOS and Ca MV 35S detected	Non-compliant
2023-17	Rice balls	Cry1Ab/Ac detected	Cry 1Ab/Ac detected	Non-compliant
2023-24	Rice cakes	CaMV 35S detected	GMO ND	Compliant

\*ND = CaMV 35S, t-NOS or Cry1Ab/Ac not detected

# PAA's in nylon kitchenware



- **Background**

- Nylon kitchenware failed on PAA migration
- FBO part – only limited number of utensils tested
- GC – tested each item individually
  - 3 examples of 6 items total 18 analysis.

- **Sample analysed using prescribed procedures**

- Immersion in 3% acetic acid at 100C for 2hours.
- Analysis by LCMS for aniline and 4,4-MDA
- Result converted to food basis using standard factors



# PAA's in nylon kitchenware



- **Aniline and 4,4-MDA migration detected in 3 samples:**
  - Ladle below regulatory LOD
  - 2 items of spatula with holes above – non-compliant.

Article number	Mean sum aniline & 4,4-MDA concentrations			
	Not less than		Not more than	
	mg kg <sup>-1</sup> food	µg kg <sup>-1</sup> food	mg kg <sup>-1</sup> food	µg kg <sup>-1</sup> food
10 Wide spatula with holes	0.01182	11.82	0.01424	14.24
16 Wide spatula with holes	0.01047	10.47	0.01252	12.52
11 Ladle	0.0081	8.10	0.00972	9.72

# Nitrofurans in prawns



- Sample received as a SEO.
- OL reported the presence of AOZ.

Parent drug	Marker metabolite	Abbreviation
Furazolidone	3-amino-2-oxazolidinone (3-amino-1,3-oxazolidin-2-one)	AOZ
Furaltadone	3-amino-5-morpholinomethyl-2-oxazolidinone (3-amino-5-(morpholin-4-ylmethyl)-1,3-oxazolidin-2-one)	AMOZ
Nitrofurantoin	1-aminohydantoin (1-aminoimidazolidine-2,4-dione)	AHD
Nitrofurazone	Semicarbazide (hydrazinecarboxamide)	SEM



# Nitrofurans in prawns



- **Sample analysed using standard procedures**
  - 3 replicates over 3 days
  - Sample extracted and derivatised
  - Analysis by LCMSMS using isotopically labelled internal standard

<b>3-amino-2-oxazolidinone (AOZ, Total)</b> <b>micrograms AOZ per kilogram drained portion, (<math>\mu\text{g kg}^{-1}</math>)</b>	
<b>Not less than</b>	<b>Not more than</b>
1.1	2.0

- **Metabolite of Furazolidone detected**
  - Sample deemed non-compliant.

# Mycotoxins



- **4 mycotoxin cases – 3 aflatoxin, 1 ochratoxin A.**
- **Analysis:**
  - All received as slurries
  - Analysed using standard procedures
    - Solvent extraction
    - Immunoaffinity clean up
    - LC fluorescence detection (following derivatisation for aflatoxins)
  - 3 replicates over 3 days
  - Confirmed by LCMSMS



# Mycotoxins - results



- **Aflatoxins**

Sample	Aflatoxin B1 / ug/kg	MU / ug/kg	Total aflatoxin / ug/kg	MU / ug/kg
In-shell Peanuts	9.4	2.8	10.6	3.0
Curry powder	13.5	4.0	14.1	4.2
Figs sample A	5.5	0.3	20.2	2.8
Figs sample B	<0.75	-	0.93	1.21

- **All samples deemed non-compliant**

# Mycotoxins - results



- **Ochratoxin A**

Sample	Result / ug/kg	MU / ug/kg
Raisin sample A	27.4	5.2
Raisin sample B	30.9	2.1

- **Sample deemed non-compliant**

# Pesticide residues



- **Captan in Flaxseed**

- Captan detected by OL in flaxseed
- FBO lab had used QuEChERS method for analysis
- GC trialled QuEChERS method – unsatisfactory recovery.
- Switched to SPE method.

- **Thifluzamide in Organic Peanuts**

- OL reported thifluzamide at 0.2mg/kg, limit 0.1mg/kg
- Analysis using QuEChERS followed by LCMSMS
- GC detected 0.009mg/kg
- However, sample described as ‘Organic’

# Pesticide residues



- **Dinotefuran in Jasmine Tea**

- Found at 0.025 mg/kg by OL
- Second portion noted presence, but below LOQ.
- Analysed using QuEChERS followed by LCMSMS

Sample	Mean Dinotefuran Concentration (mg kg <sup>-1</sup> )	U (mg kg <sup>-1</sup> )
Jasmine Tea	0.0115	0.0046

- Sample deemed satisfactory having regard to analytical uncertainty.

# Summary



- **Number of cases returning to 'normal' levels following drop due to Covid.**
- **Nature of cases diverse**
- **Mix of analysis and advisory / interpretive**
- **Referee case analysis continues to resolve disputes in the food and feed sectors outside of the court system.**

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Thank you.



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