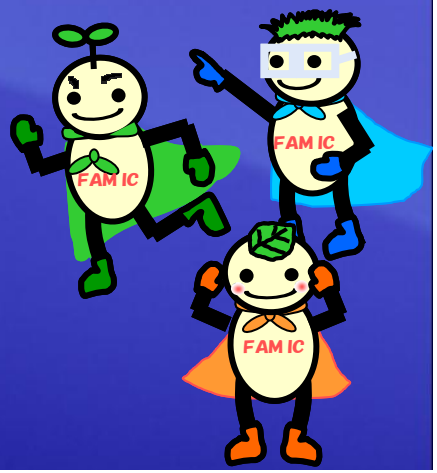
~ Key note presentation ~

Feed Safety Analysis

Part III Chemical Hazards



Toshiaki YAMATA, FAMIC (Kobe Regional Centre)

Today's Presentation

1. Outline of chemical hazards in feeds
2. Examples of methods of analysis
3. Results of monitoring for feeds

Outline of Chemical Hazards in Feeds

FAM

Chemical Hazards in Animal Feeds

Mycotoxins
Aflatoxin B_1
Deoxynivalenol
Zearalenone

Pesticides
Residual Pesticides
POPs

Heavy metals
Cadmium, Lead,
Mercury, Arsenic

Others
Nitrate Nitrogen,
Malachite green,
Melamine,
Dioxins

Threatening public safety and / or stable production of livestock products

Targets of Monitoring

1. Risk of substance

- Probability of contamination

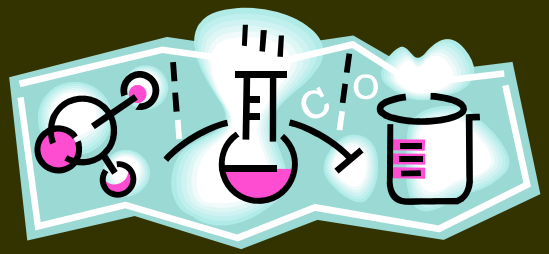
- Toxicity
  → to animals
  → to human × exposure through livestock products

2. Social impact

(Consumer's interest)

3. International trends

4. Availability of analysis



Examples of Methods of Analysis

AMI

Methods of Analysis

Main steps of chemical instrumental analyses

1. Extraction (pretreatment if needed)

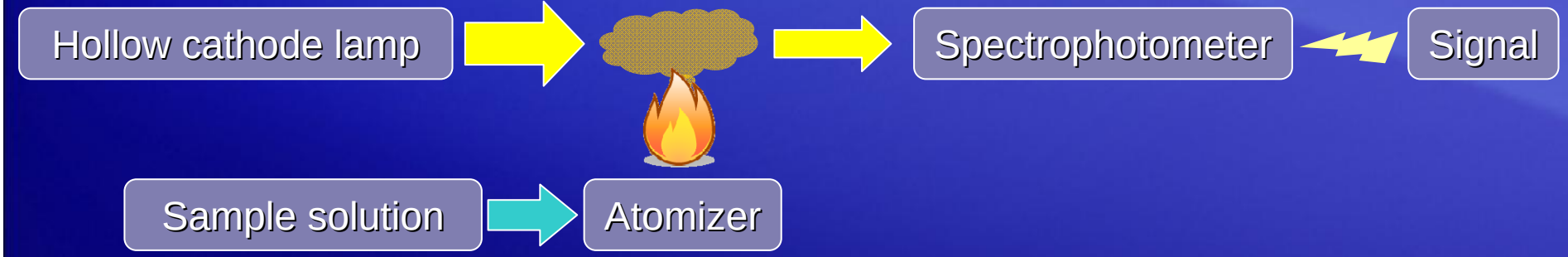
2. Cleanup (concentration if needed)

3. Measurement
   (separation, detection and quantification)

Methods of Analysis (Ex. 1)

- Determination of cadmium and lead by using atomic absorption spectrophotometer (AAS)

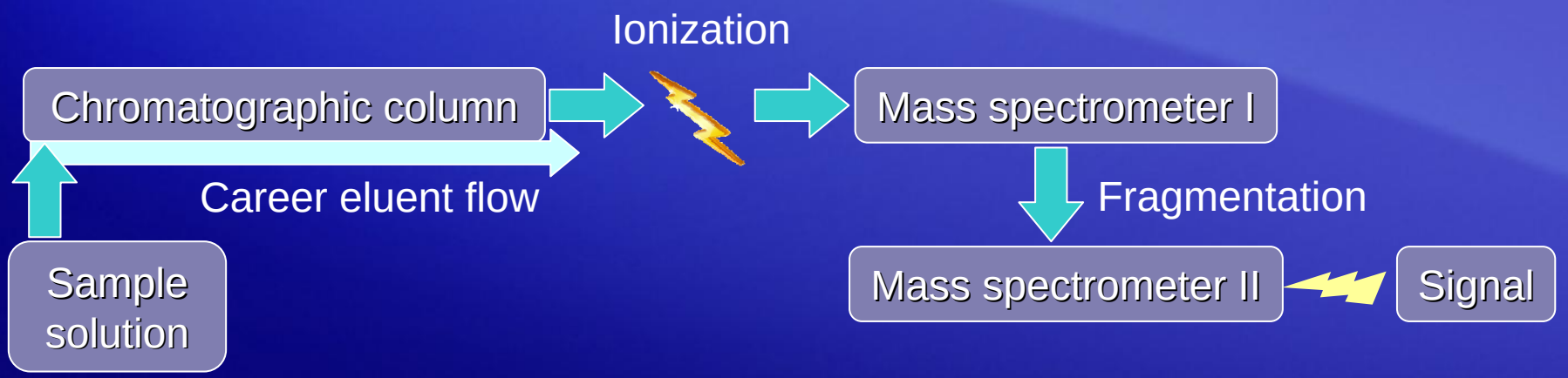
1. Pretreatment and Extraction	Hydrochloric acid after ashing
2. Cleanup	Complexation and solvent extraction
3. Measurement	AAS



Methods of Analysis (Ex. 2)

- Simultaneous determination of 11 kinds of mycotoxins by using liquid chromatograph – tandem mass spectrometer (LC-MS/MS)

1. Extraction	Aqueous acetonitrile
2. Cleanup	Multi-functional cleanup column
3. Measurement	LC-MS/MS



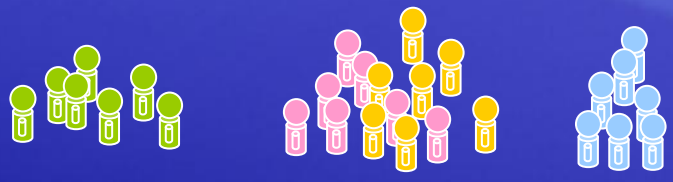
Chromatography

Separation of the components along the time axis according to their affinity for the column packing agents

Analytes (University students)

Chromatographic Columns (Attractions)

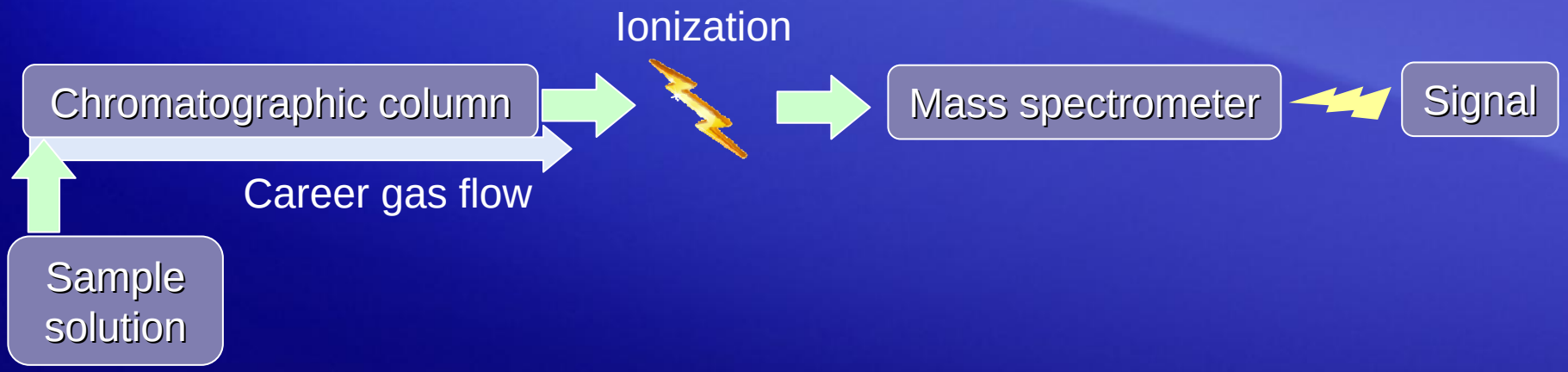
Faculty of economics
Faculty of letters
Faculty of science
Faculty of veterinary medicine



Methods of Analysis (Ex. 3)

- Simultaneous determination of 148 kinds of pesticides by using gas chromatograph – mass spectrometer (GC-MS)

1. Extraction	Aqueous acetonitrile
2. Cleanup	Diatomite column, Gel permeation chromatography, Graphitized-carbon column, etc.
3. Measurement	GC-MS



Establishment of Official Methods of Analysis

1. Development
2. Method validation
   - In-house study
     - Selectivity (Interference)
     - Trueness (e.g. Recovery test)
     - Precision (Repeatability)
     - Limit of quantification, Limit of detection
   - Collaborative study
     - Precision (Reproducibility)
3. Expert review
   - Advisory committee
4. Report to MAFF

FAMIC develops 5 ~ 10 methods of analysis every year!

Results of Monitoring for Feeds

MIC

Results of Monitoring for Feeds

FY2009 (April 2009 ~ March 2010)

1. Heavy metals

Target	Matrix	Sample tested	Maximum (mg/kg)	Maximum limit (mg/kg)
1) Cadmium	Formula feed	156	0.4	1.0
	Fish meal	36	2.3	2.5
	MBM	13	0.06	
2) Lead	Formula feed	156	2.1	3.0
	Fish meal	36	3.9	7.5
	MBM	13	1.9	
3) Mercury	Formula feed	156	0.11	0.4
	Fish meal	39	0.97	1.0
	MBM	13	0.24	
4) Arsenic	Rice straw	5	4.7	7

Results of Monitoring for Feeds

FY2009 (April 2009 ~ March 2010)

2. Mycotoxins

Analyte	Matrix	Sample tested	Maximum (mg/kg)	
1) Aflatoxin B_1	Formula feed	265	0.007	(ML: 0.01 ~ 0.02)
	Feed ingredients	207	0.026	Copra meal
			0.01	Maize
2) Deoxynivalenol	Formula feed	135	1.4	(ML: 1.0 ~ 4.0)
	Feed ingredients	171	1.4	Maize
			6.3	Corn gluten feed
			5.2	Corn gluten meal
3) Zearalenone	Formula feed	168	0.23	(ML: 1.0)
	Feed ingredients	158	5.4	Corn gluten meal

ML: Maximum limit in formula feed (mg/kg)

Results of Monitoring for Feeds

FY2009 (April 2009 ~ March 2010)

3. Pesticides

Matrix	Detected pesticides	Sample detected	Maximum (μg/kg)
Formula feed 31,568 total data for 146 kinds of pesticides	Chlorpyrifos-methyl	19	200
	Malathion	12	150
	Propargite	6	64
	Glufosinate	4	51
	Fenitrothion	4	50
	Chlorpropham	3	160
	EPN	3	49
	Pirimiphos-methyl	3	81
	Chlorpyrifos	1	40
	Piperonyl butoxide	1	32
	Fenpropathrin	1	120

Results of Monitoring for Feeds

FY2009 (April 2009 ~ March 2010)

3. Pesticides (cont.)

Matrix	Detected pesticides	Sample detected	Maximum (μg/kg)	
Feed ingredients 34,444 total data for 143 kinds of pesticides	Malathion	17	560	Screening pellet
	Chlorpyrifos-methyl	12	380	Milo
	Fenitrothion	8	170	Corn gluten meal
	Glyphosate	4	230	Maize
	Glufosinate	4	50	Maize
	Propiconazole	4	6,200	Ryegrass straw
	Pirimiphos-methyl	3	370	Milo
	EPN	2	40	Maize
	Chlorpyrifos	2	44	Maize
	Tebuconazole	2	980	Ryegrass straw
	Propargite	2	37	Wheat bran
	Endosulfan	1	21	Recycled straw
	Trifluralin	1	1,900	Alfalfa hay
	Methidathion	1	460	Fruit juice residue
	Methoxychlor	1	6	Recycled straw

Results of Monitoring for Feeds

FY2009 (April 2009 ~ March 2010)

4. Others

Analyte	Matrix	Sample tested	Maximum	
1) Nitrate nitrogen	Grass hay	24	2,100 mg/kg	Alfalfa hay
2) Malachite green	Formula feed	11	Not detected	
	Fish meal	28	Not detected	
3) Melamine	Formula feed	1	Not detected	
	Fish meal	12	Not detected	
	Garlic powder	1	Not detected	
4) Dioxins	Fish oil	10	12 ng-TEQ/kg	
	Fish meal	10	0.80 ng-TEQ/kg	
	Animal fat	10	0.20 ng-TEQ/kg	

http://www.famic.go.jp/ffis/feed/info/sub2_h21_gaiyou.html
(Only Japanese version available, now)

New Approach to the Chemical Hazard

- Guidelines for the preventing the contamination of feed products with toxic substances (March 2008)
  - Action by competent authority
  - Collection and sharing of information
  - Importation of feeds
  - Acceptance of ingredients at manufacturing plants
  - Guidelines concerning the manufacture of feeds
  - Guidelines concerning the transport and storage of feeds
  - Response to the possibility of production of toxic animal products
  - Reporting of import and manufacturing volume

Thank you for listening

We hope you have fruitful and enjoyable days.

Food and Agricultural Materials Inspection Center, I.A.A.