

	Examination	
Dpen-bo Examina	ok examination tion questions in English	70 %
Answer: Frontier	in Chinese Review (Homework)	10%
Experim	ent Performance	20%
Requirer	nent on experiments	









Main contents of the lecture

Pesticide Residue Analysis

Introduction: Pesticide residue; ADI; MRL; Guideline for safety application of pesticides; Pesticide standard; LOD; LOQ; fortified recovery; confirmation or validation Crop field trial in China (Chapter 1) Validation of pesticide residue analysis (Chapter 2) Sampling, preparation, storage (Chapter 3) Extraction, concentration, clean-up (Chapter 4, 5) Analysis: GC、HPLC、TLC、enzyme inhibition method, ELISA、Multi residue analysis (chapter 6, 7, 8,9,10,12)

Experiment

1 Comprehensive experiment including field trial **design** and sample preparation in lab for residue analysis

2 Preparation of **column chromatography** for clean-up and determination of eluting curve (or **P value** determination of pesticide in solvent pair)

3 TLC-enzyme inhibition method

4 Determination of Endocrine Disruptor or Toxin in agricultural product

5 Analysis of GMO

SCI Journal	
Anal Chem	
Environ Sci Technol	
Anal Chim Acta	
Environ Pollut	
Water Res	
Chemosphere	
Food Chem Toxicol	

SCI Journal	Food Chemistry	4.3
	J Unromatogr A	4.2
	Anai Bioanai Chem	3.5
	Papid Commun Mass	2.0 Spectrom 28
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	Fasteriaslagy and En	vinonmontal Safaty 2.2
	Ecotoxicology and En	vironmental Safety 2.5
	Food Control	2.5
	Food Additives & Cont	aminants 2.2
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J OI AUAC International 1.2 Dell Engineer Contant Taginal 1.1		
Buil Environ Contam Toxicol 1.1	L	
Chromatographia 1.0		
Mycopathologia 1.8		
Current Analytical Chemistry 1.8		
International Journal of Environment	ntal Analytical Chemistry	
Human and Ecological Risk Assess	ment 1.4	
Biomedical and Environmental Scie	ences 1.0	
Journal of Integrative Agriculture		









Pesticide residue

The benefits of pesticide: no pesticide, few food

The risk of pesticide: without pesticide residue control, worse future

Pesticide Residue Science was developed with the development of pesticide production, analytical technology and the concern of the public.

Pesticide production development

1st registered pesticide: 巴黎绿, 1900, USA 1st synthesized pesticide: DDT, 1939 1st OP: 特普(TEPP), 1943 1st carbamate: dimetan, in 1950s

fungicide in1940s herbicide

from 1940s, organic synthesized pesticides, more concern on their degradation, accumulation, toxicology, residue

Analytical technology development

Separation

Column chromatography ----TLC- --GC/LC GC packed column----capillary column LC --- HPLC---UPLC

Determination:

chemical method FID/TCD----ECD/FPD/NPD----MSⁿ UVD---DAD/FLD----MSⁿ

Sample Preparation

LLE ----SPME/LPME/MSPD/MAE.....

Public concern

Silent Spring—Rachal Carson

PIC POPs EDCs Pollution-free farm produce, green food, organic food 无公害食品行动计划 MRL establishment

	Rotterdam Convention	Forbidden in China	Reason
醋酸苯汞	1992.12	1971	High toxicity biological
发氏剂	1991	1980'	concentration
狄氏剤	1991.9	1980'	
二溴氯丙烷		1982	致癌、致突变
氟乙酰胺	1991	1982	High toxic
「六六六」	1991.9	1983	biological concentration
二溴乙烷	1992.12	1984	致癌、致突变
敌枯双		1986	致癌、致畸
毒鼠强		1991	剧毒
杀虫脒	1992.11	1993	致癌
除草醚		1997	三致
毒杀芬	1995	2002	生物富集
汞制剂	1992.12	2002	高毒、生物富集
砷类	http://blog.scier	comet.cn/blog-5	535802-581904.html
铅类		2002	高毒
鼠甘伏		2002	剧毒
氟乙酸钠		2002	剧毒
毒鼠硅		2002	剧毒
甲胺磷	1995.3	2007	High toxicity
对硫磷			
甲基对硫磷			
久效磷			
磷胺/			
八氢二丙醚		2008	High risk and hazard

Pesticide forbidden		
Ethametsulfuron-methyl 胺苯磺隆	2017-7-1	
Metsulfuron-methyl 甲磺隆	2017-7-1	
Chlorsulfuron 氯磺隆	2015-12-31	
Asomate 福美胂,urbacide福美甲胂	2015-12-31	
Limitation in vegetable: Chlopyrifos,	Triazophos 2016-12-31	

Pesticide residue control

PIC公约《关于在国际贸易中对某些危险化学品和农药采用 事先知情同意程序的<u>鹿特丹公约</u>》

Rotterdam Convention on the Prior Informed Consent Procedure for Certain Hazardous Chemicals and Pesticides in International Trade

Effective date in China: June 20, 2005

Rotterdam Convention, appendix 3

Pesticide (18): 2,4,5-T、aldrin 艾氏剂、dieldrin 狄氏剂、Captafol 敌菌丹、 Chlordane氯丹、BHC 六六六、DDT 滴滴涕、Lindane 林丹、 Heptachlor七氯、HCB 六氯苯、dinoseb 地乐酚、epoxyethane 环氧乙 烷、Ethylene Dibromide 二溴乙烷、chlordimeform 杀虫脒、 Chorobenzilate 乙酯杀螨醇、fluoroacetamide 氟乙酰胺、 pentachlorophenol 五氯苯酚、Toxaphene 毒杀芬、Hg-contained 汞化 合物

Pesticide formulation with high risk (5) : monocrotophos, methamidophos, phosphamidon, parathion, parathion-methyl

Industry chemical (5) : PBB、PCB et al

Rotterdam Convention, appendix 3

Renewal in 2011:

endosulfan alachlor aldicarb were listed in Appendix 3.



Stockholm Convention

POPs Convention

the Stockholm Convention on Persistent Organic Pollutants

Effective date, Nov. 11, 2004

POPs

Organic compounds that are resistant to **environmental degradation** through chemical, biological, and photolytic processes.

Persist in the environment, to be capable of long-range **transport, bioaccumulation** in human and animal tissue, **biomagnify** in food chains, and to have potential significant impacts on human health and the environment.

POPs List

21 POPs at total until May, 2009 (10 pesticides)

1st list: DDT 滴滴涕, Chlordane 氯丹, Mirex灭蚁灵, dieldrin 狄氏剂, Endrin 异狄氏剂, Aldrin 艾氏剂, Heptachlor 七氯, Toxaphene 毒杀酚, HCB 六氯苯;多氯 联苯、二噁英和呋喃。'dirty dozen'

2nd list:

α-六氯环己烷; β-六氯环己烷; 四溴联苯醚和五溴联 苯醚; 六溴联苯醚和七溴联苯醚; 十氯酮; 六溴联苯; lindane **林丹**; 五氯苯; 全氟辛烷磺酸、全氟辛烷磺酸盐 和全氟辛基磺酰氟。

Endocrine disrupting chemicals

Endocrine disrupting chemicals (EDC) Environmental hormone Environmental endocrine disruptors (EEDs) Pesticide endocrine disruptors Endocrine disrupting pesticides

Exogenous substances that act like hormones in the endocrine system and disrupt the physiologic function of endogenous hormones.

EDC listed by EPA

Endocrine Disruptor Screening Program (EDSP) April 2009 Final List of Chemicals for Initial Tier 1 Screening

67 chemicals including 58 active ingredients of pesticides.

2,4-D, Atrazine, Glyphosate, Metolachlor, Metribuzin, Simazine, Trifluralin

Abamectin, Carbofuran, Acephate, Chlorpyrifos, Diazinon, Dimethoate, Malathion, Methamidophos, Methyl parathion, Cyfluthrin, Cypermethrin, Permethrin Endosulfan, Imidacloprid Captan, Chlorothalonil, Metalaxyl

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AL PROPERTY.	You are here: ERA home * Office of Presention, Ratholder, Epiloar and Procedures * Final Let of Chamicals for Initial	, and Tasic Substance Tier 3 Screening	1 * Office of Scient	on Coordination and Po	dice · Endecrine Diameters Dreft	
Information	Final List of Chemicals for	Initial II	er 1 Scr	eening		
Development	April 2009 Final List of Chemicals for Initial Tier 1	Screening				
Validation	This page presents an alphabetized final list of the 6	7 pesticide active in	gredients and H	ev/pesticide inert ch	emicals selected for Tier 1 scree	ning.
ty Setting effics	The final list was published in a Federal Register Not potential only, it should not be construed or character	ice in April 2009. Be erized as a list of kr	cause this list of own or likely en	chemicals was selec doorine disruptors. E	ted on the basis of exposure ead more about this list.	
y & Guidance				_		
am Documents			Pesticide			
eholder input		CAS Number	Active	HPV/Inert		
ied Links	Chemical Name					
	240	94757				
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	ethylhexy()-3a,4,7,7a-tetrahydro-	110404	^ ·			
	Abamedin	71751412	ж			
	Acephate	30560191	ж			
	Acetone	67641		*		
http://	www.ena.gov/scinoly/	scnend	o/nubs	/nrioritys	etting/finallis	et hte



Isocarbophos

Bayer-93820 CAS: 24353-61-5 History: Evaluated, but never marketed by Bayer AG in 1960'. Patent: Ger2135349, Ger2340080 by Germany Bayer Currently manufactured and used in China.

1970' developed by 华中师范学院 1979, marketed by 沔阳农药厂

Long persistent in crop, rather than in soil High toxic, forbidden use in fruit, vegetable, tobacco, tea, herb In China, MRL, <LOD (fruit, rice), 0.02(vegetable) No MRL standard set by CAC、EU、US、UK

青岛严查"毒韭菜"其他地区加强食品检查

中国色谱网(2010-4-12 15:33:34)

据媒体报道,从4月1日开给,曹岛一些医院陆续接到9名食用韭菜后中毒的患者,他们都是 食用毒韭菜之后出现了头疼、恶心、腹泻等症状,经医院检查属于有机磷中毒,也就是说韭菜上 的残余农药严重超标导致9人中毒。目前这些患者经过救治身体已经恢复了健康。











Pesticide Residue

A pesticide residue is any substance or mixture of substances in <u>food for humans or animals</u> resulting from the use of a pesticide and includes any specified derivatives, such as degradation and conversion products, metabolites, reaction products and impurities which are considerable to be of toxicological significance.

Definition of PR

Matrix: organism, agricultural products, environment

Target: pesticide, metabolite, degradates, impurity

Characteristic : trace, toxicology



Pesticide residue definition

- For compliance with the MRL for plant commodities
- · For compliance with the MRL for animal commodities
- · For Estimation of the dietary intake for plant commodities
- · For Estimation of the dietary intake for animal commodities

Pesticide Residue aldicarb: 涕灭威砜、亚砜 倍硫磷: Sum of fenthion, its oxygen analogue ar

倍硫磷: Sum of fenthion, its oxygen analogue and their sulphoxides and sulphones, expressed as fenthion 甲拌磷及其氧化物、亚砜和砜 茚虫威及其R-异构体 Carbofuran: 3-羟基克百威 Endosulfan: alpha and beta endosulfan, endosulfan sulphate Benomyl, carbendazime and thiophanate-methyl, expressed as carbendazim Methomyl and thiodicarb, expressed as methomyl 代森类均用 CS₂表示

P8



Pesticide Residue

Extractable residues PR, which could be extracted by a standard method, such as Soxhlet solvent extraction.

Unextractable residues (Non-extractable residue)

Bound residues of pesticides Conjugated residues of pesticides

Pesticide Residue

Bound residues of pesticides

指农药或代谢物与土壤中的腐殖质、植物体的木质素、纤维素通过化学键合或物理结合作用,牢固结合形成的残留物。

Chemical species in soil, plant or animal tissue originating from a pesticide, (generally radio labelled) that are unextracted by a standard method, such as Soxhlet solvent extraction, which does not significantly change the chemical nature of the residues. These unextractable residues are considered to exclude small fragments recycled through metabolic pathways into natural products. (after Roberts, 1984)

The bound residue might be released for the change of the environmental condition, such as soil pH, climate. **Biological Bombs** \geq_{rel}

Pesticide Residue

Conjugated residues of pesticides 指农药母体或代谢物与生物体内某些内源物质如糖苷、 氨基酸、葡萄糖醛酸等在酶的作用下结合形成的极性较强、 毒性较低的残留物。

Conjugation Biosynthetic reaction in which a pesticide or its metabolite is linked to an endogenous compound.

Biotransformation where the pesticide is conjugated with a naturally occurring compound (e.g. sugars, glutathione).

Pesticide Residue

Incurred residue

Residue in a commodity resulting from <u>specific use of a</u> <u>pesticide</u>, <u>consumption by an animal or environmental</u> <u>contamination</u> in the field, as opposed to residues from laboratory fortification of samples.

Pesticide Residue

Dislodgeable residue

Portion of a pesticide residue on treated vegetation that is readily removable and may be used as an **index for risk to farm workers**. Generally measured by the residue removed when leaf discs are shaken briefly in water.

REI (Re-entry interval, Restricted-entry interval) Minimum time between pesticide application and human re-entry to a treated area. Established by a regulatory authority to assure safety of workers from exposure to residues.

Source and Effect factors of PR

Source of Pesticide Residue

Direct contamination for pesticide application PR transferred from contaminated environment to crop PR from commodity to food chain, biological concentration

Effect factor of Pesticide Residue

- A Physicochemical property
- B Crop type and edible portion
- C Application method, dose, stage, frequency
- D Environmental factor, climate

Maximum residue limit, MRL

Maximum concentration of a residue that is legally permitted or recognised as acceptable in, or on, a food, agricultural commodity or animal feedstuff as set by Codex or a national regulatory authority.

GB 2763-2014 : MRL, 3650; pesticides, 387

MRL The role of MRLs To assure effective protection of plants and animals To lead to the minimum residues in food, which are safe for any population group of consumers To enable the free trade of agriculture commodities according to international agreements (reduce the <u>pechnical Barriers to Trade</u>) Do not contaminate or adversely affect the environment.





5/8 The proposed codex MRL is submitted to the Commission at Step 5; as there seems to be no controversy and no need for further discussion at step 6 and 7, omission of these steps is recommended to the Commission.



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Pesticide Residues in Food and Feed	
PETTICIDE INDEX	
This page contains an index of perticides.	
Cloking on a particide takes the user to a page with details on acceptable uses of the particide.	
3ump to: 2 - A - B - C - D - E - F - G - H - I - K - L - M - N - O - P - Q - S - T - V - Z	
2	
* 2,4-D (20) * 2-Phenylphenol (56)	
A	
* Abameetin (177) * Acephate (95)	
* Aldicarb (117) * Aldrin and Dieldrin (1)	
* Aminopyvalid (220) * Aminog (122)	
* Amitrale (79)	
* Azinphas-Methyl (2)	
* Ameyeletin (129) * Annystrohin (229)	
3	
* Benalaxyl (155)	
* Bentazone (172)	
* Bifenazate (219)	
Bioremethin (%)	
* Bitertanal (144)	



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	他行刖	施行後
*	0. 5ppm	現行どおり
りんご	0. 1ppm	現行どおり
きゅうり	基準無し (検出されても違反でない)	 0. 2ppm (国際基準等を参考に設定)
キャベツ	基準無し (検出されても違反でない)	- 律基準(0.01ppm)を 超過すれば違反



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Health and Sa HSE Executive	eey atabases Applicant advice Farmers & grovers Arventy	Chemicals Regulation Directione Perification users Home garden Food industry Biopersticides Advisory groups Biocides	
Reach			
In this section	You are in: Databases > MRL database home		
Court MPL	III Maximum Residue Level (MRL) Database	Health and Safety Executive, UK	
Useful links Original UKIEC MRL database	Use these databases to find pesticide maximum residue la	rvels for trade in or to the UK or other EU countries	
Text size	Search For U	VEC MRLs Search For Codex MRLs	
Senal			
S Default [current] S Large	Our Maximum Residue Level (MRL) databases provide compret introduction of EC Regulation 395/2005.	hensive information on statutory and non-statutory MRLs applying to pesticides in food following the	
C Largest Save my preference Switch	MRLs are limits on pesticide residues in food. They are the main There are two types of MRL that may apply to foods marketed wi	imum residue levels likely to be left in food after it has been properly treated with a pesticide. Ibin the European Union.	
HSE is not responsible for the content of external websites.	Statutory MELs		
Constant Constant Mark	The first type is EC Statutory MRLs set under EC Regulation 3/9 involved in the marketing of floed or fleed within the EU must con may be set. Annexes 2, 3a and 3b contain the MRLs that apply 3	V2005, which came in to fince on 1 Geptember 2008. Where these EC MPLs are set everyone given them. Anext of the Pegudons lists the flood and field commodities for which the MPLs rises a more detailed description of each Annex.	
Regulation 096/2005 aims to set MRLs for all pesticipe active substances which, when used, produce an identifiable residue. The only exceptions substances whose residues cannot be distinguished from background levels, these are listed in <u>Access to</u> of the Regulation.			
	Some active substances used as pesticides will not be listed in longer used in the EU when the Annexes to Regulation 196/200 level of 0.01 mg/kg will apply to any unnamed active in combinal	any of the <u>Access</u> to Regulation 398/2005 (for instance because the pesticide concerned was no 5 were drawn up). In anticipation of these incrumstances Regulation 398/2005 states that a default ion with any of the tood and feed commodities listed under the Regulation.	
	Important note: MFLs set under Regulation 395/2005 apply on will be subject to any statution EC or UK MFLs applying at that found via a separate database, accessed via the link below. Yo	b) to produce treated on or after 1 September 2008. Produce treated before 1 September 2008 one-life internation on those MSLs in place before Regulation 266/2005 came intri fore can be a can view the old data here. <u>Https://scuru.peticides.org/ub/HSLs_EC_createstbasp</u> .	
	(2.250 H)	attps://secure pesticides gov.uk/MRI s	







1 STMR and HR from the trial

HR-

Highest residue in edible portion of a commodity found in trials used to estimate a maximum residue level in the commodity

STMR

Supervised trials median residue is the expected residue level in the edible portion of a food commodity when a pesticide has been used according to maximum Good Agriculture Practice conditions.

2 ADI from toxicology

Acceptable daily intake (ADI)

Estimate of the amount of a pesticide in food and drinking water which can be ingested daily over a lifetime by humans without appreciable health risk.

mg / kg body weight / day.

ADI=动物无作用剂量/安全系数 no observed adverse effect level, <u>NOAEL</u> safety factor

2 ADI from toxicology

NOAEL

- A Acute toxicity test--- aRfD acute reference dose
- B Subacute toxicity test
- C Chronic toxicity test--- cRfD chronic reference dose $D^{-\frac{1}{2}}$
- D"三致"effect--carcinogenic effect, Teratogenic effect, Mutagenicity

E NOAEL No Observed Adverse Effect Level

- Safety factors for the differences between species or individual 10 for the safety between human beings and animal 10 for the individual differences
 - 10 for safety avoiding unknown risk

2 ADI from toxicology

ADI -making organization

International-JMPR

China-Ministry of Health

The 1825th announcement of Ministry of Agriculture, PRC

Comparison of ADI and acute toxicity

Pesticide	ADI (mg/Kg/d)	Acute toxicity rat LD50 (mg/Kg)
氧乐果	0.0005	50
甲基对硫磷	0.001	14 (維) 24 (雌)
杀螟硫磷	0.001	250-500
五氯硝基苯	0.001	12000
氰戊菊酯	0.001	451
敌敌畏	0.004	80 (雄) 56 (雌)
对硫磷	0.005	13 (維) 3.6 (雌)
滴滴涕	0.005	113(雄) 118(雌)
甲萘威	0.01	
溴氰菊酯	0.01	70-140
克百威	0.01	8-14
林丹	0.0125	1000(雄) 900(雌)
乐果	0.02	320-380
马拉硫磷	0.02	2800
乙酰甲胺磷	0.02	945(雄) 866(雌)
氯氰菊酯	0.05	251

MRL

Zero tolerance

Limit for a pesticide residue in food or feed which is assumed to be **zero** and therefore any **detectable residue is deemed illegal**.

Zero tolerances are used by some regulatory systems, e.g. USA, where no maximum residue limits have been established for particular pesticide/crop combinations.

extraneous residue limit, ERL

ERL

Maximum concentration of a pesticide residue, arising from environmental sources (including former agricultural uses), other than from the use of a pesticide directly or indirectly on the commodity, that is recommended to be permitted in or on a feed or food commodity. (FAO)

