行政院及所屬各機關出國報告

（出國類別：出席國際會議）

出席『第卅四屆國際農藥殘留標準委員會』
(34th Session of Codex Committee on Pesticide Residues)
報告書

服務機關：行政院農委會農業藥物毒物試驗所
出國人職稱：研究員兼系主任
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出國地點：荷蘭海牙
出國期間：91年5月13日至91年5月18日
報告日期：92年2月15日

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公 務 出 國 報 告 提 要

頁數: 55　含附件: 是

報告名稱:

參加第三十四屆國際農藥殘留標準委員會

主辦機關:

行政院農業委員會農業藥物毒物試驗所

聯絡人／電話:

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出國人員:

翁慄慎　行政院農業委員會農業藥物毒物試驗所　殘毒管制系　研究員

出國類別: 其他

出國地區: 荷蘭

出國期間: 民國 91 年 05 月 11 日 -民國 91 年 05 月 20 日

報告日期: 民國 92 年 02 月 15 日

分類號/目: F0／綜合（農業類）　I5／化學與環境科學

關鍵詞: 國際農藥殘留標準委員會;農藥殘留標準;最高殘留限量;經取食之暴露量;農藥殘留分析方法準則;農藥安全評估

內容摘要: 第卅四屆國際農藥殘留標準委員會(34th Session of Codex Committee on Pesticide Residues，簡稱CCPR)於2002年5月13日至18日在荷蘭海牙召開。CCPR爲聯合國組織下食品安全主要委員會之一，由荷蘭衛生福利部主辦。每年定期集會，討論作物中農藥殘留標準等相關議題。本期計有聯合國Codex五十二個會員國及十二個國際組織代表參加。本人代表國際純化學及應用化學學會（IUPAC）出席。經費由本所公務預算支出。本會議計進行十四個議題，包括報告案及討論案。討論案內容主要爲制定部分農藥在各類作物中之最高殘留限量。同時討論農藥經取食之暴露量與殘留標準，農藥殘留分析方法準則，Codex MRL制定程序引起之貿易障礙，辛香料殘留標準研訂案，Codex殘留標準作物分類修正等議題，結論提交聯合國食品安全委員會討論，作爲世界農產品貿易及食品安全管理之依據。本人長期從事農藥研究工作，負責國內農產品農藥殘留檢驗、國內及進口容許量標準研訂及農藥暴露量之安全評估等工作。我國成爲世界貿易組織之會員國後在農產品貿易諮商談判上該類資訊更爲重要。政府應該充分了解聯合國食品標準委員會之組織及運作，尋求參與聯合國食品標準委員會議題討論之機會。重視國際農藥殘留標準委員會之重要性，參考Codex準則進行農藥殘留調查及整體性評估之運用，長期進行食品中農藥安全評估工作。檢討國內農藥管理制度，增加農藥管理專責機構之人力資源。

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國際農藥殘留標準委員會(Codex Committee on Pesticide Residues，簡稱CCPR)為聯合國世界糧農組織及世界衛生組織食品安全標準委員會之一，任務為制定農藥對人體健康相關之標準，包括農藥每日可攝入量（ADI）、農藥急性毒參考值(Acute RfD)、農藥在各類作物中之最高殘留限量（MRLs）、外加最高殘留限量(EMRL's)及經取食安全評估值（Dietary Daily Intake Risk Assessment），主辦國為荷蘭。本會議每年四至五月於荷蘭海牙召開，會期約一周，由聯合國世界糧農組織及世界衛生組織專家準備討論議題及會議資料提交會議討論，會議結論提送聯合國食品安全標準委員會(CAC)決議後作為世界性食品安全之準則，世界貿易組織（World Trade Organization）有關食品安全方面皆以此為標準，因此深受各國重視。我國非聯合國會員國，參與非開放性國際組織相關會議之機會很少，此等聯合國組織下之會議更不可能參加，對我國參與國際活動及世界貿易組織談判上，因無法及時取得國際認同之資訊而增加其困難度。本人以國際純化學及應用化學學會（IUPAC）代表身分參加，除在會中說明IUPAC與本會議之關係及主要活動及研究報告外，同時收集會議資料，了解各項議題之討論程序及決定之準則，當有助於我國進出口食品及國內農產品中農藥殘留標準制訂之國際化，及在農產品及食品之國際貿易上能引用國際準則而替提高農產品之競爭力。

過程

一、議程 Agenda

第卅四屆國際農藥殘留標準委員會（34th Session of CCPR）於2002年5月13日至18日在荷蘭海牙召開。主要討論議題依序見表一。

表一、第卅四屆國際農藥殘留標準委員會討論議題

題號	主要議題	討論文獻
	開幕詞Opening of the Session	
1	公告議題 Adoption of the agenda	CX/PR 02/1
2	推舉紀錄 Appointment of rapporteurs	
3	其它委員會轉請討論議題 Matters referred to the committee	CX/PR 02/2
4	2000及2001年JMPR報告一般性建議 Report on general considerations by the 2000 and 2001 JMPR	2000 and 2001 JMPR Reports
5	農藥經取食之暴露量與殘留標準 Deitary Exposure in Relation to MRL setting： a. Acute Dietary Exposure Assessment; b. The methodology of cumulative risk assessment; c. The application of risk analysis in the elaboration of Codex standards	CX/PR 02/3(a), 02/4(b), 02/5(c)
6	食品及動物飼料中農藥殘留標準 Proposed Draft MRL in Foods and Feeds	CX/PR 02/6
7	農藥殘留分析方法準則 Matters Related to Methods of Analysis for Pesticide Residues: a. Guidelines on Good Laboratory Practice in pesticide residue; b. The introduction section of the recommended methods of analysis for pesticide residues	CX/PR 02/7(a), 02/8(b), 02/9(c)

	c. Revision of the list of methods of analysis for pesticide residues.	
8	農藥評估優先順序 Establishment of Codex Priority Lists of Pesticides	CX/PR 02/10
9	Codex MRL制定程序引起之貿易障礙 Trade Vulnerabilities Arising from the Codex MRLs Establishment Process	CX/PR 02/11
10	辛香料殘留標準研訂案 Consideration of Elaboration of MRLs for Spices	CX/PR 02/12
11	Codex殘留標準作物分類修正 The Revision of the Codex Classification of Foods and Animal Feeds	CX/PR 02/13
12	其它待辦事項 Other business and Future Work	CX/PR 02/14
13	下次會議時間及地點 Date and Place of Next Session	
14	會議結論 Adoption of the Report	

本會議除正式議程外，另有二個 Working group 在會期中安排時間召開，分別為5月11日討論農藥評估優先順序，5月14日討論農藥殘留分析採樣準則。

二、參加人員

本屆會議計有來自聯合國五十二個Codex會員國及十二個國際組織代表共216人參加，參加國及國際組織代表人數見表二。本人代表國際純化學及應用化學學會（IUPAC）與會。

表二、參加卅四屆國際農藥殘留標準委員會國家及組織人員

國家	人數	國家	人數	國家	人數	國際組織	人數
Algeria	1	Germany	8	Mozambique	1	EC	3
Argentina	1	Ghana	2	Netherlands	10	EU	1
Australia	10	Greece	1	New Zealand	2	ITC/WTO	2
Austria	2	Guinea Ecuatorial	1	Nigeria	1	OECD	1
Bangladesh	1	Hungary	2	Norway	4	OIV	1
Belgium	3	India	2	Philippines	1	CI	3
Brazil	10	Indonesia	2	Poland	4	CLI	33
Canada	3	Iran	1	Senegal	1	IFT	1
Chili	3	Ireland	1	Slovak R	1	IBS	1
China	7	Israel	2	South Africa	1	ICA	1
Cuba	2	Italy	1	Spain	3	IUPAC	2
Czech R.	1	Jamaica	1	Sudan	1	IOSTA	2
Denmark	2	Japan	7	Sweden	2	FAO	2
Egypt	1	Korea R.	12	Switzerland	3	FAO/IAEA	1
Finland	4	Kuwait	1	Thailand	6	WHO	3
France	4	Malaysia	3	U. Arab Emirates	1	Secretariat	15
USA	18	Morocco	4	UK	5		

三、會議內容

本報告中所引用之專有名詞簡稱說明列於附件一。

開幕詞(Opening of the Session)

開幕式由荷蘭健康福利部公共衛生處Food and Non-Food部門處長Mr.De Leeuw致開幕詞。他強調各國對食品安全日益重視，更為世界貿易組織（WTO）談判之重要關鍵，必須以科學數據為基礎來制定安全標準以消除貿易障礙。他肯定本會議對農藥使用管理及食品安全所作之貢獻，並期許提昇研訂農藥殘留安全標準之時效性。

議題一、公告議題（Adoption of the Agenda）

說明本次會議之議題、特別議題會外討論時間及臨時議題，及大會秘書處服務項目。

議題二、推舉紀錄(Appointment of Rapporteurs)

大會主席推舉美國代表 Dr. C.W.Cooper及紐西蘭代表Dr. David W.Lunn 為大會紀綠。

議題三、其它委員會轉請討論議題 (Matters Referred to the Committee)

本屆會議由其它食品安全委員會及CAC執行委員會反應至本委員會討論者有：

1. FAO相關會議結論針對2000年後國際貿易改變對開發中國家之影響所作之14項特別需求及21項相關建議在會議中作成書面資料。
2. 執行委員會要求各委員會制定中長程目標（Medium-Term Plan），本議題將於下次討論。
3. 23rd 特殊用途營養及食品委員會(The Codex Committee on Nutrition and Foods for Special Dietary Uses)會議決議應針對嬰幼兒穀類食品訂定符合安全的容許量值。本會認為目前殘留安全標準之評估已考慮不同年齡層及不同族群之安全性，包括嬰幼兒食品。

議題四、2000及2001年JMPR報告一般性建議(Report on general considerations by the 2000 and 2001 JMPR)

世界糧農組織及衛生組織農藥殘留專家聯合會議(JMPR)每年九月集會一次，每次會期一個月，評估農藥毒理及殘留問題，制訂農藥每日可攝入量（ADI）、農藥急性毒參考值(Acute RfD)、農藥在各類作物中之最高殘留限量（MRLs）、

外加最高殘留限量(EMRL's)及經取食安全評估值（Dietary Daily Intake Risk Assessment）。平均每年評估廿五種農藥。會議中亦討論一般性議題，結論提翌年CCPR會議報告及討論。

本會議JMPR之報告內容摘要如下：

1. FAO/WHO研擬計畫針對農藥、動物用藥、食品污染物及添加物之毒性、攝入途徑、殘留及規格等進行安全評估通則之製訂，歡迎各國專家學者參與。
2. 農藥急性毒參考值(Acute RfD)之研訂準則及評估方式為2002年JMPR之重要工作項目。
3. 為加速JMPR藥劑評估之進行，OECD、EU、USEPA 建議成立跨國性工作小組來進行農藥資料審查。JMPR 之立場為樂觀其成，但必須克服技術、科學及政治上各國之考量差異。部分國際組織正在進行中之計畫如 OECD 之農藥登記資料最低需求、農藥殘留資料可依氣候區域分區共享或不同作物間殘留情形之共用等，其結果均可作為該工作小組之參考。

<u>議題五</u>、農藥經取食之暴露量與殘留標準(Deitary Exposure in Relation to MRL setting)

本議題有三大討論議題：

1. 經口攝食急毒性暴露量評估(Acute Dietary Exposure Assessment) - 本議題南非提供國內安全評估結果部分主食之攝食風險高於安全值25倍。印度之國民取食量調查資料即將完成。此等資料均供GEMS/Food 作資料更新。2001年JMPR計算經口攝食慢毒性暴露量評估初步結果加保利 carbaryl、合氯氟 haloxyfop 及撲克拉 prochloraz 高於ADI 值。 經口攝食急毒性暴露量評估初步結果得滅克 Aldicarb 在香蕉及馬鈴薯、chlorpropham 在馬鈴薯、納乃得

methomyl及得芬諾 tebufenozide 在部分作物中高於 Acute RfD值。大會要求美國規畫以經 probabilistic 統計分析程式之評估方法以作為對高於Acute RfD值之農藥作風險管理之依據。美國代表指出該統計評估模式可使結果較接近實際狀況，但先決條件為需有充足之不同族群取食量資料及殘留量調查資料，以現階段各國之資料現況可能不適用為國際規範。IUPAC代表指出IUPAC已發表Acute Dietary Exposure Assessment之報告可供參考。大會決議由IUPAC、荷蘭、美國及澳洲合組工作小組提供資料於下次會議討論。

2. 加成毒性之安全評估方法(The methodology of cumulative risk assessment) - 農藥毒性評估一般以單一農藥進行。然部分農藥具相同之毒性作用機制者則消費者可能由不同途徑接觸而考慮毒性加成之問題。美國在對國內有機磷劑進行整體性評估時建立此評估模式，經十個步驟整合各農藥間之毒性反應相對值及殘留量計算其風險。經討論結果因該等評估與各國使用農藥之種類及殘留量有關，且評估者必須有很強之毒理基礎。本方法宜作為國內評估之參考，目前不宜列入國際規範。
3. 風險分析在研訂Codex MRL's 之運用(The application of risk analysis in the elaboration of Codex standards) – 風險分析為危害評估之標準操作程序，Codex Committee on General Principles在1997年訂定Codex風險分析準則，各食品標準委員會也相繼研擬其風險分析準則。本提案將交JMPR研究後提下次開會討論。

<u>議題六</u>、食品及動物飼料中農藥殘留標準 (Proposed Draft MRL in Foods and Feeds)

1. Codex MRL's

此為本會最主要之議題，共進行二個會議日。即討論論由JMPR 製備完成毒理及殘留量評估之藥劑，制訂其在不同作物中之最高殘留限量(Codex MRL's)，或依毒理評估結果或作物中殘留量評估結果取消其MRL's或ADI值。

Codex食品安全標準之研提須經過八個作業及審核程序：

Step1,step2,step3: 由委員會提議議題經決議為「Criteria for the Establishment of Work Priorities」，及交付相關委員會成立工作小組，草擬「proposed draft standard」。

Step 4: 由委員會秘書將proposed draft standard工正式送交各相關委員會討論及提供建議。

Step 5: 草案提送Excusive Committee 討論通過後定義為「draft standard」。意見送回草擬委員會討論修正。

Step 6: 委員會秘書將draft standard送交所有會員國及相關國際組織徵詢意見。

Step 7: 委員會秘書彙整意見送回研擬之委員會作最後之修正。

Step 8: CAC通過後公告為 Codex Standard.

食品標準定案後由委員會秘書處予以公告。該等標準法案在網站上均可查詢下載（http//fao.codex.alimentarius）。委員會之使用語言為英文、法文、西班牙文、阿拉伯文及中文（世界衛生組織增加俄文）。

因此CCPR研議農藥在作物中之Codex MRLs之程序為:

Step 1: 由CCPR建議農藥審議名單

Step 2: 由JMPR進行毒理資料審查，制訂ADI值。由殘留量值分析研議結果建議最高殘留限量值。

Step3: 建議值第一次提交各國政府提供意見。

Step4: CCPR進行第一次審查。

Step5:由CCPR轉交食品安全委員會(CAC)認定。
Step6: 建議值第二次提交各國政府提供意見。
Step7: CCPR進行最後審查。
Step7a: 建議值等待JMPR提供定案之ADI值。
Step7b: 建議值退回JMPR再評估。
Step7c: 建議值等待新資料補充。
Step8: 建議值定案為Codex MRL (CXL)。
Step5/8: 建議值無須經step 6, 7,直接定案。

本議題討論之藥劑MRL's為進入step4及step7之藥劑。大會並同意美國之提議暫緩有機磷劑之討論待其完成加成毒性評估。本次會議共討論六十二個藥劑之ADI或MRL值，摘要如表三。另一個藥劑之EMRL值及一個藥劑之guideline level也列入討論。

表三、食品及動物飼料中農藥殘留最高限量本會討論摘要

Code No	農藥名稱	決議事項
007	Captan 蓋普丹	1.討論其皮膚過敏性對採收者及消費者之影響，因非攝食議題不作結論。 2.增修訂多項MRL's
015	Chlormequat 克美素	1.應考慮其急性毒性。 2.增修訂多項MRL's
017	Chlorpyrifos 陶斯松	1.屬有機磷暫停增訂MRL's 2.已取消作為動物用藥 3.依2000年JMPR決議取消celery, egg plant, kale, kiwifruit, lettuce, head, mushrooms, potato, raspberries(red and black),tomatoes之MRL's.

Code No	農藥名稱	決議事項
020	2,4-D	1. 增修訂多項MRL's 2.取消blackberries, meat, milks, raspberries, sorghum, vaccinium berries, carberries之MRL's
022	Diazinon 大利松	1.屬有機磷暫停增訂MRL's 2.應考量其急毒性
027	Dimethoate 大滅松	1.大部分MRL's為最低檢出界限(LOD) 2.澳洲及巴西將提peppers,citrus之資料
037	Fenitrothion 撲滅松	保留部分MRL's其餘取消
039	Fenthion 芬殺松	1.EU評估中 2.取消meat, milk之CXLs
041	Folpet福爾培	待2002JMPR評估結果後再議
049	Malathion 馬拉松	1.屬有機磷暫停增訂MRL's 2.2003/2004JMPR將評估其急性毒性 3.取消pear之MRLs
053	Mevinphos 美文松	取消大部分作物之MRL's值僅保留cabbage head。
054	Monocrotophos 亞素靈	因產品停產，決定於下次會議取消所有MRL's值。
055	Omethcate 歐滅松	因產品停產，決定取消所有因單獨使用omethoate之作物MRL's值，保留由dimethoate代謝而來之MRL's但退為step3 or step6 。
056	2-Phenylphenol	保留citrus, pear之 MRLs
058	Parathion 巴拉松	因產品停產，決定取消所有CXLs及MRLs值。

Code No	農藥名稱	決議事項
059	Parathion-M 甲基巴拉松	美國及EC均已禁用。大會取消大部分MRLs包括米。
060	Phosalone 裕必松	待2002年JMPR評估後再研訂pome 及stone fruits 之 MRLs
061	Phosphomidon 福賜米松	因停止生產考慮下次會議取消所有MRLs
063	Pyrethrins 除蟲菊類	修訂部分MRLs
064	Quintozene	取消EC禁用之MRLs，增訂US建議者
065	Thiabendazole 腐絕	2002JECFA訂定acute RfD為0.1 mg/kg b.w.修訂部分MRLs,建議研訂citrus juice MRLs，提議熱帶水果只測果肉未獲通過。
072	Carbendazim 貝芬替	Residue定義為「sum of benomyl, carbendazim and thiophanate-methyl, expressed as carbendazim」取消avocado, celery, onion bulb and sweet potato之 MRLs因不再提供資料。
074	Disulfoton 二硫松	1.屬有機磷暫停增訂MRL' 2.考慮取消potapto, radish之MRLs
075	Propoxur安丹	廠商不再生產，CXL全部取消。
077	Thiophanate-methyl 甲基多保淨	取消全部CXLs，引用carbendazim者
079	Amitrole	無急慢性毒性考量，通過所有MRLs
082	Dichlorfluanid 益發靈	2002JMPR評估

Code No	農藥名稱	決議事項
083	Dicloran 大克爛	修訂MRLs
084	Dodine多寧	建議2002JMPR研訂Acute RfD
085	Fenamiphos 芬滅松	1.屬有機磷暫停增訂MRL' 2.2002JMPR研訂Acute RfD (因太低)
087	Dinocap 白粉克	研訂二組Acute RfD分別供一般人及嬰幼兒評估用
094	Methomyl 納乃得	部分作物Acute intake值得注意。取消大部分MRLs
096	Carbofuran 加保扶	暫停評估待2002年JMPR訂定Acute RfD。
100	Methamidophos 達馬松	待2002JMPR評估毒理資料2003評估殘留完成後再討論MRLs。
103	Phosmet益滅松	2002JMPR評估
105	Dithiocarbamates二硫代胺基甲酸鹽類	2004JMPR 討論因CS2產生干擾包葉菜類分析之方法改進。
106	Ethephon 益收生長素	2002年JMPR訂定Acute RfD。
110	Imazalil依滅列	2002年JMPR訂定Acute RfD。
117	Aldicarb 得滅克	Banana,potato有急毒性顧慮。廠商提供banana新使用方法及殘留資料於2002JMPR討論。
124	Mecarbam 滅加松	廠商不再生產，建議CXL全部取消。
132	Methiocarb 滅賜克	草莓MRL至step8

Code No	農藥名稱	決議事項
137	Bendiocarb 免敵克	廠商不再生產，建議下次CXLs全部取消。
144	Bitertanol 比多農	保留apricot MRL, tomato MRL to step 8
145	Carbosulfan 丁基加保扶	待2003JMPR研訂 acute RfD再議。
146	Cyhalothrin 賽洛寧	EC提議因廠商不再生產應取消CXLs, 大會決議情況明確後再議。
147	Methoprene 美賜平	修訂MRLs
151	Dimethipin 獲萎得	修訂MRLs
161	Paclobutazol 巴克素	廠商不再生產，建議下次CXLs全部取消。
163	Anilazine	廠商不再生產，建議CXLs全部取消。
165	Flusilazole 護矽得	保留所有CXLs
166	Oxydemeton-methyl滅多松	保留待2002JMPR評估 acute RfD及ADI再議。
167	Terbufos 托福松	取消barley MRL
170	Hexaconazole 菲克利	廠商不再生產，建議下次CXLs全部取消。
171	Profenofos 佈飛松	修訂MRL's
175	Glufosinate-AL 固殺草	1.修訂MRLs 2.注意基因作物之管理

Code No	農藥名稱	決議事項
177	Abemectin 阿巴汀	建議提供papaya residue資料至JMPR以研訂MRL
187	Clethodim	取消大部分動物及其產品之MRLs.分析方法應區分clethodim及 sethoxidam
188	Fenpropimorph 芬普福	1.EU較JMPR有較低之acute RfD 2.banana MRL 至 step8
193	Fenpyroximate 芬普蟎	修訂MRLs
194	Haloxyfop 合氯氟	廠商將提供新的資料及新劑型(haloxyfop-R)供JMPR 重新評估
196	Tebufenozide 得芬諾	2001JMPR評估結果在cabbage head, grapes, leafy vegetables之急性毒性值得注意。決議下次會議檢討所有作物之XCLs
199	Kresoxim-methyl克收欣	修訂MRLs
200	Pyriproxifen 百利普芬	所有MRLs至step 8

2. EMRLs

EMRLs(外加殘留限量)指非施用農藥而是吸收存在於環境中之物質而來。定義須已禁用且有污染食品影響健康或國際農產品貿易者才要列入訂定EMRL之名單。訂定標準依各地區各項食品之調查報告(Monitoring Data)評估，以介於0.2-0.5%之超量率為標準。

本會議討論畜肉中DDT之EMRL維持1999年會議依紐西蘭調查報告及建議所研訂之5 mg/kg (fat)。加拿大代表指出依其

國內取食暴露量評估1mg/kg對/6歲以下孩童即有風險。禽肉中DDT之EMRL也有爭議，建議下次會議討論。

3. Guideline Level

澳化甲烷（Methyl Bromide）雖為Montreal Protocol管制之藥劑，然因防疫需要故仍保留其Guideline Levels，不同之處理有不同之管制標準，詳見表四及說明。

表四、澳化甲烷（Methyl Bromide）之Guideline Levels

Commodity	GL (mg/kg)	
Bread and other cooked cereal products	0.01*	1
Cacao beans	5 Po	2
Cereal grains	5 Po	2
Cocoa products	0.01* Po	1
Dried fruits	0.01* Po	1
Dried fruits	2 Po	2
Milled cereals products	0.01* Po	1
Milled cereals products	1 Po	2
Peanut	0.01* Po	1
Peanut	10 Po	2
Tree nuts	0.01* Po	1
Tree nuts	10 Po	2

1. To appy to commodity at point of retail sale or when offered for consumption.
2. To apply at point of entry into a country and, in case of cereal for milling, if product has been freely exposed to air for a period of at last 24h after fumigation and before sampling.

議題七、農藥殘留分析方法準則(Matters Related to Methods of Analysis for Pesticide Residues)

本議題亦為本次會議之主要討論重點，包括三個討論案：

1. 農藥殘留分析實驗室標準操作準則(Guidelines on Good Laboratory Practice in pesticide residue)本準則之討論已進入step5，整套文件包含Codex已公告之五個準則及新增之一個準則:

（1） Recommended method of sampling for the determination of pesticide residues

（2） Portion of commodities to which Codex Maximum Residue Limits apply and which should be analysed.

（3） Explanatory notes on Codex Maximum Limits for pesticide residues

（4） Recommendations for methods of analysis of pesticide residues

（5） Codex classification of food and animal feed

（6） Codex guidelines on good practice in pesticide residue analysis (新增)

Codex農藥殘留實驗室操作準則重點有三：（1） the analyst；（2） basic resources;（3） the analysis. 其它品保品管及實驗室管理可參考ISO/IEC 17025及OECD GLP Guidance documents.本草案見附件三。

2. 農藥殘留分析方法採用建議(The introduction section of the recommended methods of analysis for pesticide residues)本草案建議選擇採用分析方法應考慮(1)國家或國際組織公告之分析方法或研究報告；(2)經實驗室間認証之方法；(3)可同時檢出多種農藥殘留之方法；(4)適用於多種作物分析且檢出界限低於最高殘留限量；(5)用於國家農藥管制單位實驗室之方法(見附件二)。

3. 各種農藥之農藥殘留分析方法彙編(Revision of the list of methods of analysis for pesticide residues)大會呼籲各國及國際組織提供農藥殘留分析方法，由FAO彙編供各國使用。

<u>議題八</u>、農藥評估優先順序(Establishment of Codex Priority Lists of Pesticides)

CCPR會議討論之農藥都經 JMPR先行評估，評估藥劑分為New Compounds(新藥劑)、Periodic Reevaluations(定期預先評估)及Evaluation(評估)三類。大會委請澳大利亞代表主持討論評估順序。由於JMPR每年能評估之農藥有限，因此以資料完整者及有可能因無標準而易引起國際貿易爭議者如持久性污染物為優先。對於「較安全」之農藥之定義應包括降低作物中殘留、作業環境安全、保障大眾健康、維護生態環境安全及加速在環境中之降解等。另針對農藥規格所新成立之專家委員會JMPS (Joint FAO/WHO Meeting on Pesticide Specification) 其評估結論也會作為JMPR建議之一部分。附件四為2002至2010年之藥劑評估名單。

<u>議題九</u>、Codex MRL制定程序引起之貿易障礙(Trade Vulnerabilities Arising from the Codex MRLs Establishment Process)

由於Codex MRLs制訂程序嚴謹，且JMPR每年評估藥劑有限，因此對於新農藥或擴大作物範圍之Codex MRLs未能即時研訂，致使許多以Codex MRLs為國家標準者因無限量標準而禁止檢出未有Codex MRLs農藥殘留之農產品或食品進口，造成國際貿易上非常大的困擾。大會委請美國及澳洲針對此問題進行原因分析及提出改善之方案。經討論後建議成立工作小組，就以「國家容許量」作為Codex暫行標準之可行性進

行研究。另也歡迎其它國際組織與JMPR合作，分擔其工作量，以提昇Codex MRLs研訂之時效。

議題十、辛香料殘留標準研訂案 (Consideration of Elaboration of MRLs for Spices)

南非及辛香料主要生產國提案因辛香料皆小面積種植，或間作於主要糧食作物間，很難依GAP之規範提出制訂 Codex MRLs之完整殘留量資料。另如乾辣椒(dried chilli)應屬加工食品不適合引用新鮮辣椒之安全值。且因辛香料之取食量及取食比率皆偏低，農藥殘留不致影響人體健康，因此建議能以地區性之調查資料（monitoring data）作為制訂MRLs之殘留量資料。大會原則同意，並要求JMPR研擬以monitoring data作為殘留量資料之評估準則，及南非收集及提供各國主要辛香料中農藥殘留之調查資料。

議題十一、Codex殘留標準作物分類修正 (The Revision of the Codex Classification of Foods and Animal Feeds)

大會委請荷蘭代表就Codex作物分類進行檢討及修正提大會討論。會中討論分類修正是否只作部分增訂或作大幅度修正。由於目前分類對於開發中國家之主要作物尤其是熱帶及亞熱帶之蔬菜水果太過粗糙，部分加工品、辛香料、非主要作物（minor crop）等作物也未含蓋，對於農產品國際貿易及作物安全標準之擴大解釋上皆造成困擾。因此多數出席代表認同大幅度修正，包括類別定義、作物歸屬分類、MRLs之共用等。會議決議由荷蘭代表與Codex秘書處收集與會各國及國際組織之意見，尤其是應顧及熱帶及亞熱帶開發中國家之特殊作物之農藥使用及殘留問題，再作修正後提下次會議討論。

議題十二、下次會議時間及地點(Date and place on next session)

下次（第三十五屆）CCPR會議將於2003年3月31日至4月5日於荷蘭鹿特丹召開。

議題十三、會議結論(Adoption of the Report)

會議最後一天由大會記錄及秘書作為會議結論，由與會人員討論報告內容及結論。會議結論將送2003年6月30日至7月5日在義大利羅馬召開之「Twenty-fifth Session of Joint FAO/WHO Food Standards Programme Codex Alimentarius Commission」(CAC)討論。

心得

一、聯合國食品標準委員會之組織及運作

聯合國為保障消費者健康及建立食品國際貿易之公平性，於1962年由世界糧農組織及衛生組織聯合成立食品標準委員會(Codex Alimentarius Commission（CAC）)(中國譯為食品法典委員會)以制訂食品安全之相關標準及執行規範。CAC下設Execusive Committee負責業務推行，並依食品、任務及地區分設各類委員會討論規範，分別為General Subject Committees、Commodity Committees、 Regional Coordinating Committees、ad hoc Intergovernmental Task Forces。詳列表五至表八。

表五、食品類別委員會(Commodity Committees）摘要說明

委員會名稱Codex Committee	主辦國	成立年
Codex Committee on Cocoa Products and Chocolate (CCCPC)	瑞士	1963
Codex Committee on Sugars （CCS）	英國	1964
Codex Committee on Fat and Oils（CCFO ）	英國	1964
Codex Committee on Processed Fruits and Vegetables （CCPFV）	美國	1964
Codex Committee on Fish and Fishery Products (CCFFP)	挪威	1966
Codex Committee on Natural Mineral Waters （CCNMW）	瑞士	1966
Codex Committee on Meat and Poultry Hygiene (CCMPH)	紐西蘭	1972
Codex Committee on Vegetable Proteins （CCVP）	加拿大	1980
Codex Committee on Cereal,Pulses and Lgumes （CCCPL）	美國	1980
Codex Committee on Fresh Fruits andVegetables （CCNMFFV）	墨西哥	1988
Codex Committee on Milk and Milk Products (CCMMP)	紐西蘭	1994

表六、共通議題委員會(General Subject Committees）摘要說明

委員會名稱Codex Committee	主辦國	成立年
Codex Committee on Food Hygiene 食品衛生委員會(CCFH)	美國	1964
Codex Committee on Food Additives and Contaminants 食品添加物及污染物委員會(CCFAC)	荷蘭	1964
Codex Committee on General Principles 一般準則委員會（CCGP）	法國	1965
Codex Committee on Food Labelling 食品標示委員會(CCFL)	加拿大	1965
Codex Committee on Methods of Analysis and Sampling 分析及採樣方法委員會(CCMAS)	匈牙利	1965
Codex Committee on Pesticide Residues 農藥殘留委員會(CCPR)	荷蘭	1966
Codex Committee on Nutrition and Foods for Special Dietary Uses 特殊食品及營養委員會（CCNFDU）	德國	1966
Codex Committee on Residues of Veterinary Drugs in Foods 食品中動物用藥殘留委員會(CCRVDF)	美國	1986
Codex Committee on Food Import and Export Inspection and Certification System 食品進出口檢驗及認証委員會(CCFICS)	澳洲	1992

表七、特殊任務委員會(ad hoc Intergovernmental Task Force）摘要說明

委員會名稱ad hoc Codex Intergovernmental Task Force	主辦國	執行期限
On Foods derived from Biotechnology（CCFBT）	日本	2000-2003
On Fruits and Vegetable Juices (CCFJ)	巴西	2000-2005
On Animal Feeding Practices (CCAF)	丹麥	2000-2003

表八、區域性合作委員會(Regional Coordinationg Committees）摘要說明

委員會名稱Codex Committee	主辦國	成立年
FAO/WHO Coordinationg Committee for Europe（CCEURO）	Slovak Republic	1965
FAO/WHO Coordinationg Committee for Africa（CCAFRICA）	Uganda	1974
FAO/WHO Coordinationg Committee for Latin America and the Caribbean（CCLAC）	Dominican Repub.	1976
FAO/WHO Coordinationg Committee for Asia（CCASIA）	Malaysia	1977
FAO/WHO Coordinationg Committee for North America and the Southwest Pacific（CCNASWP）	Canada	1990
FAO/WHO Coordinationg Committee for Near East（CCNEA）	Egypt	2001

聯合國食品標準委員會之會員為凡聯合國之會員國均可申請入會。至2001年7月計有165個會員國。會員國代表有權利參與會議及制訂各項準則。非會員國代表或其它國際組織若有興趣亦可申請以觀察員名義參加。然非會員國必須為聯合國之會員，國際組織則應事先申請，說明組織之性質與會議主題之相關性，並証明與會代表為該組織成員及其專業背景，始能與會。

CAC及各委員會之運作有其一定之作業流程。各項標準之制定或準則之研訂必須依一定之格式草擬內容及方案，並經八個程序審查後始能定案。對於各項名詞也有明確之定義，譬如其所討論之「食品（Food）」即指所有人類所食用之食物、飲料及口香糖包括原料、半成品及加工品，但不含藥品、化粧品及煙草。

食品標準之草案內容須包括以下items:

1. Name of the Standard
2. Scope

3. Description
4. Essential composition and quality factors
5. Food additives
6. Contaminants
7. Hygiene
8. Weights and measures
9. Labelling
10. Methods of analysis and sampling

二、國際農藥殘留標準委員會之定位及重要性

國際農藥殘留標準委員會(CCPR)於1966年在荷蘭海牙召開第一屆委員會，2002年為第卅四屆。國際農藥殘留委員會之主要任務有六：

1. 制訂農藥在單項食品或大類食品中之最高殘留限量。
2. 制訂農藥殘留在動物飼料中最高殘留限量，以涉及國際貿易及對人體健康有影響者為限。
3. 制訂化學性質與農藥相近之環境及工業污染物在食品中之最高殘留限量。
4. 制訂與作物及食品中農藥殘留安全標準相關之試驗規範，如分析準則、農作物分類等。
5. 決定JMPR評估農藥之優先順序。
6. 討論其它與農藥殘留有關之食品安全問題。

CCPR會議至今已完成近三百種農藥之毒理及殘留量評估，並制訂超過二千五百個最高殘留限量(MRLs)。本人與會多次之感想為會議過程嚴謹，議題事前之資料準備齊全，各會員國代表與會前作充分溝通，會議全程參與熱烈討論，而且對新的問題皆能即時回應並作出具體結論。據與大會主席Dr. W.H. Van Eck交談得知其在會前一個月必須全力詳讀資料及作準備

才能完全控制會議進度。與會人士對大會主席的主持會議能力及對農藥知識之廣博也備加讚賞。因此本會議結論提交聯合國食品安全委員會能作為食品安全管制標準，並為世界貿易組織引用為農畜產品交易之當然約定標準。世界各國也都非常重視此一會議，以美國為例，每次與會代表超過十名，包括環境保護署、食品藥物管理局、農業部及廠商代表，在國內則成立Codex工作小組，每月集會一次，針對會議相關議題進行討論。各國也利用每年一次集會的機會，在會場分送國內相關報告互作交流。本人除代表IUPAC國際組織介紹Agrochemicals and Environment Commission之工作摘要、主要出版報告外，同時也把握機會與各國與會代表交換農藥殘留容許量研訂之準則及了解各國對進出口農產品農藥殘留管制之原則。

三、農藥殘留調查及整體性評估之運用

本次會議討論農藥殘留分析方法、農作物分類及各國農藥殘留調查及評估資料，都可能影響農產品國際貿易之成敗。因此各國對於國內之農藥殘留檢驗方法之標準化及是符合國際規範均非常重視。

藥試所對於國內容許量研訂一直非常積極參與，於民國七十年發表「食用作物中農藥最高殘留容許量之訂定方法」，研訂一百廿種農藥在登記使用作物上之殘留容許量。七十五年九月召開「農產品中農藥殘留容許量研訂方法研討會」，會議通過容許量之訂定原則及程序，由衛生署委託藥毒所研訂後送交農委會函轉衛生署審查公告之。同年十二月衛生署公告殘留農藥安全容許量，正式取代施行十二年之暫行標準。本研訂方法及程序延用至今，至九十年九月共公告306種農藥計1242組安全容許量。同時為製備國內農藥最高殘留容許量，於1973年即開始計算各類農產品之取食量，並以作物分類之方式以含蓋各類作物，每五年更新一次。1998年依據衛生署於1997年完成之

「國民營養健康狀況變遷調查1993-1996」（NAHSIT 1993-1996）以二十四小時飲食回顧法調查國人膳食資料分析整理所得，製備十三歲至六十四歲計十二組之取食量資料，並考慮進口之農產品。另藥試所於1993年以Codex建議之模式完成國民經取食農藥殘留暴露量安全評估報告，其模式與本次會議討論之方式幾近相同，1998年進行為期二年全省經口取食農藥暴露量評估計畫。2001年起衛生署委託藥試所進行食品污染物國人總膳食調查計畫（Taiwan Total Diet Study），依本國國民之取食調查資料規畫採樣食物種類及烹調食譜，不同地區及季節採集生鮮食材或即食樣品，經食前處理後以食用狀態分析其中污染物之含量，再依不同年齡層及性別之國民取食量計算其檢出污染物中可能之暴露量，以評估國人經由攝食之危害風險。該等資料由於規畫時均已參考Codex之準則，因此研究成果應可運用於我國農產品之外銷諮商。

四、由Codex MRLs制訂流程檢討國內農藥管理制度

Codex制訂之各項食品安全標準及準則所以可以在WTO及國際貿易上得到各國之重視，其嚴謹之製備流程是主要的因素。由CCPR所建立之農藥殘留最高限量每一個數值均須經一定的審查步驟，而提供每一農藥MRL's值之JMPR每年皆須以一個月的時間來進行毒理資料及殘留量資料的評估資料，對於每一項資料製備的標準流程也有準則可遵循。我國農藥管理法於1972年公告實施，多年來為了扶持國內農藥產業及減輕廠商負擔，有關農藥登記所需提供之資料及田間試驗規範均與國際標準有相當之差距。我國成為WTO會員國後，中央農業主管人員參與國際談判後也體會到建立符合國際標準工作準則之重要性，開始要求國內各項試驗必要具有符合GAP或GLP的實驗數據，然卻忽略了這些數據的產生不是一個公文一句命令就可以做到，是須要多年的扶持及相關政令的配合才能達到。中

央主管農藥的官員不到十人，農藥管理業務的經費嚴重不足，地方政府農藥管理是兼辦業務，「農藥」二個字在國科會的科技研究項目中連名字都消失了因為「形象不好」。再再顯示台灣的農藥管理及研究如何不被重視。農藥登記相關試驗非由廠商在提出申請前自行完成而是依法由政府單位完成；農藥毒理資料審查由研究人員兼辦，無專責人員負全責；農藥殘留量田間試驗依附於藥效試驗下，無法以足夠的經費時間及人力製備完整之資料供 Codex 研擬我國主要作物之容許量。台灣農藥管理若無法由政策上進行全面改革，入關後門戶開放，各國品質不一之農藥充斥市面，農藥管理制度不易推行，農產品安全品質之提昇更會增加許多困難。

建議

一、 積極運用聯合國食品標準委員會制定之準則

聯合國食品標準委員會所制訂之各項食品安全標準及標示或管理準則雖未必與各國現行之食品安全衛生管理法相同，但參與起草及討論之會員國會將其國內之考量因素列入準則之規範中，且在食品進出口之協議上尤其是WTO會員國間Codex標準一向被為視為共同遵守之準則。我國雖非Codex之會員國但對Codex standard草擬作業之方式及其進行之議題仍應如其它國家一樣重視。許多國家都設置National Codex Office，定期討論Codex之各項準則對國內食品管理及食品貿易間之利害關係及影響。我國在加入WTO以後，台灣廣大之消費人口是許多國家傾銷食品之重要市場，同時政府也應協助農民及食品工廠積極拓展外銷市場，Codex standard的分析及運用不論對進口食品之查驗檢疫管理，或對出口食品之品管文件及貿易談判之內容，均佔非常之角色。CAC所研擬之2003至2007年中長期策略方案即希望Codex Standard對各國食品之生產、製造、管理及貿易達到最大之影響力，因此也針對消費者、食品業者及農民進行說帖呼籲其重視Codex standard對其產業及生活之影響。我國在因應WTO之食品產業策略上應重視Codex standard之影響，應成立專責部門，對Codex各委員會之會議決議與國內之現行法令立即進行利益評估，且應將公告之Codex guidelines及standard以中文版全文或摘錄之方式，介紹給政府相關單位、食品業者及生產者參考。

二、 尋求參與聯合國食品標準委員會議題討論之機會

我國因中國強力阻撓及非聯合國會員國，因而無法參與許多以政府為與會主體之會議，長久以往容易導致政府及人民忽視國際間之重要會議及其協議，而使我國在國際市場之開拓及

政府間之談判因不熟知國際通則而無法達到最有利之結果。現我國開放國內之食品市場，龐大的消費人口及消費能力是許多國家如美國及澳洲的貿易目標。我國應利用此等貿易談判之機會，要求該等國家以Codex委員會主辦國及視我國為WTO會員國為前題，尋求出席聯合國食品標準委員會或參與各項議題討論之機會。或將我國對Codex準則之意見及立場在雙方談判時反應給對方，尋求共識及支持，以保障我國之權益。對於進口食品之安全品質查驗及管理應研究Codex準則以尋求對消費者健康達到最大之保障又不違反國際貿易之公平性。對出口之食品或農產品面臨進口國因與我國管理制度不同而造成之貿易障礙也應尋求利用Codex之準則予以突破。使我國在進出口食品之管理及貿易上達到政府、業者及消費者最大利益及保障。

三、長期進行食品中農藥安全評估工作

農藥為植物保護的資材，合法合理使用才能降低對環境生態及國民健康的威脅。農藥殘留最高容許量或最高殘留限量是食品安全的標準，食品衛生管理上可作為農產品例行安全檢驗之法則。然農藥殘留是因使用而造成，因此市售農產品上之農藥殘留也因使用情形而異。以近來年藥檢局或藥試所蔬果農藥殘留抽檢結果，有農藥殘留者約佔40%，不符合容許量標準者約3%，遠低於每人每日可攝入量。為了解國民經取食可能受到農藥殘留的影響，及提供社會大眾一個合理的評估數據，成立長期性之食品污染物國人總膳食計畫，定期依國民取食農畜水產品之比率，依季節性及地區性進行市售樣品採集及經食前處理及烹煮後進行農藥殘留分析及安全評估。此等資料的建立除對國民食品安全品質作長期監測評估外，在食品安全管理上也是非常重要的國際性安全指標，應積極辦理。

四、增加農藥管理專責機構之人力資源

我國為配合加入WTO之需要，將大量之人力及財力投入檢防疫體系。且為迎合消費者對有機農產品之迷思及避免接觸敏感之農藥殘留問題，政府對整個植物保護的方向完全偏向配合有機農業栽培及非農藥防治方法的研發，植物保護大部分人力及業務偏向檢疫及防疫而忽略檢驗之重要性。植物保護工作是農作物的醫療體系，值得政府成立專一的管理機關來作整體性的規劃，忽略農藥合理使用的研究或逃避繁雜體系的建立都不能使台灣農業得到永續的發展。目前農藥管理必須改進的地方很多，包括修訂農藥管理法規，健全登記資料審查制度，重新檢討田間試驗規範，成品農藥品質維護及農藥使用對農民及消費者之保障等。政府應增列預算增加人力，附於專責機構有足夠的資源建立完善的農藥管理體系。

附件一、本報告引用專有名詞簡稱說明

簡稱	說明
Acute RfD	Acute Reference Dose
ADI	Acceptable Daily Intake
CAC	Codex Alimentarius Commission
CCNFSDU	Codex Committee on Nutrition and Foods for Special Dietary Uses
CCPR	Codex Committee on Pesticide Residues
CCRVDF	Codex Committee on Residues of Veterinary Drugs in Foods
CIPAC	Collaborative International Pesticides Analytical Council
CXL	Codex Maximum Residue Limit for Pesticide
EMRL	Extraneous Maximum Residue Limit
FAO	Food and Agriculture Organization
GAP	Good Agricultural Practice
GAP	Good Laboratory Paractice
GEMS	Global Environment Monitoring System
GLP	Good Laboratory Practice
IEDI	International Estimated Daily Intake
IUPAC	International Union of Pure and Applied Chemistry
JMPR	Joint FAO/WHO Meeting on Pesticide Residues
MRL	Maximum Residue Limit
SPS	Sanitary and Phytosanitary Measures
WHO	World Health Organization
WTO	World Trade Organization

附件二、**PROPOSED DRAFT AMENDMENTS TO THE INTRODUCTORY SECTION OF THERECOMMENDED METHODS OF ANALYSIS FOR PESTICIDE RESIDUES** (At Steps 5/8 of the Procedure)

1. INTRODUCTION

1.1 Scope

The analytical methods listed are those which may, from practical experience of the Codex Committee on Pesticide Residues, be considered for the determination of pesticide residues for regulatory purposes. The list, given in par.2, is not exhaustive and methods not mentioned in the list can also be applied, provided that they can be shown to produce valid results by the analyst using them.

1.2 Criteria for the selection of analytical methods

Whenever possible, the CCPR used the following criteria when selecting analytical methods:

i. Available through national or international standards organizations, books, manuals, open literature, the internet;

ii. collaboratively studied or known to have been validated in a number of laboratories. For single laboratory validated methods validation must have taken place according to Guidelines on Good Practice in Pesticide Residue Analysis as a minimum;

iii. capable of determining more than one residue, i.e. multi-residue methods;

iv. suitable for as many commodities as possible at concentrations at or below the specified MRLs;

v. applicable in a regulatory laboratory equipped with generally available analytical instrumentation. Preference was given to gas chromatography or high performance liquid chromatography as the separation step for the methods. Under certain conditions however, screening methods as defined in the Guidelines on Good Practice in Residue Analysis may be applicable. Screening methods are indicated in the list.

1.3 Application of methods

Before applying the methods it will always be necessary to validate the method and to demonstrate the competence of the analyst. There is a further need for regular verification of the performance of the method during use. Validation and performance verification are described in the Guidelines on Good Practice in Residue Analysis.

附件三、PROPOSED DRAFT REVISED GUIDELINES ON GOOD LABORATORY PRACTICE IN RESIDUE ANALYSIS

(At Step 5 of the Procedure)

FOREWORD

The Guidelines are intended to assist in ensuring the reliability of analytical results in checking compliance with maximum residue limits of foods moving in international trade. Reliable analytical results are essential to protect the health of consumers and to facilitate international trade. In addition to the present Guidelines, other relevant Codex recommendations elaborated by the Codex Committee on Pesticide Residues (CCPR) in the field of enforcement of Codex maximum limits for pesticide residues are as follows:

1 Recommended Method of Sampling for the Determination of Pesticide Residues (ref.: CAC/VOL XIII - Ed.2, Part VI or CAC/PR 5-1984), as amended with respect to meat and poultry (ALINORM 91/40; see also ALINORM 89/24A, Appd. II and ALINORM 91/24A Appd. VIII).

2 Portion of Commodities to which Codex Maximum Residue Limits Apply and which should be analysed (ref.: CAC/VOL XIII - Ed. l, Part V or CAC/PR6-1984).

3 Explanatory Notes on Codex Maximum Limits for Pesticide Residues (ref.: CAC/VOL XIII - Ed. 1, Part III).

4 Recommendations for Methods of Analysis of Pesticide Residues (ref.: CAC/VOL XIII Ed. 2 part VIII or CAC/PR 8-1984).

5 Codex Classification of Food and Animal Feed (ref.: CAC/PR4-1989).

CODEX GUIDELINES ON GOOD PRACTICE IN PESTICIDE RESIDUE ANALYSIS

1. INTRODUCTION

The Codex document ALINORM 76/24 Appendix IV (Report of the ad hoc Working Group on Methods of Analysis) contained the following statement:

"It was considered that the ultimate goal in fair practice in international trade depended, among other things, on the reliability of analytical results. This in turn, particularly in pesticide residue analysis, depended not only on the availability of reliable analytical methods, but also on the experience of the analyst and on the maintenance of 'good practice in the analysis of pesticides'." These guidelines define such good analytical practice and may be considered in three inter-related parts:

The Analyst (par. 2);
Basic Resources (par. 3);
The Analysis (par.4).
The requirements for facilities, management, personnel, quality assurance and quality control, documentation of results and raw data, and relevant subjects, which are considered as prerequisites for obtaining reliable and traceable results, are described in general in the ISO/IEC 17025 Standard (1999) and in a series of OECD GLP Guidance Documents, in the corresponding national laws and regulations. This Codex Guidelines, which are not exhaustive, outline the most essential principles and practices to be followed in the analysis of pesticide residues.

2. THE ANALYST

2.1 Residue analysis consists of a chain of procedures, most of which are known, or readily understood, by a trained chemist, but because the analyte concentrations are in the range ug/kg to mg/kg and because the analyses can be challenging, attention to detail is essential. The analyst in charge should have an appropriate professional qualification and be experienced and competent in residue analysis. Staff must be fully trained and experienced in the correct use of apparatus and in appropriate laboratory skills. In addition, each analyst using the method for the first time should complete the tests specified in sections 4.4.5 of Table 4 to demonstrate that they can use the method within the expected performance parameters established during method validation prior to analysis of samples. They must have an understanding of the principles of pesticide residue analysis and the requirements of Analytical Quality Assurance (AQA) systems. They must understand the purpose of each stage in the method, the importance of following the methods exactly as described and of noting any unavoidable deviations. They must also be trained in the evaluation and interpretation of the data that they produce. A record of training and experience must be kept for all laboratory staff.

2.2 When a laboratory for residue analysis is set up, the staff should spend some of their training period in a well established laboratory where experienced advice and training is available. If the laboratory is to be involved in the analysis for a wide range of pesticide residues, it may be necessary for the staff to gain experience in more than one expert laboratory.

3. BASIC RESOURCES

3.1 THE LABORATORY

3.1.1. The laboratory and its facilities must be designed to allow tasks to be allocated to well-defined areas where maximum safety and minimum chance of contamination of samples prevail. Laboratories should be constructed of, and utilise, materials resistant to chemicals likely to be used within them. Under ideal conditions, separate rooms would be designated for sample receipt and storage, for sample preparation, for extraction and clean-up and for instrumentation used in the determinative step. The area used for extraction and clean-up must meet solvent laboratory specifications and all fume extraction facilities must be of high quality. Sample receipt, storage and preparation should be handled in areas devoted to work at residue levels. Maintenance of sample integrity and adequate provisions for personal safety are priority requirements.

3.1.2 Laboratory safety must also be considered in terms of what is essential and what is preferable, as it must be recognised that the stringent working conditions enforced in residue laboratories in some parts of the world could be totally unrealistic in others. No smoking, eating, drinking or application of cosmetics should be permitted in the working area. Only small volumes of solvents should be held in the working area and the bulk of the solvents stored separately, away from the main working area. The use of highly toxic solvents and reagents should be minimised whenever possible. All waste solvent should be stored safely and disposed of both safely and in an environmentally friendly manner taking into account specific national regulations where available.

3.1.3 The main working area should be designed and equipped for utilisation of an appropriate range of analytical solvents. All equipment such as lights, macerators and refrigerators should be "spark free" or "explosion proof". Extraction, clean-up and concentration steps should be carried out in a well ventilated area, preferably in fume cupboards.

3.1.4 Safety screens should be used when glassware is used under vacuum or pressure. There should be an ample supply of safety glasses, gloves and other protective clothing, emergency washing facilities and a spillage treatment kit. Adequate fire fighting equipment must be available. Staff must be aware that many pesticides have acutely or chronically toxic properties and therefore, great care is necessary in the handling of standard reference compounds.

3.2 EQUIPMENT AND SUPPLIES

3.2.1 The laboratory will require adequate, reliable, supplies of electricity and water. Adequate supplies of reagents, solvents, gas, glassware, chromatographic materials, etc., of suitable quality are essential.

3.2.2 Chromatographic equipment, balances, spectrophotometers etc. must be serviced and calibrated regularly and a record of all servicing/repairs must be maintained for every such item of equipment. Calibration is essential for equipment performing measurements. Calibration curves and comparison with standards may suffice.

3.2.3 Regular calibration and re-calibration of measuring equipment must be done where the possible change in nominal value may significantly contribute to the uncertainty of the measurement. Balances and automated pipettes/ dispensers and similar equipment must be calibrated regularly. The operating temperatures of refrigerators and freezers should be continually monitored or be checked at specified intervals. All records should be kept up-to-date and retained.

3.2.4 Equipment used must be fit for purpose.

3.2.5 All laboratories require pesticide reference standards of known and acceptably high purity. Analytical standards should be available for all parent compounds for which the laboratory is monitoring samples, as well as those metabolites that are included in MRLs.

3.2.6 All analytical standards, stock solutions and reagents whose integrity could be influenced by degradative processes must be clearly labelled with an expiry date and stored under proper conditions. "Pure" reference standards must be kept under conditions that will minimise the rate of degradation, e.g. low temperature, exclusion of moisture, darkness. Equal care must be taken that standard solutions of pesticides are not decomposed by the effect of light or heat during storage or become concentrated owing to solvent evaporation.

4. THE ANALYSIS

The methods applied for the determination of pesticide residues should generally satisfy the criteria given in Table 3.

4.1 AVOIDANCE OF CONTAMINATION

4.1.1 One of the significant areas in which pesticide residue analysis differs significantly from macro-analysis is that of contamination and interference. Trace amounts of contamination in the final samples used for the determination stage of the method can give rise to errors such as false positive or false negative results or to a loss of sensitivity that may prevent the residue from being detected. Contamination may arise from almost anything that is used for, or is associated with, sampling, sample transport and storage, and the analyses. All glassware, reagents, organic solvents and water should be checked for possible interfering contaminants before use, by analysis of a reagent blank.

4.1.2 Polishes, barrier creams, soaps containing germicides, insect sprays, perfumes and cosmetics can give rise to interference problems and are especially significant when an electron-capture detector is being used. There is no real solution to the problem other than to ban their use by staff while in the laboratory.

4.1.3 Lubricants, sealants, plastics, natural and synthetic rubbers, protective gloves, oil from ordinary compressed air lines and manufacturing impurities in thimbles, filter papers and cotton-wool can also give rise to contamination.

4.1.4 Chemical reagents, adsorbents and general laboratory solvents may contain, adsorb or absorb compounds that interfere in the analysis. It may be necessary to purify reagents and adsorbents and it is generally necessary to use re-distilled solvents. Deionised water is often suspect; re-distilled water is preferable, although in many instances tap water or well water may be satisfactory.

4.1.5 Contamination of glassware, syringes and gas chromatographic columns can arise from contact with previous samples or extracts. All glassware should be cleaned with detergent solution, rinsed thoroughly with distilled (or other clean) water and then rinsed with the solvent to be used. Glassware to be used for trace analysis must be kept separate and must not be used for any other purpose.

4.1.6 Pesticide reference standards should always be stored at a suitable temperature in a room separate from the main residue laboratory. Concentrated analytical standard solutions and extracts should not be kept in the same storage area.

4.1.7 Apparatus containing polyvinylchloride (PVC) should be regarded as

suspect and, if shown to be a source of contamination, should not be allowed in the residue laboratory. Other materials containing plasticizers should also be regarded as suspect but PTFE and silicone rubbers are usually acceptable and others may be acceptable in certain circumstances. Sample storage containers can cause contamination and glass bottles with ground glass stoppers may be required. Analytical instrumentation ideally should be housed in a separate room. The nature and importance of contamination can vary according to the type of determination technique used and the level of pesticide residue to be determined. For instance contamination problems which are important with methods based on gas chromatography or high performance liquid chromatography, may well be less significant if a spectrophotometric determination is used, and vice versa. For relatively high levels of residues, the background interference from solvents and other materials may be insignificant in comparison with the amount of residue present. Many problems can be overcome by the use of alternative detectors. If the contaminant does not interfere with the residue determination, its presence may be acceptable.

4.1.8 Residues and formulation analyses must have completely separate laboratory facilities provided. Samples and sample preparation must be kept separate from the all residue laboratory operations in order to preclude cross contamination.

4.2 RECEPTION AND STORAGE OF SAMPLES

4.2.1 Every sample received into the laboratory should be accompanied by complete information on the source of the sample, on the analysis required and on potential hazards associated with the handling of that sample.

4.2.2 On receipt of a sample it must immediately be assigned a unique sample identification code which should accompany it through all stages of the analysis to the reporting of the results. If possible, the samples should be subject to an appropriate disposal review system and records should be kept.

4.2.3 Sample processing and sub-sampling should be carried out using procedures that have been demonstrated to provide a representative analytical portion and to have no effect on the concentration of residues present.

4.2.4 If samples cannot be analysed immediately but are to be analysed quickly, they should be stored at (1 - 5°C), away from direct sunlight, and

analysed within a few days. However, samples received deep-frozen must bekept at under -16 oC until analysis. In some instances, samples may require storage for a longer period before analysis. In this cases, storage temperature should be approximately - 20 ãC, at which temperature enzymic degradation of pesticide residues is usually extremely slow. If prolonged storage is unavoidable, the effects of storage should be checked by analysing fortified samples stored under the same conditions for a similar period. Useful information on storage stability of pesticide residues can be found in the annual publications of FAO titled: Pesticide Residues - Evaluations prepared by the FAO/WHO JMPR, and in the information submitted by the manufacturers for supporting the registration of their pesticides.

4.2.5 When samples are to be frozen it is recommended that analytical test portions be taken prior to freezing in order to minimise the possible effect of water separation as ice crystals during storage. Care must still be taken to ensure that the entire test portion is used in the analysis.

4.2.6 The containers must not leak. Neither the containers used for storage nor their caps or stoppers should allow migration of the analyte(s) into the storage compartment.

4.3 STANDARD OPERATING PROCEDURES (SOPS)

4.3.1 SOPs should be used for all operations. The SOPs should contain full working instructions as well as information on applicability, expected performance, internal quality control (performance verification) requirements and calculation of results. It should also contain information on any hazards arising from the method, from standards or from reagents.
4.3.2 Any deviations from a SOP must be recorded and authorised by the analyst in charge.

4.4 VALIDATION OF METHODS1

4.4.1 Guidelines have been published for validation of analytical procedures for various purposes. The principles described in this section are considered practical and suitable for validation of pesticide residue analytical methods. The guidance is not normative. The analyst should decide on the degree of validation required to demonstrate that the method is fit for the intended purpose, and should produce the necessary validation data accordingly. For instance, the requirements for testing for compliance with MRLs or providing data for intake estimation may be quite different.

4.4.2 An analytical method is the series of procedures from receipt of a sample to the production of the final result. Validation is the process of verifying that a method is fit for the intended purpose. The method may be developed in-house, taken from the literature or otherwise obtained from a third party. The method may then be adapted or modified to match the requirements and capabilities of the laboratory and/or the purpose for which the method will be used. Typically, validation follows completion of the development of a method and it is assumed that requirements such as calibration, system suitability, analyte stability, etc., have been established satisfactorily. When validating and using a method of analysis, measurements must be made within the calibrated range of the detection system used. In general, validation will precede practical application of the method to the analysis of samples but subsequent performance verification is an important continuing aspect of the process. Requirements for performance verification data are a sub-set of those required for method validation.
Proficiency testing (or other inter-laboratory testing procedures), where practicable, provides an important means for verifying the general accuracy of results generated by a method, and provides information on the betweenlaboratory variability of the results. However, proficiency testing generally does not address analyte stability or homogeneity and extractability of analytes in the processed sample.
Where uncertainty data are required, this information should incorporate performance verification data and not rely solely on method validation data.

4.4.3 Whenever a laboratory undertakes method development and/or method modification, the effects of analytical variables should be established, e.g. by using ruggedness tests, prior to validation. Rigorous controls must be exercised with respect to all aspects of the method that may influence the results, such as: sample size; partition volumes; variations in the performance of the clean-up systems used; the stability of reagents or of the derivatives prepared; the effects of light, temperature, solvent and storage on analytes in extracts; the effects of solvent, injector, separation column, mobile phase characteristics (composition and flow-rate), temperature, detection system, co-extractives etc. on the determination system. It is most important that the qualitative and quantitative relationship between the signal measured and the analyte sought are established unequivocally.

4.4.4 Preference should be given to methods having multi-residue and or multi-matrix applicability. The use of representative analytes or matrices is

important in validating methods. For this purpose, commodities should be differentiated sufficiently but not unnecessarily. For example, some products are available in a wide range of minor manufactured variants, or cultivated varieties, or breeds, etc. Generally, though not invariably, a single variant of a particular commodity may be considered to represent others of the same commodity but, for example, a single fruit or vegetable species must not be taken to represent all fruit or vegetables (Table 5). Each case must be considered on its merits but where particular variants within a commodity are known to differ from others in their effects on method performance, analyses of those variants are required. Considerable differences in the accuracy and precision of methods, especially with respect to the determination step, may occur from species to species.

4.4.4.1 Where experience shows similar performance of extraction and clean-up between broadly similar commodities/sample matrices, a simplified approach may be adopted for performance validation. A representative commodity may be selected from Table 5 to represent each commodity group having common properties, and used for validation of the procedure or method. In Table 5, the commodities are classified according to the Codex Classification2.

&^. Some examples of how far the validation data may be extended to other commodities are: cereals, validation for whole grains cannot be taken to apply to bran or bread but validation for wheat grain may apply to barley grain or wheat four;

&^ animal products, validation for muscle should not be taken to apply to fat or offal but validation for chickenfat may apply to cattle fat;

&^ fruit and vegetables, validation for a whole fresh product cannot be taken to apply to the dried product but validation for cabbages may apply to Brussels sprouts.

4.4.4.2 Similarly representative analytes may be used to assess the performance of a method. Compounds may be selected to cover physical and chemical properties of analytes that are intended to be determined by the method. The selection of representative analytes should be made based on the purpose and scope of analysis taking into account the following.

(a) The representative analytes selected should:

(i) possess sufficiently wide range of physico-chemical properties to include those ofrepresented analytes;

(ii) be those which are likely to be detected regularly, or for which critical decisions will bemade based on the results.

(b) As far as practicable, all analytes included in the initial validation process should be those which will have to be tested regularly and which

can be determined simultaneously by the determination system used.
(c) The concentration of the analytes used to characterise a method should be selected to cover the accepted limits (AL, see Glossary) of all analytes planned to be sought in all commodities. Therefore the selected representative analytes should include, among others, those which have high and low ALs. Consequently, the fortification levels used in performance testing with representative analytes/representative commodities may not necessarily correspond to the actual ALs.

4.4.5 Where appropriate data are already available, it may not be necessary for the analyst to perform all the tests. However, all required information must be included or referred to in the validation records. Table 1 provides an overview of parameters to be assessed for method validation according to the status of the method to be validated. Specific parameters and criteria to be assessed are listed in table 2. Parameters to be assessed should be restricted to those that are appropriate both to the method and to the purpose for which the particular method is to be applied. In many cases, performance characteristics with respect to several parameters may be obtained simultaneously using a single experiment. Test designs where different factors are changed at the same time (factorial experiment designs), may help to minimise the resources required. The performance of the analytical method should be checked, both during its development and during its subsequent use as indicated in section 4.5, according to the criteria given in Table 3.

4.4.6 Individual (single residue) methods should be fully validated with all analyte(s) and sample materials specified for the purpose, or using sample matrices representative of those to be tested by the laboratory.
4.4.7 Group specific methods (GSM) should be validated initially with one or more representative commodities and a minimum of two representative analytes selected from the group.

4.4.8 MRMs may be validated with representative commodities and representative analytes.

4.5 PERFORMANCE VERIFICATION

4.5.1 The main purposes of performance verification are to:
&^. monitor the performance of the method under the actual conditions prevailing during its use;
&^ ttake into account the effect of inevitable variations caused by, for instance, the composition of samples, performance of instruments,

quality of chemicals, varying performance of analysts and laboratoryenvironmental conditions;

&^ demonstrate that the performance characteristics of the method are broadly similar to those established at method validation, showing that the method is under "statistical control", and the accuracy anduncertainty of the results are comparable to those expected of the method. For this purpose, data obtained during method validation may be updated with data collected from performance verification during the regular use of the method.

The results of internal quality control provide essential information on the long term reproducibility and other performance characteristics of the method including the analytes and commodities which were incorporated during the extension of the method.

The basic performance characteristics to be tested and the appropriate test procedures are described in Table 2.

For effective performance verification, analyse samples concurrently with appropriate quality control analyses (blank and recovery determinations, reference materials, etc.). Control charts may be used to check for trends in performance of the method and to ensure that statistical control is maintained.

4.5.2 Construction and use of control charts.

4.5.2.1 Control charts may be a useful tool for demonstrating the performance of a method and the reproducibility of its selected parameter. One example for that is the control chart for recoveries. Its application depends on the tasks of the laboratory. When a large number of the same type of sample is analysed for the same active ingredients the control chart is based on the mean recovery and its standard deviation obtained during the regular use of the method. When small numbers of each of a large variety of samples are analysed for a great number of analytes with a multi-residue procedure the control charts cannot be applied in the usual way. In such cases, initially a control chart is constructed with the average recovery (Q) of representative analytes in representative matrices and the typical within-laboratory reproducibility coefficient of variation (CVAtyp), obtained as described below. . When the average recovery data and their coefficient of variation obtained during method validation for individual analyte/sample matrices are not statistically different, each can be considered as an estimate of the true recovery and precision of the method, and with their appropriate combination the typical recovery (Qtyp) and coefficient of variation (CVAtyp) of the method can be established and used for constructing the initial control chart. The warning and action

limits are Qtyp+-2*CVAtyp*Q and Qtyp+-3*CVAtyp*Q, respectively.

4.5.2.2 When the method is applied for regular analysis of various analyte/matrix combinations represented during the validation of the method, the individual recoveries are plotted on the chart. The reproducibility of the method during its normal use may be somewhat higher then obtained at the validation of the method. Therefore, if some of the recoveries are outside the warning limits or occasionally the action limits, but they are within the ranges calculated from the CVA values specified in Table 3, no special action is required.

4.5.2.3 Based on the additional 15-20 recovery tests performed during the regular use of the method, as part of performance verification, the mean or typical recovery and the CVA shall be recalculated and a new control chart constructed which reflects the long term reproducibility of the application of the method. The new parameters established must be within the acceptable ranges specified in Table 3.

4.5.2.4 If this is not achievable, for example in the case of particularly problematic analytes, results from samples should be reported as having poorer accuracy or precision than is normally associated with pesticide residues determination.

4.5.2.5 During the regular use of the method, if the average of the first >=10 recovery tests for a particular analyte/sample matrix is significantly different (P=0.05) from the average recovery obtained for the representative analyte/sample matrices, the Qtyp and CVtyp are not applicable. Calculate new warning and action limits for the particular analyte/sample matrix, applying the new average recovery and the CV values measured.

4.5.2.6 If performance verification data repeatedly fall outside the warning limits (1 in 20 measurements outside the limit is acceptable), the application conditions of the method must be checked, the sources of error(s) identified, and the necessary corrective actions taken before use of the method is continued.

4.5.2.7 If performance verification data are outside the refined action limits established according to 4.5.2.1 to 4.5.2.3 section, the analytical batch involved (or at least samples in which residues found are >=0.7 AL or 0.5 AL, for regularly and occasionally detected analytes, respectively) should be repeated.

4.5.2.8 Re-analysis of analytical portions of positive samples is another powerful way of performance verification. Their results can be used to calculate the overall within-laboratory reproducibility of the method (CVLtyp) in general or for a particular analyte/sample matrix. In this case, the CVLtyp will also include the uncertainty of sample processing, but will not indicate if the analyte is lost during the process.

4.6 CONFIRMATORY TESTS

4.6.1 When analyses are performed for monitoring or enforcement purposes, it is especially important that confirmatory data are generated before reporting on samples containing residues of pesticides that are not normally associated with that commodity, or where MRLs appear to have been exceeded. Samples may contain interfering chemicals that may be misidentified as pesticides. Examples in gas chromatography include the responses of electron-capture detectors to phthalate esters and of phosphorus-selective detectors to compounds containing sulphur and nitrogen. As a first step, the analysis should be repeated using the same method, if only one portion was analyzed initially. This will provide evidence of the repeatability of the result, if the residue is confirmed. It should be noted that the only evidence supporting the absence of detectable residues is provided by the performance verification data.

4.6.2 Confirmatory tests may be quantitative and/or qualitative but, in most cases, both types of information will be required. Particular problems occur when residues must be confirmed at or about the limit of determination but, although it is difficult to quantify residues at this level, it is essential to provide adequate confirmation of both level and identity.

4.6.3 The need for confirmatory tests may depend upon the type of sample or its known history. In some crops or commodities, certain residues are frequently found. For a series of samples of similar origin, which contain residues of the same pesticide, it may be sufficient to confirm the identity of residues in a small proportion of the samples selected randomly. Similarly, when it is known that a particular pesticide has been applied to the sample material there may be little need for confirmation of identity, although a randomly selected results should be confirmed. Where "blank" samples are available, these should be used to check the occurrence of possible interfering substances.

4.6.4 Depending upon the initial technique of determination, an alternative procedure which may be a different detection technique, may be necessary for verification of quantity. For qualitative confirmation (identity) the use

of mass-spectral data, or a combination of techniques based on different physico-chemical properties, is desirable (see Table 6).

4.6.5 The necessary steps to positive identification are a matter of judgement on the analyst's part and particular attention should be paid to the choice of a method that would minimise the effect of interfering compounds. The technique(s) chosen depend(s) upon the availability of suitable apparatus and expertise within the testing laboratory. Some alternative procedures for confirmation are given in Table 6.

4.7 MASS SPECTROMETRY

4.7.1 Residue data obtained using mass spectrometry can represent the most definitive evidence and, where suitable equipment is available, it is the confirmatory technique of choice. The technique can also be used for residue screening purposes. Mass spectrometric determination of residues is usually carried out in conjunction with a chromatographic separation technique to provide retention time, ion mass/charge ratio and ion abundance data simultaneously. The particular separation technique, the mass spectrometer, the interface between them and the range of pesticides to be analysed are usually interdependent and no single combination is suitable for the analysis of all compounds. Quantitative transmission of labile analytes through the chromatographic system and interface is subject to problems similar to those experienced with other detectors. The most definitive confirmation of the presence of a residue is the acquisition of its "complete" electron-impact ionisation mass spectrum (in practice generally from m/z50 to beyond the molecular ion region). The relative abundances of ions in the spectrum and the absence of interfering ions are important considerations in confirming identity. This mode of analysis is one of the least selective and interference from contaminants introduced during the production or storage of extracts should be scrupulously avoided. Mass spectrometer data systems permit underlying interference (eg column bleed) signals to be removed by "background subtraction" but this technique must be used with caution. Increased sensitivity can usually be achieved by means of limited mass range scanning or by selected ion monitoring but the smaller the number of ions monitored (especially if these are of low mass), the less definitive are the data produced. Additional confirmation of identity may be obtained (i) by the use of an alternative chromatographic column; (ii) by the use of an alternative ionisation technique (eg chemical ionisation); (iii) by monitoring further reaction products of selected ions by tandem mass spectrometry (MS/MS or MSn); or (iv) by monitoring selected ions at increased mass resolution.

For quantification, the ions monitored should be those that are the most specific to the analyte, are subject to least interference and provide good signal-to-noise ratios. Mass spectrometric determinations should satisfy similar analytical quality control criteria to those applied to other systems.

4.7.2 Confirmation of residues detected following separation by HPLC is generally more problematic than where gas chromatography is used. If detection is by UV absorption, production of a complete spectrum can provide good evidence of identity. However, UV spectra of some pesticides are poorly diagnostic, being similar to those produced by many other compounds possessing similar functional groups or structures, and co-elution of interfering compounds can create additional problems. UV absorption data produced at multiple wavelengths may support or refute identification but, in general, they are not sufficiently characteristic on their own. Fluorescence data may be used to support those obtained by UV absorption. LC-MS can provide good supporting evidence but, because the spectra generated are generally very simple, showing little characteristic fragmentation, results produced from LC-MS are unlikely to be definitive. LC-MS/MS is a more powerful technique, combining selectivity with specificity, and often provides good evidence of identity. LC-MS techniques tend to be subject to matrix effects, especially suppression, and therefore confirmation of quantity may require the use of standard addition or isotopically-labelled standards. Derivatisation may also be used for confirmation of residues detected by HPLC (paragraph 4.6.5.4).

4.7.3 In some instances, confirmation of gas chromatographic findings is most conveniently achieved by TLC. Identification is based on two criteria, Rf value and visualisation reaction. Detection methods based on bioassays (e.g. enzyme -, fungal groth or chloroplast inhibition) are especially suitable for qualitative confirmation as they are specific to certain type of compounds, sensitive and normally very little affected by the co-extracts. The scientific literature contains numerous references to the technique, the IUPAC Report on Pesticides (13) (Bátora, V., Vitorovic, S.Y., Thier, H. - P. and Klisenko, M.A.; Pure & Appl. Chem., 53, 1039-1049 (1981)) reviews the technique and serves as a convenient introduction. The quantitative aspects of thin-layer chromatography are, however, limited. A further extension of this technique involves the removal of the area on the plate corresponding to the Rf of the compound of interest followed by elution from the layer material and further chemical or physical confirmatory analysis. A solution of the standard pesticide should always be spotted on the plate alongside the sample extract to obviate any

problems of non-repeatability of Rf. Over-spotting of extract with standard pesticide can also give useful information. The advantages of thin layer chromatography are speed, low cost and applicability to heat sensitive materials; disadvantages include (usually) lower sensitivity and separation power than instrumental chromatographic detection techniques and need for more efficient cleanup in case of detections based on chemicals colour reactions.

4.8 DERIVATISATION

This area of confirmation may be considered under three broad headings.

(a) Chemical reactions

Small-scale chemical reactions resulting in degradation, addition or condensation products of pesticides, followed by re-examination of the products by chromatographic techniques, have frequently been used. The reactions result in products possessing different retention times and/or detector response from those of the parent compound. A sample of standard pesticide should be treated alongside the suspected residue so that the results from each maybe directly compared. A fortified extract should also be included to prove that the reaction has proceeded in the presence of sample material. Interference may occur where derivatives are detected by means of properties of the derivatising reagent. A review of chemical reactions which have been used for confirmatory purposes has been published by Cochrane, W.P. (Chemical derivatisation in pesticide analysis, Plenum Press, NY (1981)). Chemical reactions have the advantages of being fast and easy to carry out, but specialised reagents may need to be purchased and/or purified.

(b) Physical reactions

A useful technique is the photochemical alteration of a pesticide residue to give one or more products with a reproducible chromatographic pattern. A sample of standard pesticide and fortified extract should always be treated in a similar manner. Samples containing more than one pesticide residue may give problems in the interpretation of results. In such cases pre-separation of specific residues may be carried out using TLC, HPLC or column fractionation prior to reaction.

(c) Other methods

Many pesticides are susceptible to degradation/transformation by enzymes. In contrast to normal chemical reactions, these processes are very specific and generally consist of oxidation, hydrolysis or de-alkylation. The conversion products possess different chromatographic characteristics from the parent pesticide and may be used for confirmatory purposes if

compared with reaction products using standard pesticides.

4.9 THE CONCEPT OF LOWEST CALIBRATED LEVEL (LCL)

4.9.1 When the objective of the analysis is to monitor and verify the compliance with MRLs or other ALs, the residue methods must be sufficiently sensitive to reliably determine the residues likely to be present in a crop or an environmental sample at or around the MRL or AL. However, for this purpose it is not necessary to use methods with sufficient sensitivity to determine residues at levels two or more orders of magnitude lower. Methods developed to measure residues at very low levels usually become very expensive and difficult to apply. The use of LCL (see Glossary) would have the advantage of reducing the technical difficulty of obtaining the data and would also reduce costs. The following proposals for LCLs in various samples may be useful in enabling the residue chemist to devise suitable methods.

4.9.2 For active ingredients with agreed MRLs, the LCL can be specified as a fraction of the MRL. For analytical convenience this fraction will vary and could be as follows:

MRL (mg/kg)	LCL (mg/kg)
5 or greater	0.5
0.5 up to 5	0.1 increasing to 0.5 for higher MRLs
0.05 up to 0.5	0.02 increasing to 0.1 for MRLs
less than 0.05	0.5 x MRL

When the MRL is set at the limit of determination of the analytical method, the LCL will also be at this level.

4.10 EXPRESSION OF RESULTS

For regulatory purposes, only confirmed data should be reported, expressed as defined by the MRL. Null values should be reported as being less than lowest calibrated level, rather than less than a level calculated by extrapolation. Generally results are not corrected for recovery, and they may only be corrected if the recovery is significantly different from 100%. If results are reported corrected for recovery, then both measured and corrected values should be given. The basis for correction should also be reported. Where positive results obtained by replicate determinations (e.g. on different GC columns, with different detectors or based on different ions of mass spectra) of a single test portion (sub-sample), the lowest valid value obtained should be reported. Where positive results derive from analysis of multiple test portions, the arithmetic mean of the lowest valid values obtained from each test portion should be reported. Taking into

account, in general, a 20-30% relative precision, the results should be expressed only with 2 significant figures (e.g.: 0.11, 1.1, 11 and 1.1x102). Since at lower concentrations the precision may be in the range of 50%, the residue values below 0.1 should be expressed with one significant figure only.

附件四、PRIORITY LIST OF CHEMICALS SCHEDULED FOR EVALUATION AND RE-EVALUATION BY JMPR

The following are the tentative schedules to be evaluated by the FAO /WHO Joint Meeting on Pesticides Residues (JMPR) from 2002 to 2010

2002 JMPR

Toxicological evaluations	**Residue evaluations**
New compounds	***New compounds***
esfenvalerate (purified isomer of fenvalerate) Flutolanil	esfenvalerate (purified isomer of fenvalerate) flutolanil imidacloprid
Periodic re-evaluations	***Periodic re-evaluations***
acephate (095) lindane (048) metalaxyl-M (purified isomer of metalaxyl) methamidophos (100) oxamyl (126) tolyfluanid (162) triazophos (143)	carbaryl (008) deltamethrin (135) diflubenzuron (130) oxamyl (126) propagite (113) tolyfluanid (162)
Evaluations	***Evaluations***
carbofuran (096) –acute toxicity ethephon (106) –acute toxicity fenamiphos (085) –acute toxicity folpet (041) - acute toxicity oxydemeton methyl –acute toxicity	aldicarb (117) bitertenol (144) carbosulfan (145) carbofuran (096) cyfluthrin (157) phosmet (103) pyriproxifen (200)

2003 JMPR

Toxicological evaluations	**Residue evaluations**
New compound	***New compounds***
cyprodinil famoxadone methoxyfenozide pyraclostrobin	cyprodinil famoxadone methoxyfenozide pyraclostrobin
Periodic re-evaluations	***Periodic re-evaluations***
carbosulfan (145) cyhexatin (067)/azocyclotin (129) paraquat (057) terbufos (167) to be clarified	acephate (095) fenitrothion (037) lindane (048) methamidophos (100) pirimiphos-methyl (086)
Evaluations	***Evaluations***
dimethoate (027) - acute toxicity malathion (049) - acute toxicity pyrethrins (063)	carbendazim (072)/thiophanate-methyl dimethoate (027) dicloran (083) dodine (084) myclobutanil (181) pyrethrins (063)

2004 JMPR

Toxicological evaluations	**Residue evaluations**
New compounds	***New compounds***
fludioxinil trifloxystrobin	fludioxinil trifloxystrobin
Periodic re-evaluations	***Periodic re-evaluations***
glyphosate (158) phorate (112) pirimicarb (101) triadimefon (133) {should be evaluated triadimenol (168) {together	alpha- and zeta- cypermthrin cypermethrin (118) ethoprophos (149) metalaxyl-M paraquat (057) prochloraz (142) propineb
Evaluations	***Evaluations***
guazatine (114) fenpyroximate (193) – acute toxicity dhaloxyfop (194)	chlorpyrifos (017) dithiocarbamates (105) guazatine (114) malathion (047) oxydemeton-methyl (116) 2-phenylphenol (056)

2005 JMPR

Toxicological evaluations	**Residue evaluations**
New compounds	***New compounds***
dimethenamid-P fenhexamid indoxacarb novaluron	dimethenamid-P fenhexamid indoxacarb novaluron
Periodic re-evaluations	***Periodic re-evaluations***
benalaxyl (155) clofentezine (156) propamocarb (148) propiconazole (160)	cyhexatin (067)/ azocyclotin (129) endosulfan (032) methoprene (147) glyphosate (158) phorate (112) terbufos (167)
Evaluations	***Evaluations***
ethoxyquin (035)	ethoxyquin (035) oxydemeton-methyl (166) methiocarb (132)

2006 JMPR

Toxicological evaluations	**Residue evaluations**
Periodic re-evaluations	***Periodic re-evaluations***
cyromazine (169) flusilazole (165) procymidone (136) profenofos (171)	pirimicarb (101) triazophos (143) triadimefon (133) {should be evaluated triadimenol (168) {together

2007 JMPR

Toxicological evaluations	**Residue evaluations**
Periodic re-evaluations	***Periodic re-evaluations***
azinphos-methyl (002) cyfluthrin (157)/beta cyfluthrin fentin (040) vinclozolin (159)	clofentezine (156) permethrin (120) fpropamocarb (148) propiconazole (160) triforine (116)

2008 JMPR

Toxicological evaluations	**Residue evaluations**
Periodic re-evaluations	***Periodic re-evaluations***
bioresmethrin (93) buprofezin (173) chlorpyrifos-methyl (090) hexythiazox (176)	benelaxyl (155) cyromazine (169) *lambda*-cyhalothrin replacement of cyhalothrin flusilazole (165) procymidone (136) profenofos (171)

2009 JMPR

Toxicological evaluations	**Residue evaluations**
Periodic re-evaluations	***Periodic re-evaluations***
	azinphos-methyl (002) cyfluthrin/beta cyfluthrin (157) fentin (040) vinclozolin (159)

2010 JMPR

Toxicological evaluations	**Residue evaluations**
Periodic re-evaluations	***Periodic re-evaluations***
	bioresmethrin (93) buprofezin (173) chlorpyrifos-methyl (090) hexythiazox (176)

ANNEX I

CANDIDATE CHEMICALS FOR PERIODIC RE-EVALUATION –NOT YET SCHEDULED (confirmation of support required by November 2002)

amitraz (122) (residues only) ;dithianon (180);bifenthrin (178); ethion (034); cadusafos (174) ;fenvalerate (119) ;; chlorothalonil (081) ;fenbutatin oxide (109); cycloxydim (179) ;penconazole (182)
;

ANNEX II

CHEMICALS PROPOSED FOR PRIORITY LISTING BUT FOR WHICH FURTHER CONSIDERATION IS REQUIRED BEFORE A DECISION CAN BE MADE.

DDT (EMRLs); gentamicin; oxytetracycline
MRLs for various pesticides on spices based on monitoring data