codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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CX 4/60 **CL 2007/04-RVDF**
January 2007

TO: Codex Contact Points
Interested International Organizations

FROM: Secretary, Codex Alimentarius Commission
Joint FAO/WHO Food Standards Programme
00153 Rome, Italy

DEADLINE: **31 May 2007**

SUBJECT: **METHODS OF ANALYSIS FOR VETERINARY DRUG RESIDUES**

BACKGROUND

1. The 16th Session of the Codex Committee on Residues of Veterinary Drugs in Foods (CCRVDF), (Cancun, Mexico, 8-12 May 2006), agreed to forward to the 29th Session of the Codex Alimentarius Commission the Compendium of Methods of Analysis Identified as Suitable to Support Codex MRLs, recommended by the *ad hoc* Working Group on Methods of Analysis and Sampling (ALINORM 06/29/31, para. 120 and Appendix X). The list included fully validated methods; provisionally validated methods (single-laboratory validation only); and methods for some substances without MRLs which had been considered by JECFA and CCRVDF.

2. The Committee endorsed the recommendation of the *ad hoc* Working Group on Methods of Analysis and Sampling to ask the Codex Secretariat to issue a Circular Letter requesting that members and observers review the list of methods; review and update any addresses of contact point for information; advise of any methods for which they are no longer able to provide information; and provide information on substance and matrices for which validated methods are still required (ALINORM 06/29/31, para. 119).

3. The Committee agreed to reconvene the *ad hoc* Working Group on Methods of Analysis, under the co-Chairmanship of Canada and United Kingdom, prior to its next session to continue work on identification of suitable methods of analysis for residues of veterinary drugs in foods on the basis of information received in response to the Circular Letter (ALINORM 06/29/31, para. 121).

4. The 29th Session of the Codex Alimentarius Commission (Geneva, Switzerland, 3-7 July 2006) noted the existence of the Compendium developed by the Committee, without adopting it as a Codex text, and agreed that the Secretariat would make it publicly available in such a way as to make it most useful to Members. The CCRVDF was invited to revise the Compendium regularly to keep it updated (ALINORM 06/29/41, para. 196).

REQUEST FOR INFORMATION/COMMENTS

5. Governments and international organizations with observer status with Codex are invited to provide comments on the compendium of methods prepared by the 16th Session of CCRVDF (see Annex 1) and in particular to:

- review the content for any missing information:

- review the methods on which information was provided by their delegation and to update any addresses given as sources for the information;
- advise us of any methods on which they may no longer be able to provide information;
- provide information by using Annex 3 on any methods which will address the current gaps - either "validated method required" (see Annex 2) or supporting information to advance a method submitted by another delegation which has "provisional" status to "full recommendation". Annex 4 contains a list of some of the key performance requirements for methods used for screening, quantitative determination or confirmation;
- provide information on new methods which may provide alternatives to the analysis of compounds for which methods have already been recognized by the Committee.

Governments and interested international organizations wishing to provide information on the above should do so in writing to the U.S. Codex Office, Food Safety and Inspection Service - US Department of Agriculture, Room 4861 South Building, 14000 Independence Ave., SW - Washington, DC, 2025 USA (fax. +1 202 720 3157; e-mail: uscodex@usda.gov, ***preferably***) with a copy to the Secretary, Codex Alimentarius Commission, Viale delle Terme di Caracalla, 00153 Rome, Italy (fax +39 06 57054593; e-mail codex@fao.org - ***preferably***), **not later than 31 May 2007.**

Annex 1

COMPENDIUM OF METHODS OF ANALYSIS IDENTIFIED AS SUITABLE TO SUPPORT CODEX MRLs DEVELOPED BY THE CODEX COMMITTEE ON RESIDUES OF VETERINARY DRUGS IN FOODS[1]

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
Abamectin	Abamectin B_{1a}	Yes	LC	liver	cattle	100	5	45th JECFA	FAO Food & Nutrition Paper 41/8	full recommendation
				kidney	cattle	50	5			
				fat	cattle	100	5			
Albendazole	2-amino-benzimidazole, as parent drug equivalents	Yes	LC	liver	cattle	5000		3-lab trial, data provided to CCRVDF	*Chemistry Laboratory Guidebook*. United States Department of Agriculture, Food Safety and Inspection Service, Science Program, Washington, D.C. Contact: AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France	full recommendation
		Yes	LC	milk	cattle	100		info provided to 13th Meeting, CCRVDF		provisional recommendation
		Yes	LC	liver	sheep	5000		3-lab trial,data provided to CCRVDF	*Chemistry Laboratory Guidebook*. United States Department of Agriculture, Food Safety and Inspection Service, Science Program, Washington, D.C.	full recommendation

[1] This Compendium is made publicly available and it is to be regularly updated by the Codex Committee on Residues of Veterinary Drugs in Foods (see ALINORM 06/29/41, para. 196).

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
Azaperone	sum of azaperone and azaperol	Yes	LC	liver	pig	100	2	Data provided to CCRVDF by U.K.	Rose, M.D., and Shearer, G. (1992). *J. Chromatogr*. 624: 471-477	provisional recommendation
		Yes	LC	kidney	pig	100	2 (ref 1); 2.5 (ref. 2)	Data provided to CCRVDF by Netherlands (1, 2) and U.K. (3)	**1.** Keukens, H.J., and Aerts, M.M.L. (1989). *J. Chromatogr*. 464: 149-161. **2.** Van Ginkel, L.A., Schwillens, P.L.W.J., and Olling, M. (1989). *Anal. Chim. Acta* 225: 137- 146. **3.** Rose, M.D., and Shearer, G. (1992). *J. Chromatogr*. 624: 471-477.	provisional recommendation
				muscle	pig	60		info provided to 13th Meeting, CCRVDF	Contact: AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France	provisional recommendation
Benzylpenicillin	benzylpenicillin	Yes	LC	liver	All species	50	5	Original data submitted by Canada, confirmed by UK, Brazil, data provided to CCRVDF	Boison, J.O., Salisbury, C.D.C., Chan, W., and MacNeil, J.D. (1991). *J. Assoc. Offic. Anal. Chem*. 74: 497-501.	full recommendation
				kidney	All species	50	5			
				muscle	All species	50	5			
		Yes	GC	milk		4		Method provided to CCRVDF	Compilation of methods proposed as regulatory methods or used in regulatory programs in European Union, prepared for Working Group by France: Method for penicillins in milk by capillary gas chromatography from the	provisional recommendation

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									"Collection of official methods under Article 35 of the German Federal Foods Act"; see Meetschen, U., & Petz, M. (1991) *Z. Lebensm. Unters. Forsch*., 193: 337-343; see also *Bundesgesundhbl*. 36: 118-121 (1993).	
				kidney muscle fat milk						
Carazol	Carazol	Yes	LC	liver	pig	25	2	Data provided to CCRVDF by U.K. (1) and Germany (2)	**1.** Rose, M.D., and Shearer, G. (1992). *J. Chromatogr*. 624: 471-477. **2.** Rudolph, M., and Steinhart, H. (1987). *J. Chromatogr*. 392: 371-378.	provisional recommendation
				kidney		25	0.3		**1.** Keukens, H.J., and Aerts, M.M.L. (1989). J. Chromatogr. 464: 149-161. **2.** Rose, M.D., and Shearer, G. (1992). J. Chromatogr. 624: 471-477. 3. Rudolph, M., and Steinhart, H. (1987). *A second laboratory evaluation of this method was provided by the UK*	provisional recommendation

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									to the 12th CCRVDF. J. Chromatogr. 392: 371-378. **4.** Vogelsgang, J. (1989). *Deutsch. Lebensm.Rndsch*. 85: 251-258.	
Ceftiofur	desfuroylceftiofur acetamide	Yes	LC	liver	cattle	2000	100	Data provided to 12th Meeting, CCRVDF, and 47th JECFA	Report of 12th Meeting, CCRVDF; FAO Food & Nutrition Paper 41/8	provisional recommendation
				kidney		6000	50	data provided to CCRVDF and to 47th JECFA	FAO Food & Nutrition Paper 41/8; see also Hornish, R.E., Hamlow, P.J., & Brown, S.A.. (2003) *J. AOAC Int*. 86: 30-38 for report of 4-laboratory trial of method for analysis of kidney and muscle (cattle and pig) and milk.	full recommendation
				muscle		1000	50			
				fat		2000		Data provided to 12th Meeting, CCRVDF, and 47th JECFA	Report of 12th Meeting, CCRVDF; FAO Food & Nutrition Paper 41/8; method LOD given as 50 µg/kg, LOQ not reported.	provisional recommendation

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
				milk		100	50	data provided to CCRVDF and to 47th JECFA	FAO Food & Nutrition Paper 41/8; see also Hornish, R.E., Hamlow, P.J., & Brown, S.A.. 2003. *J. AOAC Int*. 86: 30-38 for report of 4-laboratory trial of method for analysis of kidney and muscle (cattle and pig) and milk.	full recommendation
				liver	pig	2000	1000	Data provided to 12th Meeting, CCRVDF, and 47th JECFA	Report of 12th Meeting, CCRVDF; FAO Food & Nutrition Paper 41/8; method LOD given as 50 µg/kg, LOQ not reported. UK reported method evaluation to 12th CCRVDF indicating acceptable accuracy and precision from 1000 to 4000 µg/kg from pig liver, but recoveries in range of 60%	provisional recommendation
				kidney		6000	100	data provided to CCRVDF and to 47th JECFA	FAO Food & Nutrition Paper 41/8; see also Hornish, R.E., Hamlow, P.J., & Brown, S.A.. 2003. *J. AOAC Int*. 86: 30-38 for report of 4-laboratory trial of method for analysis of kidney and muscle (cattle and	full recommendation
				muscle		1000	30			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									pig) and milk.	
				fat		2000		Data provided to 12th Meeting, CCRVDF, and 47th JECFA	Report of 12th Meeting, CCRVDF; FAO Food & Nutrition Paper 41/8; method LOD given as 50 µg/kg, LOQ not reported.	provisional recommendation
Chlortetracycline, oxytetracycline, tetracycline	Parent drugs, singly or in combination	Yes	LC	liver	cattle	600	50-100	data provided to CCRVDF	AOAC 995.09 extension (Canada)	full recommendation
				kidney		1200	200-250	**1.** AOAC collaborative study, data provided to CCRVDF; **2.** 6-lab method trial, data provided to CCRVDF by Poland	**1.** MacNeil JD, Martz VK, Korsrud GO, Salisbury CDC, Oka H, Epstein RL, Barnes CJ. (1996) *J. AOAC Int.* 79: 405 - 417. See also AOAC Official Method 995.09: Chlortetracycline, Oxytetracycline and Tetracycline in Edible Animal Tissues. (1996). *AOAC Official Methods of Analysis, 16th edition, Supplement March 1996*. AOAC International, Gaithersburg, MD. **2.** Posyniak, A. Zmudzki, J., Ellis, R.L., Semeniuk, S., & Niedzielska, J. (1999) J. AOAC Int. 82: 862-865.	full recommendation
				muscle		200	100-200			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
		Yes	LC	milk		100	15	AOAC collaborative study, data provided to CCRVDF	Carson, MC, & Breslyn, W. (1996) *J. AOAC Int*. 79: 29 - 42. See also AOAC Official Method 995.04 : Multiple Tetracycline Residues in Milk. (1996). AOAC Official Methods of Analysis, 16th edition, Supplement March 1996. AOAC International, Gaithersburg, MD.	full recommendation
				liver	sheep	600				provisional recommendation
				kidney		1200				
				muscle		200	100-200			
				milk		100				
				liver	pig	600		data provided to CCRVDF	AOAC 995.09 extension (Canada)	full recommendation
				kidney		1200	200 - 600	AOAC collaborative study, data provided to CCRVDF	MacNeil JD, Martz VK, Korsrud GO, Salisbury CDC, Oka H, Epstein RL, Barnes CJ. (1996) *J. AOAC Int.* 79: 405 - 417. See also AOAC Official Method 995.09: Chlortetracycline, Oxytetracycline and Tetracycline in Edible Animal Tissues. (1996). AOAC Official Methods of Analysis, 16th edition, Supplement March	full recommendation
				muscle		200	100-200			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									1996. AOAC International, Gaithersburg, MD.	
				liver	poultry	600				provisional recommendation
				kidney		1200				
				muscle		200				
				eggs		400				
		Yes		muscle	giant prawn	100		JECFA review; Data provided to CCRVDF	AOAC 995.09 by extension (validation data provided by Thailand to JECFA and CCRVDF); additional data provided by Thailand to 16th CCRVDF	full recommendation
	OTC only			muscle	fish	200		58th JECFA; Data provided to CCRVDF	FAO Food & Nutrition Paper 41/14; additional data provided by Canada to 16th CCRVDF	full recommendation
Clenbuterol	Clenbuterol	Yes	GC/MS	liver	cattle	0.6		47th JECFA	FAO Food & Nutrition Paper 41/9	full recommendation
				kidney		0.6				
				muscle		0.2				
				fat		0.2				
				milk		0.05				
				liver	horse	0.6				
				kidney		0.6				
				muscle		0.2				
				fat		0.2				
Closantel	Closantel	Yes		liver	cattle	1000		info provided to 13th Meeting,	Contact: AFSSA-LERMVD, Javene, BP090203-35302,	provisional recommendation

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
								CCRVDF	Fougeres, France	
		Yes	LC	muscle		1000			Michiels, M., Meuldermans, W., and Heykants, J. (1987). *Drug Metab. Rev*. 18: 235-251.	full recommendation
		Yes	LC	muscle	sheep	1500			Michiels, M., Meuldermans, W., and Heykants, J. (1987). *Drug Metab. Rev*. 18: 235-251.	full recommendation
Cyfluthrin	Cyfluthrin	Yes	GC	liver	cattle	20	10	48th JECFA	FAO Food & Nutrition Paper 41/10	full recommendation
				kidney		20	10			
				muscle		20	10			
				fat		200	10			
				milk		40	5			
Cyhalothrin	Cyhalothrin	Yes	GC	liver	cattle	20	10	54th JECFA	FAO Food & Nutrition Paper 41/13	full recommendation
				kidney		20	10			
				muscle		20	10			
				fat		400	10			
				milk		30	10			
				liver	sheep	50	50	54th JECFA, revised by 62nd JECFA		
				kidney		20	10	54th JECFA		
				muscle		20	10			
				fat		400	10			
				liver	pig	20	10			
				kidney		20	10			
				muscle		20	10			
				fat		400	10			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
Cypermethrin	Cypermethrin	Yes	GC	liver	sheep	50	10	58th JECFA; revised by 62nd JECFA	FAO Food & Nutrition Papers 41/14 & 41/16	full recommendation
				kidney		50	10			
				muscle		50	10			
				fat		100	10			
α-Cypermethrin	α-Cypermethrin	Yes	GC	liver	cattle	50	10	58th JECFA; revised by 62nd JECFA	FAO Food & Nutrition Papers 41/14 & 41/16	full recommendation
				kidney		50	10			
				muscle		50	10			
				fat		1000	100			
				milk		100	10			
				liver	sheep	50	10			
				kidney		50	10			
				muscle		50	10			
				fat		1000	100			
Danofloxacin	Danofloxacin	Yes	LC	liver	cattle	400	10	48th JECFA; info also provided to 13th CCRVDF by France.	FAO Food & Nutrition Paper 41/10; see also Report of 12th & 13th Meetings, CCRVDF. Contact for method provided to CCRVDF: AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France	full recommendation
				kidney		400	10			
				muscle		200	10			
				fat		100	10			
				liver	pig	50	10			
				kidney		200	10			
				muscle		100	10			
				fat		100	10			
				liver	chicken	400	10			
				kidney		400	10			
				muscle		200	10			
				fat		100	10			
Deltamethrin	Deltamethrin	Yes	GC	liver	cattle	50	15	52nd JECFA	FAO Food & Nutrition Paper	full recommendation

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									41/12	
				kidney		50	15			
				muscle		30	15			
				fat		500	45			
				milk		30	15			
		Yes	GC	liver	chicken	50	15	52nd JECFA	FAO Food & Nutrition Paper 41/12	full recommendation
				kidney		50				
				muscle		30	15			
				fat		500	45			
				eggs		30	15			
				muscle	salmon	30	2			
Diclazuril	Diclazuril	Yes	GC	liver	sheep	3000	10	45th JECFA	FAO Food & Nutrition Paper 41/8	full recommendation
				kidney		2000	10			
				muscle		500	10			
				fat		1000	10			
			GC, LC	liver	poultry	3000	10, 50			
				kidney		2000	10, 50			
				muscle		500	10, 50			
				fat		1000	10, 50			
			LC	liver	rabbit	3000	50			
				kidney		2000	50			
				muscle		500	50			
				fat		1000	50			
Dicyclanil	Dicyclanil	Yes	LC	liver	sheep	125	10	60th JECFA	FAO Food & Nutrition Paper 41/15	full recommendation
				kidney		125	10			
				muscle		150	10			
				fat		200	10			
Dihydrostreptomycin, streptomycin	Dihydrostreptomycin, streptomycin	Yes	LC	liver	cattle	600	200 - 300	58th JECFA; info provided to CCRVDF	FAO Food & Nutrition Paper 41/14; see also Gerhardt, G.C.,	full recommendation
				kidney		1000	200 -			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
							300		Salisbury, C.D.C., & MacNeil, J.D. (1994) *J. AOAC Int.* 77: 334-337; data provided to CCRVDF by Canada, 2nd laboratory verification of performance reported by UK. For additional methods, contact AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France; Australian Government Analytical Laboratories, GPO Box 1844, Canberra ACT 2601, Australia.	
				muscle		600	200 - 300			
				fat		600	200 - 300			
				milk		200	50			
				liver	pig	600	200 - 300			
				kidney		1000	200 - 300			
				muscle		600	200 - 300			
				fat		600	200 - 300			
				liver	sheep	600	200 - 300			
				kidney		1000	200 - 300			
				muscle		600	200 - 300			
				fat		600	200 - 300			
				milk		200	50			
				liver	chicken	600	200 - 300			
				kidney		1000	200 - 300			
				muscle		600	200 - 300			
				fat		600	200 - 300			
Diminazene	Diminazene	Yes	LC	liver	cattle	12000	300	42nd JECFA	FAO Food & Nutrition Paper	provisional recommendation
				kidney		6000	300			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
				muscle		500	300		41/6: info on method for cattle muscle, liver, kidney, fat and milk provided to 10th CCRVDF.	
				milk		150	150			
Doramectin	Doramectin	Yes	LC	liver	cattle	100	2.5	45th JECFA	FAO Food & Nutrition Paper 41/8	full recommendation
				kidney		30	2.5			
				muscle		10	2.5			
				fat		150	5			
		Yes	LC	milk		15	3	62nd JECFA	FAO Food & Nutrition Paper 41/16	
				liver	pig	100	2.5	52nd JECFA	FAO Food & Nutrition Paper 41/12	
				kidney		30	2.5			
				muscle		5	2.5			
				fat		150	5			
									NOTE: For regulatory methods provided to CCRVDF contact AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France; Australian Government Analytical Laboratories, GPO Box 1844, Canberra ACT 2601, Australia.	
Eprinomectin	Eprinomectin	Yes	LC	liver	cattle	2000	2	50th JECFA	FAO Food & Nutrition Paper 41/11;	full recommendation
				kidney		300	2		NOTE: For regulatory method provided to	
				muscle		100	2			
				fat		250	2			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
				milk		20	1		CCRVDF contact Australian Government Analytical Laboratories, GPO Box 1844, Canberra ACT 2601, Australia.	
Febantel, fenbendazole, oxfendazole	sum, expressed as oxfendazole sulfone equivalents	Yes	LC	liver	cattle	500	5	50th JECFA	FAO Food & Nutrition Paper 41/11	full recommendation
				kidney		100	5			
				muscle		100	5		See also Chemistry Laboratory Guidebook. United States Department of Agriculture, Food Safety and Inspection Service, Science Program, Washington, D.C. (data provided to CCRVDF by United States). Additional contact for method for analysis of milk provided to CCRVDF: AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France.	
				fat		100	5			
				milk		100	5			
				liver	sheep	500	5			
				kidney		100	5			
				muscle		100	5			
				fat		100	5			
				liver	pig	500	5			
				kidney		100	5			
				muscle		100	5			
				fat		100	5			
				liver	horse	500	5			
				kidney		100	5			
				muscle		100	5			
				fat		100	5			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
				liver	goat	500	5			
				kidney		100	5			
				muscle		100	5			
				fat		100	5			
Fluazuron	Fluazuron	Yes	LC	liver	cattle	500	20	48th JECFA	FAO Food & Nutrition Paper 41/10	full recommendation
				kidney		500	20			
				muscle		200	20			
				fat		7000	10			
Flubendazole	Flubendazole	Yes	LC	liver	pig	10			Marti, A.M., Mooser, A.E., and Koch, H. (1990). *J. Chromatogr*. 498: 145-157; data provided to CCRVDF by Switzerland.	provisional recommendation
				muscle		10				
				liver	poultry	500				
				muscle		200				
				eggs		400				
Flumequine	Flumequine	Yes	LC	liver	cattle	500	50	48th JECFA	FAO Food & Nutrition Paper 41/10	provisional recommendation
		Yes	LC	kidney		3000	50	54th JECFA	FAO Food & Nutrition Paper 41/13	full recommendation
				muscle		500	50			
				fat		1000	25			
				liver	pig	500	50			
				kidney		3000	50			
				muscle		500	50			
				skin/fat		1000	50			
				liver	sheep	500	5		Additional supporting data provided to CCRVDF from compilation of methods proposed as regulatory methods or used in regulatory programs in	
				kidney		3000	5			
				muscle		500	5			
				fat		1000	5			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									European Union, prepared for Working Group by France.	
		Yes	LC	liver	chicken	500	100	48th JECFA	FAO Food & Nutrition Paper 41/10	provisional recommendation
		Yes	LC	kidney		3000	100	54th JECFA	FAO Food & Nutrition Paper 41/13	full recommendation
				muscle		500	25		additional info on regulatory method provided to CCRVDF, contact AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France.	
				fat		1000	50			
Gentamicin	Gentamicin	Yes	LC	liver	cattle	2000	200	50th JECFA	FAO Food & Nutrition Paper 41/11	full recommendation
				kidney		5000	1000			
				muscle		100	100			
				fat		100	100			
				milk		200	100			
				liver	pig	2000	200		NOTE: Additional info on regulatory method for pork kidney provided to CCRVDF, contact AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France. A 2nd laboratory evaluation of the method of McLaughlin, L. & Henion, J. (1994) *Biological Mass Spectrometry* 23: 417-429 for	
				kidney		5000	1000			
				muscle		100	100			
				fat		100	100			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									analysis of pig liver was reported to the 12th CCRVDF by the UK.	
Imidocarb	Imidocarb	Yes	LC	liver	cattle	2000	100	50th JECFA	FAO Food & Nutrition Paper 41/11; report notes that additional validation of method for tissues for species other than cattle required.	full recommendation
				kidney		1500	100			
				muscle		300	50			
				fat		50	50			
				milk		50	10			
Isometamidium	Isometamidium			liver	cattle	500		data provided to CCRVDF	Data provided on performance of drug sponsor's method.	provisional recommendation
				kidney		1000				
				muscle		100				
				fat		100				
				milk		100				
Ivermectin	Ivermectin B_{1a}	Yes	LC	liver	cattle	100	2	data provided to CCRVDF	***1.*** *Chemistry Laboratory Guidebook*. United States Department of Agriculture, Food Safety and Inspection Service, Science Program, Washington, D.C. **2.** Tway, P.C., Wood, J.S., Jr., and Downing, G.V. (1981). *J. Agr. Food Chem.* 29: 1059-1063. **3.** Salisbury, C.D.C. (1993) J. AOAC Int. 76: 1149-1151, submitted by Canada, 2nd laboratory	full recommendation
				fat		40	2			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									confirming data on method performance provided by UK.	
		Yes	LC	milk		10		54th JECFA; info provided to 13th CCRVDF	Method considered by JECFA requires validation; info on regulatory method provided to CCRVDF by France, contact AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France.	provisional recommendation
		Yes	LC	liver	pig	15	2	data provided to CCRVDF	***1.*** *Chemistry Laboratory Guidebook*. United States Department of Agriculture, Food Safety and Inspection Service, Science Program, Washington, D.C. **2.** Tway, P.C., Wood, J.S., Jr., and Downing, G.V. (1981). *J. Agr. Food Chem*. 29: 1059-1063. **3.** Salisbury, C.D.C. (1993) *J. AOAC Int*. 76: 1149-1151, submitted by Canada, 2nd laboratory confirming data provided by UK.	full recommendation
				fat		20	2			
				liver	sheep	15	2			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
Levamisole	Levamisole	Yes	LC	liver	cattle	100		Data provided to CCRVDF by Denmark.	**1.** Danish National Food Agency, Method F40251. Data for cattle, pig and sheep liver only. NOTE: For additional regulatory method provided to CCRVDF contact Australian Government Analytical Laboratories, GPO Box 1844, Canberra ACT 2601, Australia.	provisional recommendation
		Yes	GC (Method 2)	liver	pig	100		Data provided to CCRVDF by Denmark and US.	**1.** Danish National Food Agency, Method F40251. Data for cattle, pig and sheep liver only. **2.** Chemistry Laboratory Guidebook. United States Department of Agriculture, Food Safety and Inspection Service, Science Program, Washington, D.C. Data for pig liver only.	provisional recommendation
		Yes		liver	sheep	100		Data provided to CCRVDF by Denmark.	Danish National Food Agency, Method F40251. Data for cattle, pig and sheep liver only.	provisional recommendation
Lincomycin	Lincomycin	Yes	GC/MS	milk	cattle	150	15	54th JECFA	FAO Food & Nutrition paper 41/13	full recommendation
				liver	pig	500	60			
				kidney		1500	60			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
				muscle		200	17			
				fat		100	17			
				liver	chicken	500	17			
				kidney		500	17			
				muscle		200	17			
				fat		100	17			
Melengesterol acetate	Melengesterol acetate	Yes	LC/MS	liver	cattle	5	0.5	58th JECFA	FAO Food & Nutrition Paper 41/14	full recommendation
				fat		8	0.5		NOTE: For regulatory method for cattle muscle and fat provided to CCRVDF contact RIVM, Community Reference Laboratory, Antonie van Leeuwenhoeklaan 9, 3720 BA Bilthoven, the Netherlands.	
Moxidectin	Moxidectin	Yes	LC	liver	cattle	100	10	45th JECFA; data also provided to CCRVDF	FAO Food & Nutrition Paper 41/8	full recommendation
				kidney		50	10			
				muscle		20	10			
				fat		500	10			
				liver	sheep	100	10		Information on regulatory methods for residues in liver of various species supplied to CCRVDF contact: AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France; Australian Government Analytical Laboratories, GPO Box 1844,	
				kidney		50	10			
				muscle		50	10			
				fat		500	10			
				liver	deer	100	2			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									Canberra ACT 2601, Australia.	
				kidney		50	2			
				muscle		20	2			
				fat		500	2			
Neomycin	Neomycin	Yes	LC	liver	cattle	500	100	52nd JECFA; data provided to CCRVDF	FAO Food & Nutrition Paper 41/12; Giggisberg, D. , and Koch, H. (1995). *Mitt. Gebeite Lebensm. Hyg*. 86: 14-28 - single lab data prvided to CCRVDF by Switzerland.	full recommendation
				kidney		10000	100			
				muscle		500	100			
				fat		500	100			
				milk		1500	100	60th JECFA	FAO Food & Nutrition Paper 41/15	
				liver	pig	500	100	43rd JECFA; data provided to CCRVDF	FAO Food & Nutrition Paper 41/7; Giggisberg, D. , and Koch, H. (1995). *Mitt. Gebeite Lebensm. Hyg*. 86: 14-28.	provisional recommendation
				kidney		10000	100			
				muscle		500	100			
				fat		500	100			
		Yes	LC	kidney		10000	100		For method provided to 13th CCRVDF, contact Animal Research Institute, Chemical Residue Laboratory, 665 Fairfield Road, Yeerongpilly QLD 4105, Australia. See Report of 12th Meeting, CCRVDF.	provisional recommendation
		Yes		eggs	chicken	500	450			provisional recommendation
Nicarbazin	N,N'-bis-(4-nitrophenyl) urea	Yes	LC	liver	chicken	200	100	50th JECFA; data provided to CCRVDF	FAO Food & Nutrition Paper 41/11; Data provided by	full recommendation

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									Argentina to 16th CCRVDF	
				kidney		200	100			
				muscle		200	100			
				fat/skin		200	100			
Oxfendazole (see febantel, etc.)										full recommendation
Oxytetracycline (see chlortetracycline, etc.)										As per chlortetracycline
Phoxim	Phoxim	Yes	LC	liver	pig	50	10	52nd JECFA	FAO Food & Nutrition Paper 41/12	full recommendation
				kidney		50	10			
				muscle		50	10			
				fat		400	10			
			GC	liver	sheep, goat	50	50			
				kidney		50	50			
				muscle		50	50			
				fat		400	50			
Pirlimycin	Pirlimycin	Yes	LC/MS	liver	cattle	1000	250	62nd JECFA	FAO Food & Nutrition Paper 41/16 - additional validation with current generation equipment requested.	provisional recommendation
				kidney		400	50			
				muscle		100	50			
				fat		100	50			
				milk		100	50			
Procaine benzylpenicillin (see benzylpenicillin)	Benzylpenicillin	Yes	LC	liver	cattle, pig, chicken	50	5		See benzylpenicillin	As per benzylpenicillin
				kidney		50	5			
				muscle		50	5			
				fat		50	5			
Ractopamine	Ractopamine	Yes	LC	liver	cattle	40	5	62nd JECFA	FAO Food & Nutrition Paper 41/16	full recommendation
				kidney		90	5			
				muscle		10	5			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
				fat		10	5			
				liver	pig	40	5			
				kidney		90	5			
				muscle		10	5			
				fat		10	5			
Sarafloxacin	Sarafloxacin	Yes	LC	liver	chicken	80	5	50th JECFA	FAO Food & Nutrition Paper 41/11	full recommendation
				kidney		80	5		NOTE: For regulatory method for poultry and fish muscle provided to CCRVDF contact AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France.	
				muscle		10	5			
				fat		20	5			
				liver	turkey	80	5			
				kidney		80	5			
				muscle		10	5			
				fat		20	5			
Spectinomycin	Spectinomycin	Yes	LC	liver	cattle	2000	100	50th JECFA	FAO Food & Nutrition Paper 41/11; see also Report of 12th Meeting, CCRVDF: method issued by German Federal Institute for Consumer Health Protection and Veterinary Medicine, applicable to spectinomycin residues in muscle, kidney, liver and fat of calves, pigs and chickens, and in egg.	full recommendation
				kidney		5000	100			
				muscle		500	100			
				fat		2000	100			
				milk		200	100			
				liver	pig	2000	100			
				kidney		5000	100			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
				muscle		500	100			
				fat		2000	100			
				liver	sheep	2000	100			
				kidney		5000	100			
				muscle		500	100			
				fat		2000	100			
				liver	chicken	2000	100			
				kidney		5000	100			
				muscle		500	100			
				fat		2000	100			
				eggs		2000	250	42nd JECFA	FAO Food & Nutrition Paper 41/6; further method validation for analysis of tissues provided in FAO Food & Nutrition Paper 41/11.	
Spiramycin	Sum of Spiramycin and Neospiramycin	Yes	LC	liver	cattle	600	62.5	data provided to CCRVDF; 43rd & 47th JECFA	FAO Food & Nutrition Papers 41/7 & 41/9; data (1 lab) provided to CCRVDF for LC; 47th JECFA reviewed microbiological growth inhibition and LC methods; NOTE: For regulatory method provided to CCRVDF for muscle tissue contact AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France.	full recommendation
				kidney		300	30			
				muscle		200	30			
				fat		300	47			
		Yes	microbial growth inhibition	milk		200	62		FAO Food & Nutrition Paper 41/7; LOQ listed is	provisional recommendation

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									for the microbiological growth inhibition assay using ATCC 9341 as indicator organism.	
		Yes	LC	liver	pig	600	300	47th JECFA	FAO Food & Nutrition Paper 41/9	full recommendation
				kidney		300	300			
				muscle		200	100			
				fat		300	115			
				liver	chicken	600	100	43rd JECFA	FAO Food & Nutrition Paper 41/7; method suitability confirmed by 47th JECFA, FAO Food & Nutrition Paper 41/9.	
				kidney		800	200			
				muscle		200	50			
				fat		300	75			
Streptomycin (see Dihydrostreptomycin and Streptomycin)									FAO Food & Nutrition Paper 41/14; see also Gerhardt, G.C., Salisbury, C.D.C., & MacNeil, J.D. (1994) *J. AOAC Int*. 77: 334-337; data provided to CCRVDF by Canada, 2nd laboratory verification of performance reported by UK.	full recommendation
Sulfadimidine	Sulfadimidine	Yes	TLC	liver	cattle	100	20	Data provided to CCRVDF by U.S. and Canada.	AOAC Official Method 983.31: Sulfonamide Residues in Animal Tissues. (1995). *AOAC Official*	full recommendation
				kidney		100	20			
				muscle		100	20			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
									Methods of Analysis, 16th edition. AOAC International, Gaithersburg, MD. (method extension).	
		Yes	LC	milk		25	10	Data provided to CCRVDF by U.S.	AOAC Official Method 992.21 : Sulfamethazine Residues in Raw Bovine Milk. (1996). *AOAC Official Methods of Analysis, 16th edition, Supplement March 1996*. AOAC International, Gaithersburg, MD.	full recommendation
		Yes	TLC	liver	pig	100	20	Data provided to CCRVDF by U.S.	AOAC Official Method 983.31: Sulfonamide Residues in Animal Tissues. (1995). *AOAC Official Methods of Analysis, 16th edition*. AOAC International, Gaithersburg, MD.	full recommendation
				kidney		100	20	Data provided to CCRVDF by U.S. and Canada.	AOAC Official Method 983.31: Sulfonamide Residues in Animal Tissues. (1995). *AOAC Official Methods of Analysis, 16th edition*. AOAC International, Gaithersburg, MD. (method extension).	full recommendation

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
			TLC, LC	muscle		100	20	1. Data provided to CCRVDF by US. 2. Data provided to CCRVDF by Germany.	1. AOAC Official Method 983.31: Sulfonamide Residues in Animal Tissues. (1995). *AOAC Official Methods of Analysis, 16th edition*. AOAC International, Gaithersburg, MD. 2. Malisch, R., Bourgeois, B. and Lippold, R. (1992). *Deutsch. Lebensm. Rdsch*. 88: 205-216 .	full recommendation
		Yes	TLC	liver	sheep	100	20	Data provided to CCRVDF by U.S. and Canada.	AOAC Official Method 983.31: Sulfonamide Residues in Animal Tissues. (1995). *AOAC Official Methods of Analysis, 16th edition*. AOAC International, Gaithersburg, MD. (method extension).	full recommendation
				kidney		100	20			
				muscle		100	20			
		Yes	TLC	liver	poultry	100	20	Data (turkey, duck) provided to CCRVDF by U.S. Extension to chicken: U.S. and Canada.	AOAC Official Method 983.31: Sulfonamide Residues in Animal Tissues. (1995). *AOAC Official Methods of Analysis, 16th edition*. AOAC International, Gaithersburg, MD.	full recommendation
				kidney		100	20			
				muscle		100	20			

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
Tetracycline (see chlortetracycline, oxytetracycline, tetracycline)	Parent drug, alone or in combination	Yes	LC						See Chlortetracycline, Oxytetracycline, Tetracycline (above).	as per chlortetracycline
Thiabendazole	Sum of thiabendazole and 5-hydroxythiabendazole	Yes		liver	cattle	100		Info provided to 13th CCRVDF	NOTE: For regulatory method provided to CCRVDF by Australia, contact Amdel. 36-40 Halloran St., Lilyfield NSW 2040, Australia.	provisional recommendation
		Yes		milk		100		Info provided to 13th CCRVDF	NOTE: For regulatory method provided to CCRVDF contact AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France	provisional recommendation
		Yes		liver	pig	100		Info provided to 13th CCRVDF	NOTE: For regulatory method provided to CCRVDF by Australia, contact Amdel. 36-40 Halloran St., Lilyfield NSW 2040, Australia.	provisional recommendation
		Yes		liver	sheep	100		Info provided to 13th CCRVDF	NOTE: For regulatory method provided to CCRVDF by Australia, contact Amdel. 36-40 Halloran St., Lilyfield NSW 2040, Australia.	provisional recommendation

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
Tilmicosin	Tilmicosin	Yes	LC	liver	cattle	1000	50	47th JECFA	FAO Food & Nutrition Paper 41/9	provisional recommendation
		Yes	LC	kidney		300	10	Data provided to CCRVDF by Canada; 2nd laboratory data provided by UK.	Chan, W., Gerhardt, G.C., & Salisbury, C.D.C. 1994. *J. AOAC Int.* 77:331-333. NOTE: For alternate regulatory method for muscle tissue provided to CCRVDF contact AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France.	full recommendation
				muscle		100	10			
		Yes	LC	fat		100	50	47th JECFA	FAO Food & Nutrition Paper 41/9	
				liver	pig	1500	20			
		Yes	LC	kidney		1000	10	Data provided to CCRVDF by Canada; 2nd laboratory data provided by UK.	Chan, W., Gerhardt, G.C., & Salisbury, C.D.C. 1994. *J. AOAC Int.* 77:331-333.	
				muscle		100	10			
		Yes	LC	fat		100	20	47th JECFA	FAO Food & Nutrition Paper 41/9	
				liver	sheep	1000	50			
		Yes	LC	kidney		300	10	Data provided to CCRVDF by Canada; 2nd laboratory data provided by UK.	Chan, W., Gerhardt, G.C., & Salisbury, C.D.C. 1994. *J. AOAC Int.* 77:331-333.	
				muscle		100	10			
		Yes	LC	fat		100	50	47th JECFA	FAO Food & Nutrition Paper 41/9	

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
			LC	milk		50	50	47th JECFA	FAO Food & Nutrition Paper 41/9: LOQ is usually at least 1/2 the MRL - validation of method to 25 µg/kg recommended.	provisional recommendation
Trenbolone acetate	β-Trenbolone (liver)	Yes	LC	liver	cattle	10		Data provided to 15th CCRVDF by Canada	MacNeil, J.D., Reid, J.A., Neiser, C.D. & Fesser, A.C.E. (2003). *J. AOAC Int.* 86: 916-924.	provisional recommendation
	α-Trenbolone (muscle)			muscle		2				
Trichlorfon (metrifonate)	Trichlorfon	Yes	GC/MS	liver	cattle	50	50	54th JECFA	FAO Food & Nutrition Paper 41/13; MRLs for tissue are based on LOQ of method - no residues were deteted in tissues in the depletion studies.	full recommendation
				kidney		50	50			
				muscle		50	50			
				fat		50	50			
				milk		50	25			
Triclabendazole	5-Chloro-6-(2',3'-dichlorophenoxy)-benzimidazole-2-one	Yes	LC	liver	cattle	300	20-50	Data provided to CCRVDF by Switzerland.	Marti, A.M., Mooser, A.E., and Koch, H. (1990). *J. Chromatogr.* 498: 145-157. Data provided to CCRVDF for performance of method for pig liver, kidney and muscle tissues.	provisional recommendation
				kidney		300	20-50			
				muscle		200	20-50			
	NOTE: For regulatory method for triclabendazole residues in cattle and sheep liver provided to CCRVDF by Australia, contact Amdel. 36-40 Halloran St., Lilyfield NSW 2040, Australia.									

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
Zeranol	Zeranol	Yes	GC/MS	liver	cattle	10	0.5	Data provided to CCRVDF by U.S. and Canada.	Chemistry Laboratory Guidebook. United States Department of Agriculture, Food Safety and Inspection Service, Science Program, Washington, D.C. Results of multi-lab trial provided for review to CCRVDF.	full recommendation
				muscle		2	0.5			

Annex 2

Compounds for which validated analytical methods are required

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
Azaperone	Sum of azaperone and azaperol			muscle	pig	60		info provided to 13th Meeting, CCRVDF	Contact: AFSSA-LERMVD, Javene, BP090203-35302, Fougeres, France	provisional recommendation
		No		fat	pig	60				validated method required
Carazolol		No		muscle	pig	5				validated method required
				fat/skin	pig	5				
Closantel		No		kidney	cattle	3000				validated method required
		No		fat	sheep	3000				validated method required
				liver	sheep	1500				
		No		kidney	sheep	5000				validated method required
Deltamethrin		No		liver	sheep	50				validated method required
				kidney	sheep	50				
				fat	sheep	500				
				muscle	sheep	30				
Flumequine		No		muscle	black tiger shrimp (*P. monodon*)	500			Data requested by 62nd JECFA	validated method required

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
Levamisole		No		kidney	cattle	10				
				muscle	cattle	10				validated method required
				fat	cattle	10				
		No		kidney	pig	10				
				muscle	pig	10				validated method required
				fat	pig	10				
		No		kidney	sheep	10				validated method required
				muscle	sheep	10				
				fat	sheep	10				
				liver	poultry	100				validated method required
				kidney	poultry	10				
				muscle	poultry	10				
				fat	poultry	10				
Neomycin		No		liver	sheep/goat	500				validated method required
				kidney	sheep/goat	10000	20			
				muscle	sheep/goat	500	20			
		No		fat	sheep/goat	500				validated method required
		No		liver	chicken, turkey, duck	500				validated method required
				kidney	chicken, turkey, duck	10000	20			
				muscle	chicken, turkey, duck	500	20			
Neomycin		No		fat	chicken, turkey, duck	500				validated method required
				eggs	chicken	500				
Sulfadimidine				Fat	pig	100				
		No		fat	cattle	100				validated method required

Compound	Marker Residue	Method Recommended	Technique	Tissue	Species	MRL (µg/kg)	LCL or LOQ (µg/kg)	Verified By	Reference	Method Status
		No		fat	sheep	100				no method recommendation
		No		fat	poultry	100				validated method required
				muscle	cattle	100				
				fat	cattle	100				
		No		kidney	cattle	100				validated method required
				liver	cattle	100				
				milk	cattle	100				
		No		liver	pig	100				validated method required
				muscle	pig	100				
				kidney	pig	100				
				fat	pig	100				
				liver	sheep	100				
				muscle	sheep	100				
				kidney	sheep	100				
				fat	sheep	100				
				muscle	goat	100				
		No		fat	goat	100				validated method required
				liver	goat	100				
				kidney	goat	100				
				milk	goat	100				
Tilmicosin				milk	sheep	50				
Triclabendazole				fat	cattle	100				
				liver	sheep	100				
				kidney	sheep	100				
				muscle	sheep	100				
				fat	sheep	100				

Annex 3

Ad Hoc Working Group on Methods of Analysis and Sampling

Analytical Method Information Summary

A. Descriptive Information

1. Name of drug or chemical:_____
2. Drug or chemical class:_____
   (e.g. antimicrobial, anthelmintic, etc)
3. Veterinary Use:_____
4. Analyte(s) measured:_____
   (specify if metabolite)
5. Intended use of the method:
   a. Screening _____
   b. Routine_____
   c. Reference_____
   d. Confirmatory_____
6. Test matrix_____
   (e.g. muscle, kidney, urine, etc)
7. Summary of principal steps in sample preparation:
   _____
   _____
   _____
8. Summary of principal steps in extraction procedure:
   _____
   _____
   _____
9. Summary of principal steps in analyte clean-up procedure:
   _____
   _____
   _____
10. Measurement procedure:
    a. Chemical
       1. Instrumentation _____
       2. Detector system _____
       3. Chromatographic column _____
          (if applicable)

b. Immunochemical/Immunoassay

1. Technique: _____
(e.g. Elisa, RIA, Immunochromatog, etc)
2. Critical reagents: _____

_____
(e.g. antibody specificity and availability)
3. Special equipment required: _____

c. Microbiological

1. Technique: _____
2. Organism: _____
3. Media: _____
4. Special equipment required:_____

11. Sample/Analyte Stability
Warning (if applicable): _____

12. Literature References available:

_____
_____
_____

13. Contact for Information:

a. Name _____
b. Country _____
c. Affiliation _____
d. Address _____
_____
e. Telephone _____
f. FAX _____
g. Email _____

B. Method Performance

1. a. Limit of Detection (LOD) (mg/kg) _____
How was LOD determined?_____
_____

b. Limit of Quantification (LOQ) (mg/kg)_____
How was LOQ determined?_____
_____

c. Method sensitivity_____
(The smallest difference in concentration that can be measured)

2. JECFA MRL_____
3. Are analytical data corrected for recovery? Yes____ No_____
4. How is recovery estimated _____
(e.g. external standard; internal standard. etc)
5. Accuracy

a. Concentration(s) tested _____ _____ _____
b. Concentration(s) measured _____ _____ _____
c. Recovery (%) _____ _____ _____

6. Precision using fortified control tissue

   a. Concentration(s) tested _____ _____ _____
   b. Repeatability (within lab CV) _____ _____ _____
   c. Reproducibility (between lab CV) _____ _____ _____

7. Precision using tissue containing incurred drug residues

   a. Concentration(s) tested _____ _____ _____
   b. Repeatability (within lab CV) _____ _____ _____
   c. Reproducibility (between lab CV) _____ _____ _____

8. Selectivity of the method

   This information is often referenced as "Specificity". Selectivity refers to the ability of the method to provide accurate measurement of the analyte of interest when other chemicals or drugs are also resident in the laboratory sample. Data of interest in this regard are the effects of:

   a. Drugs of similar structure or drug class or other veterinary drugs that may also be used along with the analyte of interest _____.

   b. Contaminants that are likely to be present in the sample _____

9. Type of Validation studies

   a. Single laboratory _____
   b. Multi-laboratory _____
   c. AOAC or other official procedure _____

C. Information relevant to laboratory implementation

1. Training and experience recommended for analyts

2. Critical steps in the method

3. Information on availability of unusual reagents or equipment

4. Special reagent or sample stability concerns

5. Reagent handling and safety concerns (if any)

6. Literature references or other useful information

Annex 4

OUTLINE OF SCIENTIFIC ISSUES COMMONLY CONSIDERED IN THE DEVELOPMENT AND VALIDATION OF ANALYTICAL METHODS

1. Determinative (Quantitative) Method

A. Purpose of the Method

*Scope of application (intended use)
*Target tissue
*Marker residue (analyte)
*Limit of quantification (LOQ), Limit of Detection (LOD) or other Lowest Validated Level

B. Experimental data

*Reagents (purity, strength, grade)
*Apparatus and Equipment
*Analytical Standards (quality, concentration and solvents)
*Tissue Samples (procedure for preparation for analysis)
*Analyte Extraction Procedures
*Analyte Clean-up
*Instumental Procedures and Calibrations
*Calculations

C. Quality Assurance

*Storage Stability of the Analyte in Tissue
*Quality Control Samples
*System Suitability Criteria
*Readiness to perform assessment
*Data Acceptability Criteria

2. Confirmation Procedure

*Sample preparation
*Instrumental procedures and calibrations
*Standards employed
*Criteria for positive identification

3. Validation considerations

*Accuracy
*Recovery
*Precision (repeatability and reproducibility)
*Sensitivity and LOQ
*Specificity