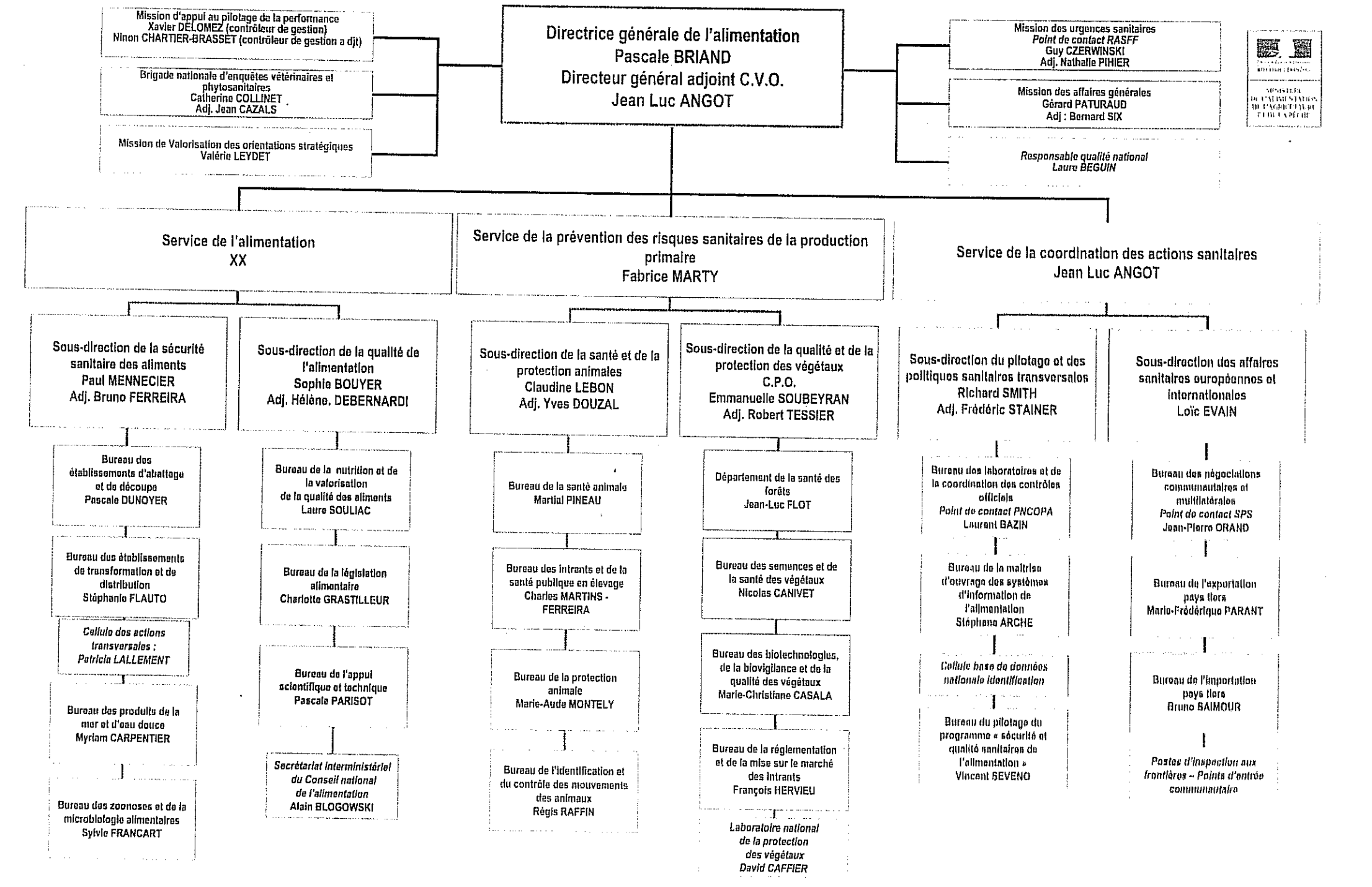

Mission d'appui au pilotage de la performance
Xavier DELOMEZ (contrôleur de gestion)
Ninon CHARTIER-BRASSET (contrôleur de gestion a djt)
Brigade nationale d'enquêtes vétérinaires et phytosanitaires
Catherine COLLINET
Adj. Jean CAZALS
Mission de Valorisation des orientations stratégiques
Valérie LEYDET
Directrice générale de l'alimentation
Pascale BRIAND
Directeur général adjoint C.V.O.
Jean Luc ANGOT
Mission des urgences sanitaires
Point de contact RASFF
Guy CZERWINSKI
Adj. Nathalie PIHIER
Mission des affaires générales
Gérard PATURAUD
Adj : Bernard SIX
Responsable qualité national
Laure BEGUIN
Service de l'alimentation
XX
Service de la prévention des risques sanitaires de la production primaire
Fabrice MARTY
Service de la coordination des actions sanitaires
Jean Luc ANGOT
Sous-direction de la sécurité sanitaire des aliments
Paul MENNECIER
Adj. Bruno FERREIRA
Sous-direction de la qualité de l'alimentation
Sophie BOUYER
Adj. Hélène. DEBERNARDI
Sous-direction de la santé et de la protection animales
Claudine LEBON
Adj. Yves DOUZAL
Sous-direction de la qualité et de la protection des végétaux
C.P.O.
Emmanuelle SOUBEYRAN
Adj. Robert TESSIER
Sous-direction du pilotage et des politiques sanitaires transversales
Richard SMITH
Adj. Frédéric STAINER
Sous-direction des affaires sanitaires européennes et internationales
Loïc EVAIN
Bureau des établissements d'abattage et de découpe
Pascale DUNOYER
Bureau des établissements de transformation et de distribution
Stéphanie FLAUTO
Cellule des actions transversales :
Patricia LALLEMENT
Bureau des produits de la mer et d'eau douce
Myriam CARPENTIER
Bureau des zoonoses et de la microbiologie alimentaires
Sylvie FRANCART
Bureau de la nutrition et de la valorisation de la qualité des aliments
Laure SOULIAC
Bureau de la législation alimentaire
Charlotte GRASTILLEUR
Bureau de l'appui scientifique et technique
Pascale PARISOT
Secrétariat interministériel du Conseil national de l'alimentation
Alain BLOGOWSKI
Bureau de la santé animale
Martial PINEAU
Bureau des intrants et de la santé publique en élevage
Charles MARTINS - FERREIRA
Bureau de la protection animale
Marie-Aude MONTELY
Bureau de l'identification et du contrôle des mouvements des animaux
Régis RAFFIN
Département de la santé des forêts
Jean-Luc FLOT
Bureau des semences et de la santé des végétaux
Nicolas CANIVET
Bureau des biotechnologies, de la biovigilance et de la qualité des végétaux
Marie-Christiane CASALA
Bureau de la réglementation et de la mise sur le marché des intrants
François HERVIEU
Laboratoire national de la protection des végétaux
David CAFFIER
Bureau des laboratoires et de la coordination des contrôles officiels
Point de contact PNCOPA
Laurent BAZIN
Bureau de la maîtrise d'ouvrage des systèmes d'information de l'alimentation
Stéphane ARCHE
Cellule base de données nationale identification
Bureau du pilotage du programme « sécurité et qualité sanitaires de l'alimentation »
Vincent SEVENO
Bureau des négociations communautaires et multilatérales
Point de contact SPS
Jean-Pierre ORAND
Bureau de l'exportation pays tiers
Marie-Frédérique PARANT
Bureau de l'importation pays tiers
Bruno SAIMOUR
Postes d'inspection aux frontières – Points d'entrée communautaire

Décrets, arrêtés, circulaires

TEXTES GÉNÉRAUX

MINISTÈRE DE L'AGRICULTURE ET DE LA PÊCHE

Arrêté du 18 mai 2009 fixant la liste des postes frontaliers de contrôle vétérinaire et phytosanitaire (1)

NOR : *AGRG0909211A*

Le ministre de l'agriculture et de la pêche et le ministre du budget, des comptes publics et de la fonction publique,

Vu le règlement (CE) n° 882/2004 du Parlement européen et du Conseil du 29 avril 2004 relatif aux contrôles officiels effectués pour s'assurer de la conformité avec la législation sur les aliments pour animaux et les denrées alimentaires et avec les dispositions relatives à la santé animale et au bien-être des animaux ;

Vu la directive 91/496/CEE du Conseil du 15 juillet 1991 modifiée fixant les principes relatifs à l'organisation des contrôles vétérinaires pour les animaux en provenance de pays tiers introduits dans la Communauté, et en particulier ses articles 3 et 6 ;

Vu la directive 97/78/CEE du Conseil du 18 décembre 1997 fixant les principes relatifs à l'organisation des contrôles vétérinaires pour les produits en provenance des pays tiers introduits dans la Communauté ;

Vu la directive 2000/29/CE du Conseil du 8 mai 2000 concernant les mesures de protection contre l'introduction dans la Communauté d'organismes nuisibles aux végétaux ou aux produits végétaux et contre leur propagation à l'intérieur de la Communauté ;

Vu la décision de la Commission 2001/812/CE du 21 novembre 2001 établissant les exigences relatives à l'agrément des postes d'inspection frontaliers chargés des contrôles vétérinaires pour les produits en provenance des pays tiers introduits dans la Communauté ;

Vu la décision de la Commission 2001/881/CE du 7 décembre 2001 établissant une liste de postes d'inspection frontaliers agréés pour les contrôles vétérinaires sur les animaux vivants et les produits animaux en provenance des pays tiers et actualisant les modalités des contrôles que doivent effectuer les experts de la Commission ;

Vu le code rural, et notamment les articles L. 236-1, L. 236-4, L. 251-18, L. 251-18-1, D. 251-1 et D. 251-22 ;

Vu le code des douanes, notamment son article 24, paragraphe 1 ;

Vu l'arrêté du 24 mai 2006 relatif aux exigences sanitaires des végétaux, produits végétaux et autres objets,

Arrêtent :

Art. 1er. – Les postes d'inspection frontaliers dont la liste figure en annexe I du présent arrêté sont habilités pour la réalisation des contrôles vétérinaires, selon le cas, des animaux vivants, des produits et sous-produits d'origine animale, des produits végétaux visés à l'article 19 de la directive 97/78/CE, des micro-organismes pathogènes pour les animaux et les produits susceptibles de les véhiculer, en provenance de pays tiers, conformément aux indications reprises dans cette annexe.

Art. 2. – Les points d'entrée désignés dont la liste figure en annexe II du présent arrêté sont désignés pour la réalisation des contrôles officiels des produits destinés à l'alimentation animale en provenance de pays tiers autres que ceux visés au paragraphe ci-dessus.

Art. 3. – Les points d'entrée communautaires dont la liste figure en annexe III du présent arrêté sont désignés pour la réalisation des contrôles phytosanitaires de tout ou partie des végétaux, des produits végétaux et autres objets soumis à exigences phytosanitaires.

Art. 4. – L'arrêté du 19 novembre 2003 modifié fixant la liste des postes d'inspection frontaliers est abrogé.

L'arrêté du 2 mai 2007 fixant la liste des points d'entrée pour les végétaux, les produits végétaux et autres objets soumis à contrôle phytosanitaire est abrogé.

Art. 5. – Le directeur général de l'alimentation et le directeur général des douanes et droits indirects sont chargés, chacun en ce qui le concerne, de l'exécution du présent arrêté, qui sera publié au *Journal officiel* de la République française.

Fait à Paris, le 18 mai 2009.

Le ministre de l'agriculture et de la pêche,
Pour le ministre et par délégation :
Le directeur général de l'alimentation,
J.-M. BOURNIGAL

Le ministre du budget, des comptes publics et de la fonction publique,
Pour le ministre et par délégation :
Le directeur général des douanes et droits indirects,
J. FOURNEL

(1) Points d'entrée pour les végétaux, produits végétaux et autres objets soumis à contrôle phytosanitaire, postes d'inspection frontaliers pour les animaux vivants et les produits d'origine animale et points d'entrée désignés pour les aliments pour animaux non d'origine animale.

ANNEXE I

POSTES D'INSPECTION FRONTALIERS

NOM	CODE TRACES	TYPE	CENTRE D'INSPECTION	PRODUITS	ANIMAUX VIVANTS
Bordeaux	FR BOD 4	Aéroport		HC-T (1), HC-NT, NHC	
Bordeaux	FR BOD 1	Port		HC-NT	
Boulogne	FR BOL 1	Port		HC-T (1) (3), HC-NT (1) (3)	
Brest	FR BES 4	Aéroport		HC-T (CH) (1) (2)	
Brest	FR BES 1	Port		HC-T (FR) (1), NHC-T (FR)	
Châteauroux-Déols	FR CHR 4	Aéroport		HC-T (2)	
Deauville	FR DOL 4	Aéroport			E
Dunkerque	FR DKK 1	Port	Caraïbes	HC-T (1), HC-NT	
			Maison Blanche	NHC-NT	
Le Havre	FR LEH 1	Port	Route des Marais	HC-T (1), HC-NT, NHC	
			Dugrand	HC-T (FR) (1) (2)	
			EFBS	HC-T (FR) (1) (2)	
			Fécamp	HC-NT (6), NHC-NT (6)	
Lorient	FR LRT 1	Port	CCIM	NHC-NT (4)	
Lyon - Saint-Exupéry	FR LIO 4	Aéroport		HC-T (1), HC-NT, NHC	O
Marseille Port (15)	FR MRS 1	Port	Hangar 14		E
			STEF	HC-T (1) (2), HC-NT (2)	
Marseille - Fos-sur-Mer	FR FOS 1	Port		HC-T (1), HC-NT, NHC	

NOM	CODE TRACES	TYPE	CENTRE D'INSPECTION	PRODUITS	ANIMAUX VIVANTS
Marseille aéroport	FR MRS 4	Aéroport		HC-T (1), HC-NT,	O
Nantes - Saint-Nazaire	FR NTE 1	Port		HC-T (1), HC-NT, NHC-NT	
Nice	FR NCE 4	Aéroport		HC-T (CH) (1) (2)	O
Orly	FR ORY 4	Aéroport	SFS	HC-T (1) (2), HC-NT (2), NHC	
Réunion - Port Réunion	FR LPT 1	Port		HC (1), NHC	
Réunion - Roland-Garros	FR RUN 4	Aéroport		HC (1), NHC	O
Roissy - Charles-de-Gaulle	FR CDG 4	Aéroport	Air France	HC-T (1), HC-NT, NHC-NT	
			France Handling	HC-T (1), HC-NT, NHC	
			Station animalière (b)		E, O (a)
Rouen	FR URO 1	Port		HC-T (1) (2), HC-NT (2), NHC	
Sète	FR SET 1	Port	Frontignan	HC-T (1), HC-NT	
Toulouse-Blagnac	FR TLS 4	Aéroport		HC-T (1) (2), HC-NT (2), NHC (2)	O
Vatry	FR VRY 4	Aéroport		HC-T (CH) (1) (2)	

HC = Tous produits de consommation humaine.
NHC = Autres produits.
NT = Sans conditions de température.
T = Produits congelés/réfrigérés.
T (FR) = Produits congelés.
T (CH) = Produits réfrigérés.
U = Ongulés : les bovins, porcins, ovins, caprins et solipèdes domestiques ou sauvages.
E = Equidés enregistrés au sens de la directive 90/426/CEE.
O = Autres animaux (y compris animaux de zoo).
(1) Contrôles dans les conditions de la décision 93/352/CEE de la Commission prise en application de l'article 19, paragraphe 3, de la directive 97/78/CE du Conseil.
(2) Produits emballés uniquement.
(3) Produits de la pêche uniquement.
(4) Uniquement protéines animales.
(6) Graisses, huiles et huiles de poisson liquides uniquement.
(15) Cette autorisation est valable jusqu'au 31 juillet 2011.
(a) Uniquement primates et animaux non dangereux (au sens de l'arrêté du 10 août 2004 [*]), et présentés en conteneurs fermés, des espèces appartenant aux invertébrés, reptiles, amphibiens et animaux aquatiques d'ornement.
(b) Cette autorisation est valable jusqu'au 31 décembre 2009.
(*) Arrêté du 10 août 2004 modifié fixant les conditions d'autorisation de détention d'animaux de certaines espèces non domestiques dans les établissements d'élevage, de vente, de location, de transit ou de présentation au public d'animaux d'espèces non domestiques.

ANNEXE II

POINTS D'ENTRÉE DÉSIGNÉS

NOM	TYPE
Baie-Mahault	Port
Bordeaux	Port
Boulogne	Port

NOM	TYPE
Brest	Port
Caen	Port
Dunkerque	Port
Fort-de-France	Port
La Rochelle-Rochefort	Port
Le Havre	Port
Le Port	Port
Lorient	Port
Marseille	Port
Nantes - Saint-Nazaire	Port
Port-la-Nouvelle	Port
Roissy - Charles-de-Gaulle	Aéroport
Rouen	Port
Saint-Laurent-du-Maroni	Route
Saint-Malo	Port
Sète	Port

ANNEXE III

POINTS D'ENTRÉE COMMUNAUTAIRES

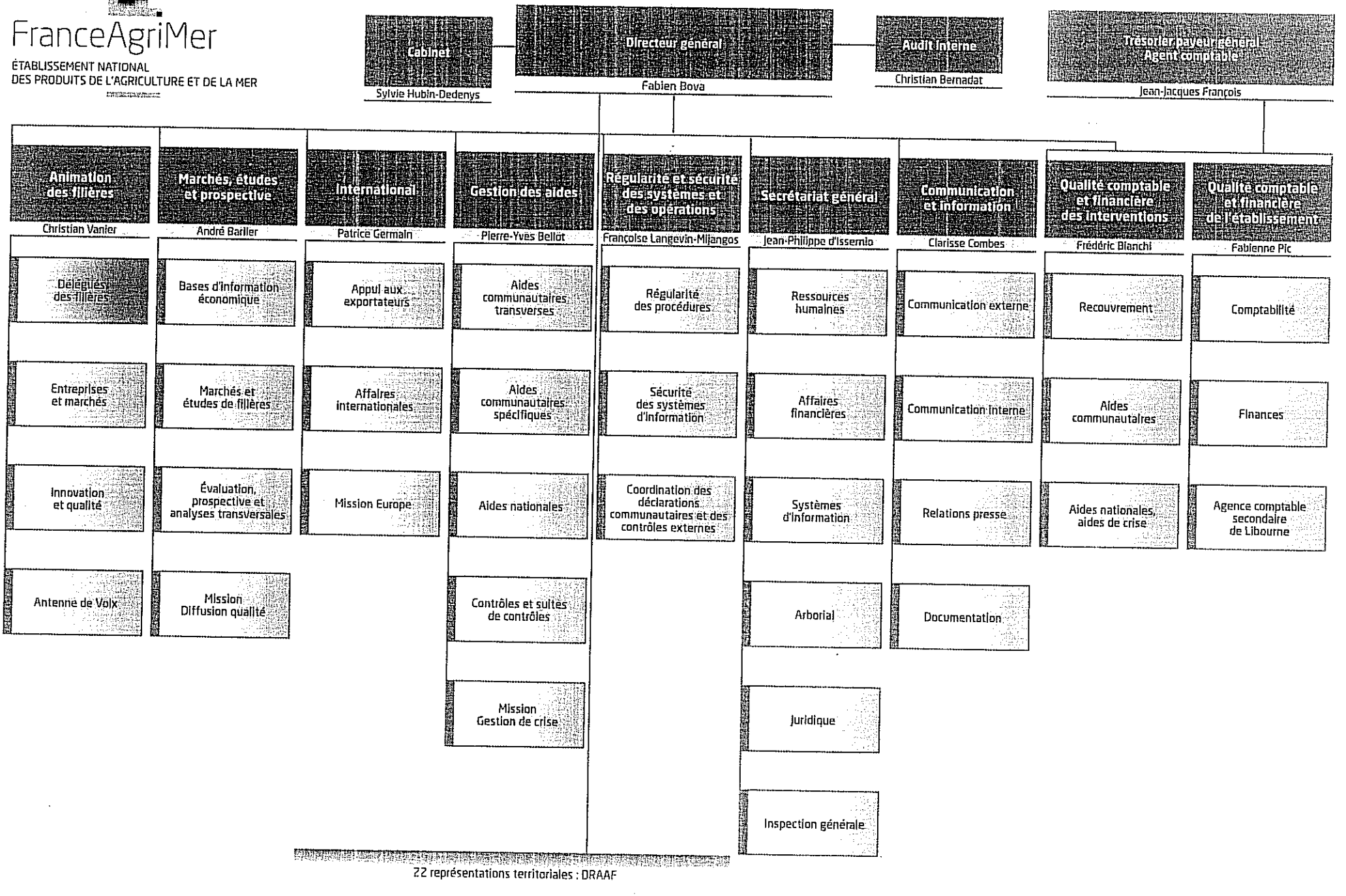
NOM	TYPE	LIEUX D'INSPECTION désignés par catégorie		
		Végétaux, produits végétaux et autres objets (y compris bois sous toutes ses formes et fruits et légumes)	Bois sous toutes ses formes exclusivement	Fruits et légumes exclusivement
Basse-Terre	Port	Tous		
Bastia	Aéroport-port	Tous		
Bordeaux	Aéroport-port	Tous		
Brest-Le Paludien	Port		Tous	
Caen-Honfleur	Port		Tous	
Degrad-de-Cannes	Port	Tous		

NOM	TYPE	LIEUX D'INSPECTION désignés par catégorie		
		Végétaux, produits végétaux et autres objets (y compris bois sous toutes ses formes et fruits et légumes)	Bois sous toutes ses formes exclusivement	Fruits et légumes exclusivement
Dunkerque	Port	Tous		
Fécamp-Le Havre-Rouen	Port	Le Havre	Fécamp Rouen	
Fort-de-France	Port	Tous		
Fos - Port-Saint-Louis	Port	Tous		
Kourou	Port	Tous		
La Rochelle - Rochefort - Tonnay-Charente	Port	La Rochelle	Rochefort - Tonnay-Charente	
Le Lamentin	Aéroport	Tous		
Le Légué - Saint-Malo	Port		Tous	
Le Port	Port	Tous		
Le Raizet	Aéroport	Tous		
Lyon - Saint-Exupéry	Aéroport	Tous		
Marseille port	Port	Tous		
Marseille-Marignane	Aéroport	Tous		
Nantes	Aéroport-port	Nantes Atlantique Montoir-de-Bretagne	Cheviré	Saint-Nazaire
Nice Côte d'Azur	Aéroport	Tous		
Orly	Aéroport	Tous		
Pau	Aéroport	Tous		
Perpignan - Port-Vendres	Aéroport-port-routier-rail	Perpignan		Port-Vendres
Pointe-à-Pitre	Port	Tous		
Rochambeau	Aéroport	Tous		
Roissy	Aéroport	Tous		
Rungis	Routier	Tous		
Saint-Denis	Aéroport	Tous		
Saint-Laurent-du-Maroni	Port	Tous		

NOM	TYPE	LIEUX D'INSPECTION désignés par catégorie		
		Végétaux, produits végétaux et autres objets (y compris bois sous toutes ses formes et fruits et légumes)	Bois sous toutes ses formes exclusivement	Fruits et légumes exclusivement
Saint-Pierre	Aéroport	Tous		
Toulouse-Blagnac	Aéroport	Tous		
Vatry	Aéroport	Tous		

FranceAgriMer

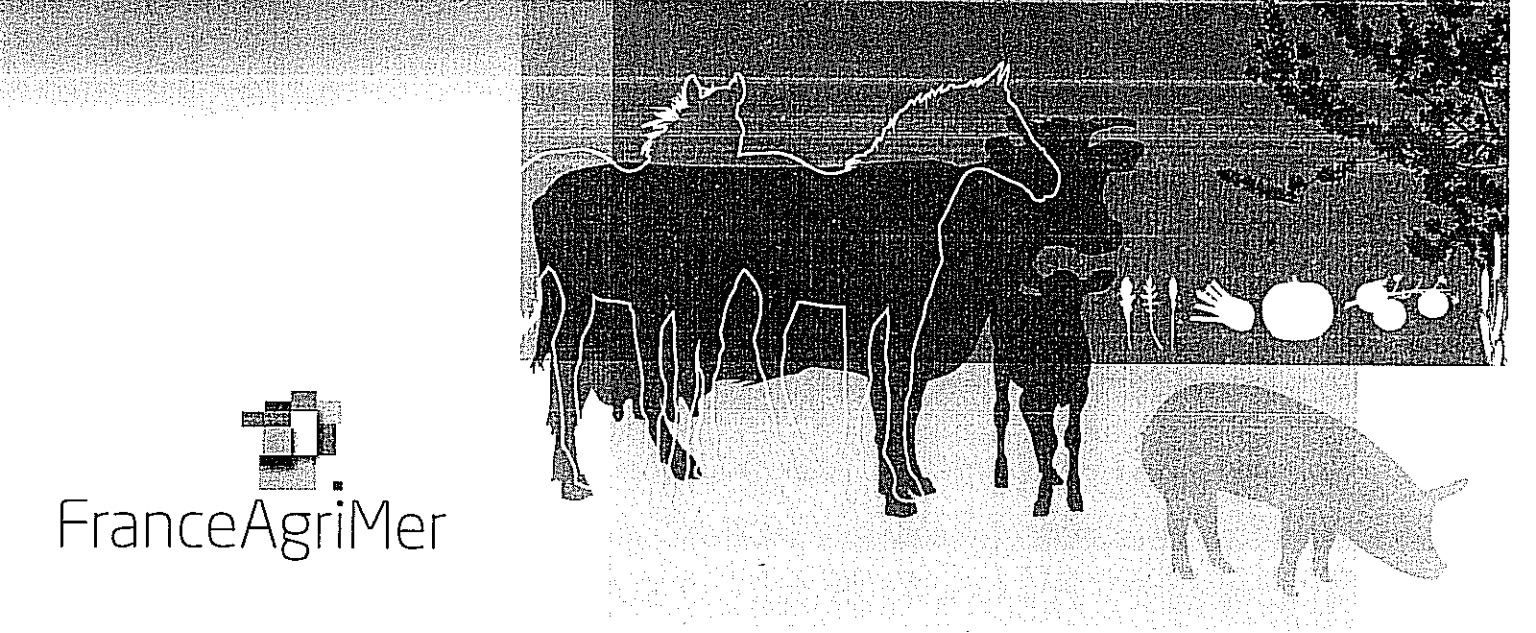
ÉTABLISSEMENT NATIONAL
DES PRODUITS DE L'AGRICULTURE ET DE LA MER



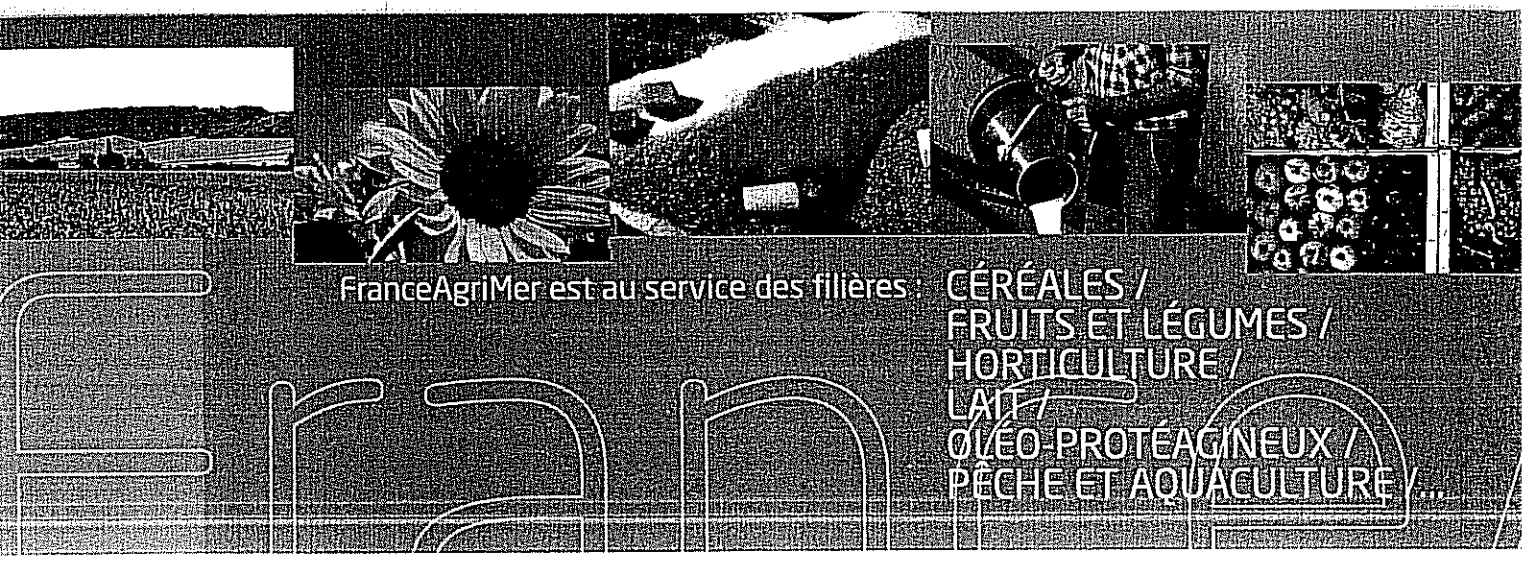
CÉRÉALES /
FRUITS ET LÉGUMES /
HORTICULTURE /
LAIT /
OLÉO-PROTÉAGINEUX /
PÊCHE ET AQUACULTURE /
PLANTES À PARFUM, AROMATIQUES ET MÉDICINALES
SUCRE /
VIANDES BLANCHES /
VIANDES ROUGES /
VINS /

FranceAgriMer

Un lieu d'échanges et d'arbitrage
entre les filières françaises de l'agriculture et de la pêche



FranceAgriMer



FranceAgriMer, établissement national des produits de l'agriculture et de la mer, est un établissement public administratif placé sous la tutelle de l'État.

FranceAgriMer est un lieu d'échanges et d'arbitrage entre les filières françaises de l'agriculture et de la pêche, rassemblées au sein d'un même établissement, en lieu et place des anciens offices agricoles[1].

Les missions de FranceAgriMer

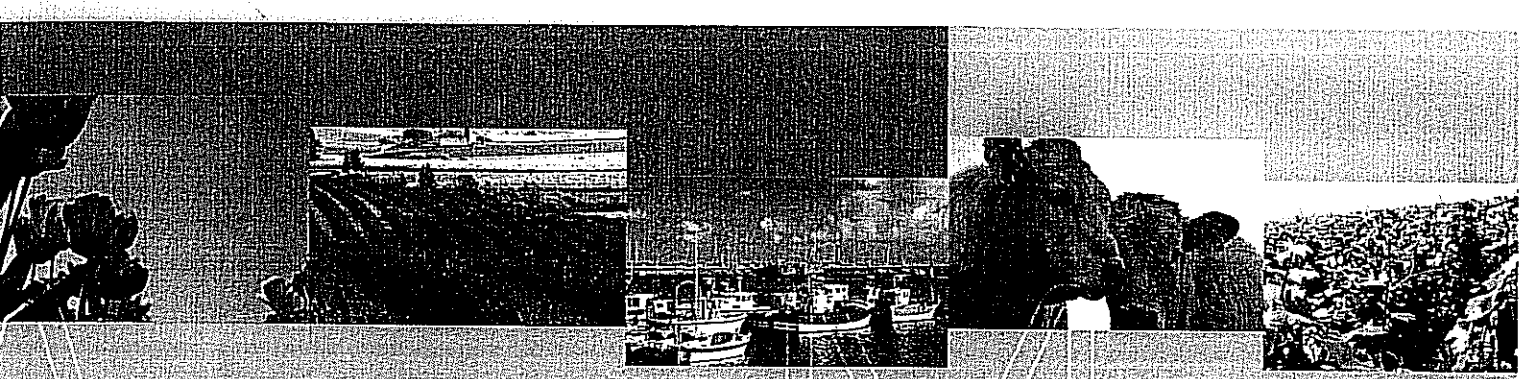
Face aux défis alimentaires, économiques, environnementaux et énergétiques qui s'annoncent, les missions de FranceAgriMer sont :

> SUR LES MARCHÉS
- assurer une veille économique et la connaissance des marchés ;
- gérer les mesures communautaires de régulation des marchés ;
- générer des alertes en cas de crise.

>> POUR LES FILIÈRES
- renforcer l'efficacité économique des filières ;
- participer à la mise en place d'une politique de développement durable et de qualité ;
- prévenir les risques préjudiciables aux filières.

>>> AUPRÈS DES PRODUCTEURS ET OPÉRATEURS DES FILIÈRES
- favoriser l'organisation des producteurs ;
- encourager le dialogue interprofessionnel ;
- contribuer à des actions de coopération technique, y compris à l'international.

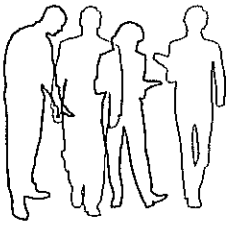
1) Créé le 1er avril 2009, FranceAgriMer est issu de la fusion de cinq offices agricoles : Ofimer, Office de l'Élevage, ONIGC, Onippam et Viniflhor.



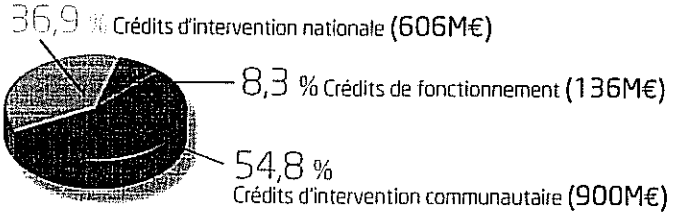
PLANTES À PARFUM AROMATIQUES ET MÉDICINALES / SUCRE / VIANDES BLANCHES / VIANDES ROUGES / VINS /

Les chiffres de FranceAgriMer

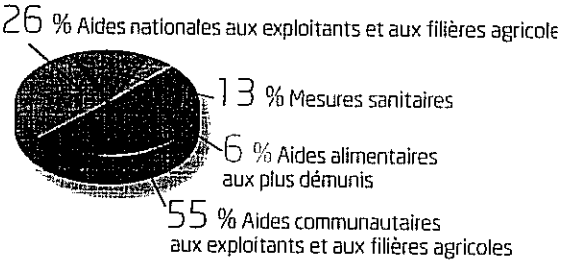
Pour effectuer l'ensemble de ses missions, FranceAgriMer emploie environ **1 350** personnes dont **450** en région.



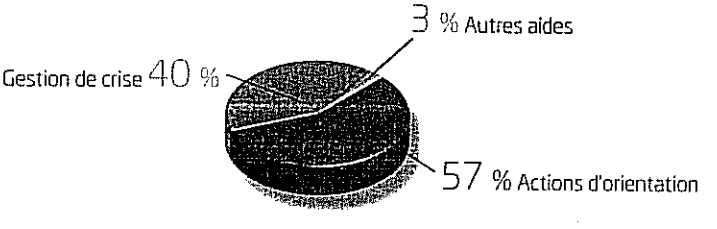
Ressources de FranceAgriMer



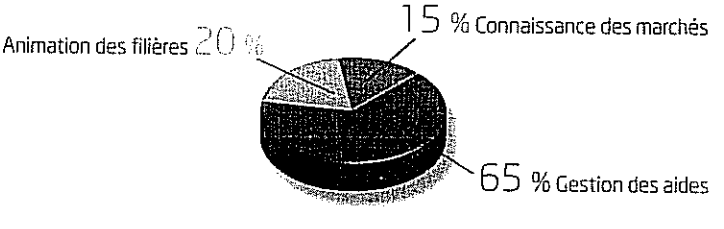
Nature d'intervention



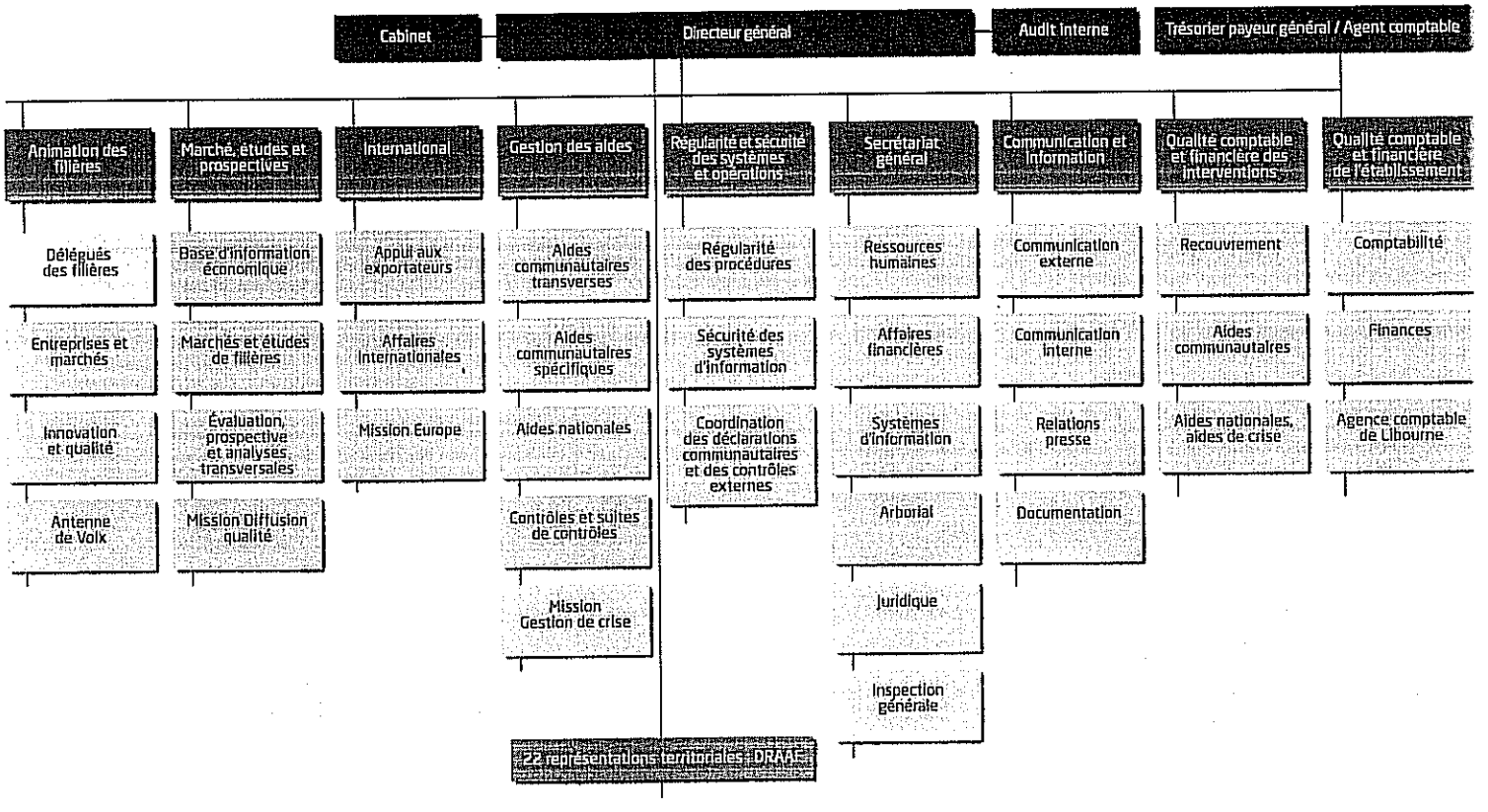
Aides nationales aux exploitants et aux filières agricoles



Crédits de fonctionnement



Pour remplir ses missions, FranceAgriMer est organisé par métier.



Le siège social de FranceAgriMer se situe à Montreuil-sous-Bois (93).

Trois délégations nationales y sont rattachées : Libourne (33), où est gérée l'organisation commune de marché vitivinicole ; La Rochelle (17), où se trouve un laboratoire d'analyses spécialisé dans les céréales ; Volx (04), où s'effectue le suivi des plantes à parfum, aromatiques et médicinales, ainsi que des productions méditerranéennes (huile d'olive et riz).

En région, FranceAgriMer dispose de délégations territoriales aujourd'hui intégrées dans les directions régionales de l'Alimentation, de l'Agriculture et de la Forêt (DRAAF). Directement en liaison avec les producteurs et opérateurs des différentes filières, les agents des régions assurent l'ensemble des missions de contrôle, de traitement des informations et des demandes d'aides qui sont du ressort de l'établissement.

FranceAgriMer
ÉTABLISSEMENT NATIONAL
DES PRODUITS DE L'AGRICULTURE ET DE LA MER

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www.franceagrimer.fr

Tableaux récapitulatifs analytes : laboratoires – modalités de prélèvement – produits d'origine animal

Tableau 1 : analyses bactériologiques

Analytes	Produits	Laboratoire	Modalités de prélèvements	Quantité minimale
Listeria monocytogènes	Produits de la pêche non histamino sensibles & viandes	LAVD 76	5 échantillons	5*200 g
	Produits de la pêche histamino sensibles	LDA 22		
	Produits laitiers	LASAT		
Salmonelles	Produits de la pêche & viandes & gélatine – collagènes & farines de poisson	LAVD 76		
	Produits laitiers	LASAT		
Vibrions	Produits de la pêche	LAVD 76		
Enterobactéries	Farines de poisson			
Entérotoxines staphylococciques	Produits laitiers	LASAT		
Histamines	Produits de la pêche sensibles	LDA 22	9 échantillons	9*200 g
Stabilité conserves	Produits de la pêche & viandes	LAVD 76	2 conserves	

Références :

- Modalité de prélèvements : règlement modifié (CE) n° 2073/2005 de la Commission du 15 novembre 2005 concernant les critères microbiologiques applicables aux denrées alimentaires.
- Laboratoires d'envoi : note de service DGAL/SDPPST/N2009-8211 du 22/07/2009 : laboratoires agréés pour la réalisation des analyses officielles dans le domaine de la microbiologie des aliments.

Tableaux récapitulatifs analytes : laboratoires – modalités de prélèvement – produits d'origine animal

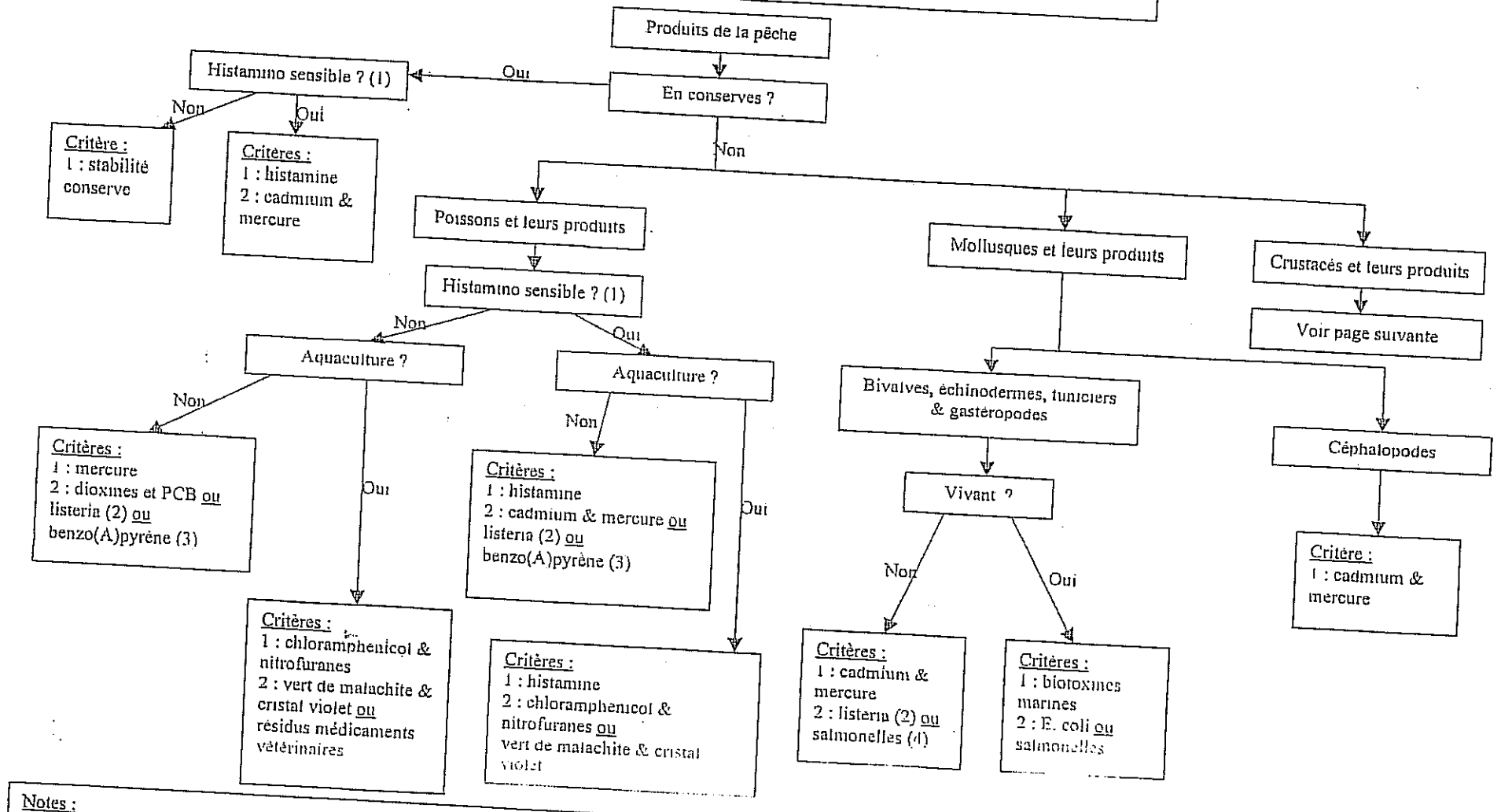
Tableau 2 : résidus de traitement

<table>
<tr><th>Analytes</th><th colspan="2">Produits</th><th>Laboratoire</th><th>Modalités de prélèvements</th><th>Quantité minimale</th></tr>
<tr><td rowspan="3">Chloramphenicol & nitrofuranes</td><td colspan="2">Produits de la pêche & viandes & œufs & boyaux & cuisses de grenouilles - escargots</td><td>LEAV 85</td><td rowspan="11">Arrêté du 5 mai 2000 :
• Produits en vrac : 5 prises élémentaires
• Produits emballés : 1% des pièces, min 2 et max 10</td><td rowspan="3">500 g</td></tr>
<tr><td colspan="2">Viandes d'ongulée</td><td>LD 72 ou LEAV 85</td></tr>
<tr><td colspan="2">Autres viandes</td><td>LEAV 85</td></tr>
<tr><td>Chloramphenicol</td><td rowspan="2" colspan="2">Miel & produits apicoles</td><td>AFSSA 06</td><td rowspan="3">200 g</td></tr>
<tr><td>Nitrofuranes</td><td>AFSSA 35</td></tr>
<tr><td>Vert de malachite</td><td colspan="2">Produits de la pêche</td><td>AFSSA 35 ou LEAV 85</td></tr>
<tr><td>Cristal violet</td><td colspan="2">Produits de la pêche</td><td>AFSSA 35</td></tr>
<tr><td rowspan="3">Résidus de médicaments vétérinaires</td><td rowspan="2">Produits de la pêche</td><td>Antibiotiques</td><td>LEAV 85</td><td rowspan="4">500 g</td></tr>
<tr><td>Quinolones</td><td>LDA 22</td></tr>
<tr><td colspan="2">Viandes & œufs & boyaux</td><td>LEAV 85</td></tr>
<tr><td>B-agonistes & stéroïdes</td><td colspan="2">Viandes d'ongulé</td><td>LD 72</td></tr>
<tr><td>Flubendazole</td><td colspan="2">Viandes de volaille</td><td>LEAV 85</td></tr>
</table>

Note : laboratoire d'envoi défini par la note de service DGAL/SDPPST/N2009-8053 du 05/02/2009 : laboratoires réalisant des analyses officielles dans le cadre des plans de surveillance et des plans de contrôle de la contamination des denrées animales et d'origine animale, et des produits destinés à l'alimentation animale pour l'année 2009

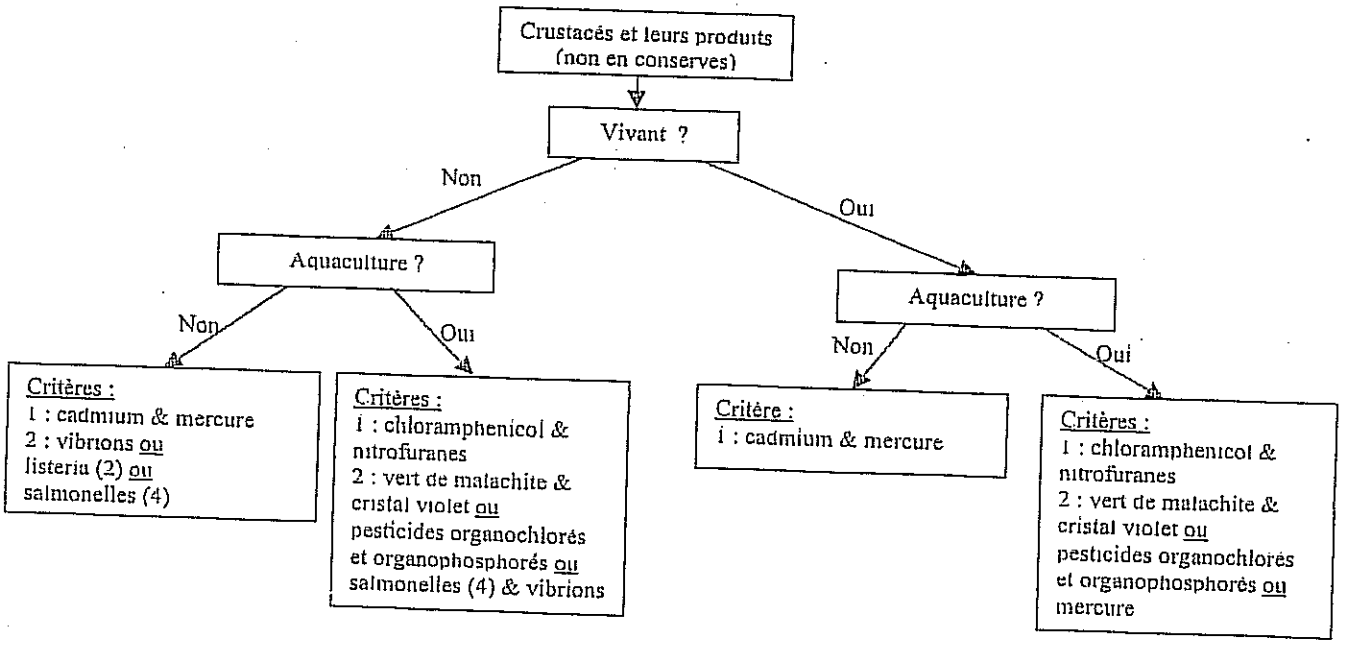
Codification : E PDS-1/7
Ind. Rév : A
Date : 18/05/09

Plan de sondage produits de la pêche consommation humaine : arbre de décision (1/2)



Notes :
(1) : poissons et leurs produits appartenant aux familles suivantes : *Scombridae*, *Clupeidae*, *Engraulidae*, *Coryphenidae*, *Pomatomidae*, *Scomberesocidae*, *Xiphiidae* et *Istiophoridae*.
(2) : uniquement sur les produits prêts à être consommés
(3) : uniquement sur les produits fumés
(4) : uniquement produits cuits

Plan de sondage produits de la pêche consommation humaine : arbre de décision (2/2)



Notes :
(2) : uniquement sur les produits prêts à être consommés
(4) : uniquement produits cuits

Tableaux récapitulatifs analytes : laboratoires – modalités de prélèvement – produits d'origine animal

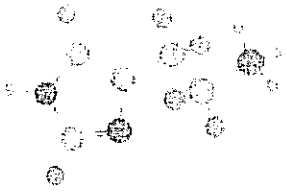
Tableau 3 : contaminants environnementaux

Analytes	Produits	Laboratoire	Modalités de prélèvements	Quantité minimale
Métaux lourds	Produits de la pêche & viandes	LEAV 85	Règlement CE 333/2007	1 kg
Dioxines et PCB-DL	Produits de la pêche & viandes & produits laitiers & ovoproduits & huiles de poissons CH	LEAV 85	Règlement CE 1883/2006	1 kg, à envoyer dans aluminium (si solide) ou flacons en verre (sinon)
	Huiles de poissons AA & farines de poisson		Règlement 152/2009 : idem prélèvements AANOA	
Benzo(A)pyrène	Produits de la pêche & viandes	LEAV 85	Règlement 333/2007	1 kg
Pesticides organochlorés ou organophosphorés	Produits de la pêche	LEAV 85	Directive 2002/63/CE	500 g

Matrice	Taille du lot	Echantillonnage	
		Reg. CE 333/2007 (métaux lourds) & 1883/2006 (dioxines & PCB-DL)	Dir. 2002/63/CE (pesticides)
Lot en vrac	< 50 kg - L	3 échantillons	3 échantillons
	50 à 500 kg - L	5 échantillons	5 échantillons
	> 500 kg - L	10 échantillons	10 échantillons
Lot emballé	< 26 unités	1 unité	1 échantillons
	26 à 100 unités	5 % d'unité, minimum de 2	5 échantillons
	> 100 unité	5 % d'unité, maximum de 10	10 échantillons

Notes :

- Laboratoire d'envoi défini par la note de service DGAL/SDPPST/N2009-8053 du 05/02/2009 : laboratoires réalisant des analyses officielles dans le cadre des plans de surveillance et des plans de contrôle de la contamination des denrées animales et d'origine animale, et des produits destinés à l'alimentation animale pour l'année 2009
- Poids minimal de chaque échantillon (pour reg. CE 333/2007 & 1883/2006) : 100 g
- Cas particulier des poissons entiers de grande taille (pour dioxines et PCB-L) : se référer au reg. 1883/2006



PRODUITS DE LA PECHE

ANALYTES BIOLOGIQUES

HISTAMINE

Lorsque certains poissons commencent à se décomposer, de l'histamine est libérée. L'histamine peut déclencher une réaction allergique grave lorsqu'elle est absorbée en doses élevées.

La présence de teneurs élevées en histamine indique toujours que la décomposition a commencé, même si elle n'est pas visible. Des quantités toxiques d'histamine peuvent être présentes avant que l'odeur ou le goût du poisson ne soient désagréables.

L'empoisonnement par l'histamine est un type d'intoxication causé par la consommation de certains poissons qui ont été incorrectement manipulés et/ou mal conservés. Certaines familles de poissons présentent un risque particulier de développement d'histamine.

Méthode de prélèvement :

Produit	Matrice	Quantité minimum à prélever	Nombre d'échantillons par prélèvement	Matériel nécessaire	Conservation
Scombridae, Clupeidae, Engraulidae, Coryphenidae, Scomberesicidae, Xiphiidae, Istiophoridae ...	Poissons entiers ou morceaux selon la taille du poisson	200 gr	9 échantillons sur individus différents	Sachet plastique stérile ou emballage d'origine	Congélation possible

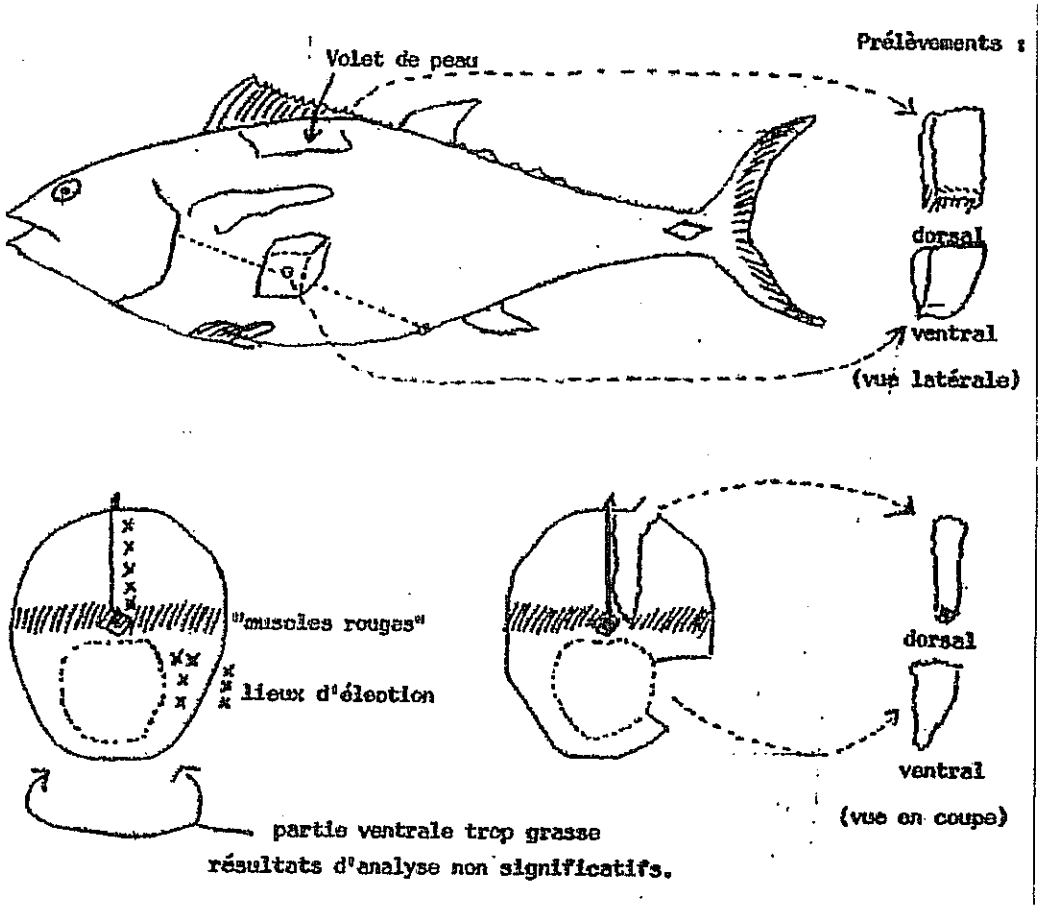
Laboratoire :

Laboratoire central de l'AFSSA
22 rue Pierre Curie
94706 MAISON ALFORT CEDEX
Tel : 01 49 77 13 14
Fax : 01 49 77 26 95

Tests :

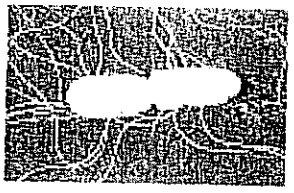
Laboratory tests subcategory : B.3 (c) : Santé publique
M ' histamine

: privilégier la partie abdominale du poisson (la queue ne représente aucun intérêt pour cette recherche).



Quelques poissons susceptibles de contenir de l'histamine :

Famille	Espèces	Noms communs en usage en anglais (y compris USA et Océanie)	Noms communs en français
Arripidae	*Arripis trutta*	sea-bass, sea-perch, kahawai, sea-salmon	loup de mer
Carangidae	*Seriola dumerili* (Risso) *Seriola lalandii*	amberjack, yellow-tail cape	seriole, limon
Coryphaenidae	*Coryphaena hippurus* (Linnaeus)	dolphin-fish, mahimahi	coryphène, mahimahi
Clupeidae	*Sardinella sirm* (Walbaum) *Amblygaster sirm* (Weber et De Beaufort) *Sardinops sp.* *Sardina pilchardus* (Walbaum) *Clupea harengus* (Linnaeus)	spotted sardinella, sprat, pilchard, sardine, herring	anchois de Norvège, sprat, sardinelle tachetée, pilchard, sardine, hareng
Istiophoridae	*Makaira (Tetrapterus) Audax* (Poey)	stripped marlin	marlin, makaire
Pomatomidae	*Pomatomus saltatrix* (Linnaeus)	bluefish	tassergal, poisson-serre
Scomberesocidae	*Cololabis saira* (Brevori)	skipper, mackerel pike, saury	balaou japonais, scombérésoce, samana
Scombridae	*Auxis thazard* (Lacépède) *Euthynnus alleteratur* (Rafinesque) *Katsuwonus pelamis* *Sarda sarda* (Bloch) *Scomber japonicus* (Houttuyn) *S. scombrus* (Linneaus) *Scomberomorus cavalla* (Cuvier) *S. maculatus* (Mitchill) *S. regalis* (Bloch) *Thunnus alalunga* (Bonnaterre) *T. albacares* (Bonnaterre) *T. obesus* (Lowe) *T. thynnus* (Linnaeus)	frigate mackerel, black skipjack, skipjack, bonito, pacific mackerel, atlantic mackerel, king mackerel, spanish mackerel, cero, albacore, yellowfin tuna, bigeye tuna, bluefin tuna	auxide, bonitou, thonine, listao, bonite à ventre rayé, bonite à dos rayé, bonite, sarde, maquereau espagnol, maquereau, thazard barré, sierra, thazard tacheté, thazard franc, germon, thon blanc, albacore, patudo, thon rouge
Salmonidae	*Salmo salar, Oncorhynchus sp.*	salmon	saumon
Siluridae	*Silurus glanis*	catfish	poisson-chat, silure
Xiphiidae	*Xiphia gladius* (Linneaus)	swordfish	espadon



PRODUITS DE LA PECHE
ANALYTES BIOLOGIQUES
BACTERIES

▲ Les prélèvements pour recherche de vibrios sont à envoyer dans les 24h ▲

La t°c doit toujours être < 15°c

Les risques microbiologiques liés aux denrées alimentaires constituent une source majeure de maladies d'origine alimentaire chez l'homme (toxi-infections alimentaires).

Les poissons sont parfois les hôtes de bactéries, par exemple *Listeria monocytogenes* ou les salmonelles. Ces bactéries peuvent être à l'origine d'infections graves, notamment chez les enfants et les personnes âgées. Dans la majorité des cas, un défaut dans la chaîne du froid est à mettre en cause.

Méthode de prélèvement :

Produit	Matrice	Quantité minimum à prélever	Nombre d'échantillons par prélèvement	Matériel nécessaire	Conservation
Produits prêts à être consommés : poissons fumés, crustacés, mollusques cuits, produits marinés ou salés et produits préparés cuits à base de chair de poisson.	Chair	100 gr	5	Sacs plastiques Stériles ou emballage d'origine	Réfrigération
Coquillages vivants	Coquillages	10 individus minimum	1	Sacs plastiques Stériles ou emballage d'origine	Réfrigération

Recherche Listeria monocytogenes :

-produits prêts à être consommées (poissons fumés, crustacés et mollusques cuits, coquillages vivants, produits marinés ou salés et produits préparés cuits à base de poisson.)

Recherche Salmonelles :

- **crustacés et mollusques cuits**

Recherche Vibrios :

- **Produits de la pêche**
- **Mollusques cuits**

Laboratoire :

Laboratoire central de l'AFSSA
22 rue Pierre Curie
94706 MAISON ALFORT CEDEX
Tel : 01 49 77 13 14
Fax : 01 49 77 26 95

Traces :

Laboratory tests subcategory : B.3 (c) : Santé publique

- Salmonelles / (M ! salmonella)
- *Listeria monocytogenes* / (M! listeria)
- Vibrions / (M ! vibrio)

FICHE DE PRELEVEMENT : métaux lourds Hg, ~~Pb~~, Cd

COMMEMORATIFS

PIF d'origine	Référence du prélèvement	Laboratoire destinataire :
Roissy CDG		LVD 14

Prélèvement effectué par (nom en majuscules) :

Téléphone :

Date du prélèvement :

Date de l'envoi du prélèvement :

Plan de surveillance ☐ Contrôle orienté ☐ Contrôle renforcé ☒ Contrôle par sondage ☐

Numéro du certificat sanitaire :

Numéro de l'annexe B :

Pays d'origine :

Pays de provenance :

Pays de destination (si autre que la France) :

Matrice prélevée : préciser :

Bovin ☐ Ovin (> 3 mois) ☐ Caprin (> 3 mois) ☐ Porcin ☐ Equin ☐	Poulet de chair ☐ Dinde ☐ Lapin ☐ Autres volailles ☐ Préciser :	Gibier à plumes ☐ Gibier à poil ☐ Préciser :	Poissons pêchés ☐ Poisson aquaculture ☐ Crustacés ☐ Coquillages : ☐ Préciser :	Lait ☐ Produits laitiers Préciser :

Signature de l'inspecteur :

ANALYSE	DEPISTAGE			CONFIRMATION*		
Date de réception						
Date d'analyse						
Envoi	Accepté ☐ Refusé ☐			Accepté ☐ Refusé ☐		
Numéro d'enregistrement						
Méthode d'analyse						
RESULTATS	Pb	Cd	Hg	Pb	Cd	Hg
Limite de détection						
Limite de quantification						
Résultat						
Valeur maximale de référence[1]				Observations :		
Résultats à confirmer	Oui ☐		Non ☐			
LABORATOIRE Nom Signature du responsable						

[1] Se reporter à la note générale

FICHE DE PRELEVEMENT : métaux lourds Hg, Pb, Cd

COMMEMORATIFS

PIF d'origine	Référence du prélèvement	Laboratoire destinataire
ROISSY CDG		LVD 44

Prélèvement effectué par (nom en majuscules) :

Téléphone :

Date du prélèvement :

Date de l'envoi du prélèvement :

Plan de surveillance ☐ Contrôle orienté ☐ Contrôle renforcé ☐ Contrôle par sondage ☒

Numéro du certificat sanitaire :

Numéro de l'annexe B :

Pays d'origine :

Pays de provenance :

Pays de destination (si autre que la France) :

Matrice prélevée : préciser :

Bovin ☐ Ovin (> 3 mois) ☐ Caprin (> 3 mois) ☐ Porcin ☐ Equin ☐	Poulet de chair ☐ Dinde ☐ Lapin ☐ Autres volailles ☐ Préciser :	Gibier à plumes ☐ Gibier à poil ☐ Préciser :	Poissons pêchés ☐ Poisson aquaculture ☐ Crustacés ☐ Coquillages : ☐ Préciser :	Lait ☐ Produits laitiers Préciser :

Signature de l'inspecteur :

ANALYSE	DEPISTAGE			CONFIRMATION*		
Date de réception						
Date d'analyse						
Envoi	Accepté ☐ Refusé ☐			Accepté ☐ Refusé ☐		
Numéro d'enregistrement						
Méthode d'analyse						
RESULTATS	Pb	Cd	Hg	Pb	Cd	Hg
Limite de détection						
Limite de quantification						
Résultat						
Valeur maximale de référence[1]				Observations :		
Résultats à confirmer	Oui ☐		Non ☐			
LABORATOIRE Nom Signature du responsable						

[1] Se reporter à la note générale

Etablissement payeur : DDSV 93

fiche de prélèvement HISTAMINE : *COMMEMORATIFS*

PIF d'origine :	Référence du prélèvement :	Laboratoire destinataire :
Roissy CDG.		AFSSA MAISON ALFORT

Prélèvement effectué par (nom en majuscules) :
Téléphone :
Date du prélèvement :
Date de l'envoi du prélèvement :
Contrôle physique par sondage ☒ Contrôle orienté ☐ Contrôle renforcé ☐
Numéro du certificat sanitaire :
Numéro de l'annexe B :
Pays d'origine : Pays de provenance : Pays de destination :
Informations complémentaires sur les produits prélevés : N° du lot de production, Date de fabrication, de congélation, DLC ; DLUO ...

Matrice prélevée : préciser :
Etat : Réfrigéré ☐ Congelé ☐ Saumure ☐

Famille : *Scombridae* ☐ *Clupeidae* ☐ *Engraulidae* ☐ *Coryphaenidae* ☐
Préciser l'espèce :
Nombre d'échantillons élémentaires prélevés 9 **Nombre de résultats attendus** 9

Signature de l'inspecteur :

ANALYSE	**DEPISTAGE**	**CONFIRMATION***
Date de réception		
Date d'analyse		
Envoi	Accepté ☐ Refusé ☐	Accepté ☐ Refusé ☐
Numéro d'enregistrement		
Méthode d'analyse		
RESULTATS	Teneur moyenne < 100 ppm * / Deux échantillons peuvent avoir un teneur dépassant 100 ppm mais n'atteignant pas 200 ppm * /Aucun échantillon ne doit avoir une teneur dépassant 200 ppm * * les poissons qui ont subi un traitement de maturation enzymatique dans la saumure peuvent avoir des teneurs en histamine plus élevées mais ne dépassent le double des valeurs indiquées le double de ces valeurs	
LABORATOIRE Nom Signature du responsable Cachet du laboratoire		

DEPISTAGE

Echantillon	1	2	3	4	5	6	7	8	9

Si l'analyse à porté sur un seul échantillon, dans le cadre de faibles quantités reçues, indiquer le seul résultat obtenu

Résultats à confirmer : oui ☐ non ☐

CONFIRMATION

Echantillon	1	2	3	4	5	6	7	8	9

ETABLISSEMENT PAYEUR : Direction Départementale des Services Vétérinaires de
Opérateur : DDSV 93

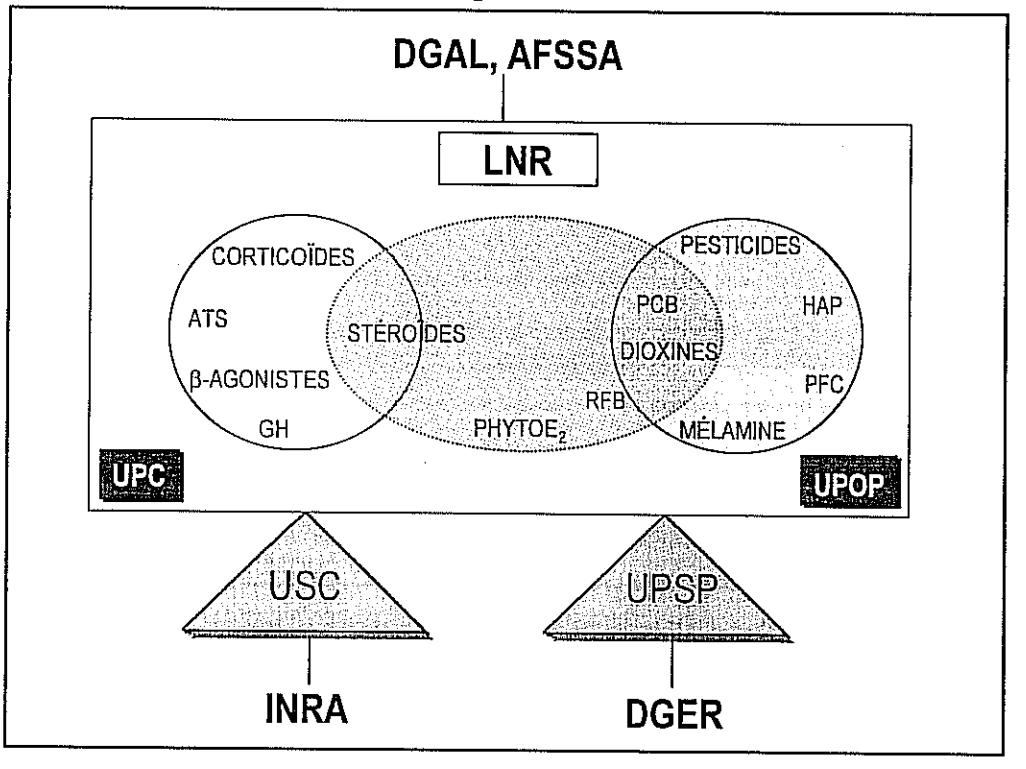
LABERCA

Laboratoire d'Étude des Résidus et Contaminants dans les Aliments

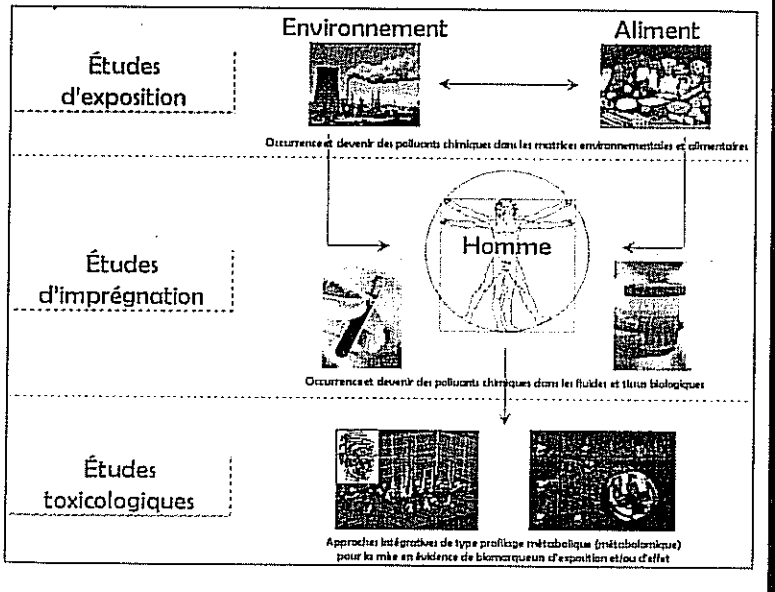
ÉCOLE NATIONALE VÉTÉRINAIRE DE NANTES
UPSP DGER 95995302, USC INRA 2013 http://www.laberca.org - laberca@vet-nantes.fr

Une unité de recherche (40 collaborateurs) au service d'un
LABORATOIRE NATIONAL DE RÉFÉRENCE

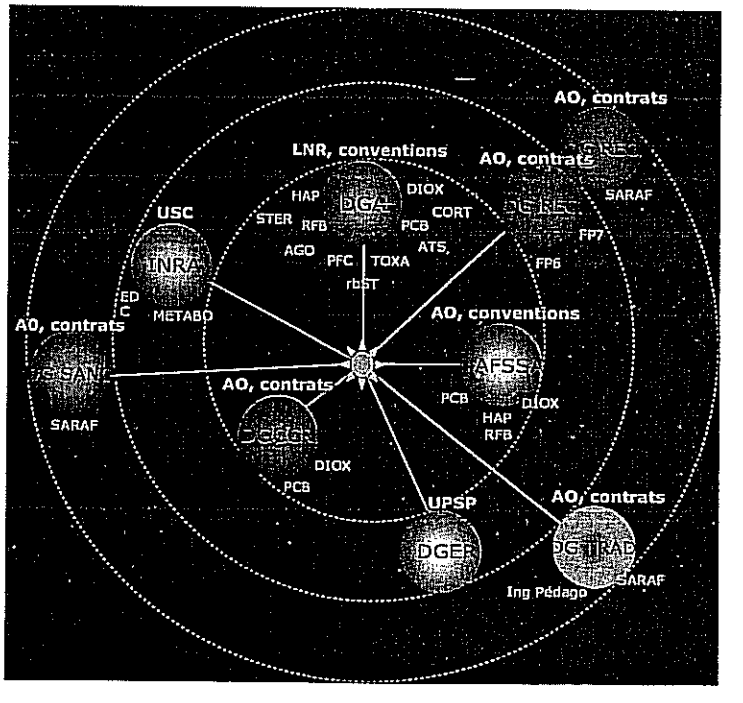
L'organisation



Les domaines de compétence



Partenaires, Prescripteurs et Financeurs



Un acteur de la recherche
RÉGIONALE, NATIONALE et **EUROPÉENNE**

Région PAYS DE LA LOIRE

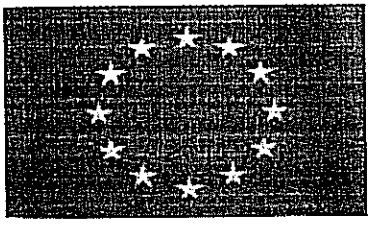
VEGEPOLYS — VALORIAL — Région BRETAGNE

Multiples projets à l'échelon régional
Conseils Régionaux Pays de la Loire, Bretagne
Pôles de Compétitivité VALORIAL, VEGEPOLYS

ANR AGENCE NATIONALE DE LA RECHERCHE

afsset — afssa

Quatre projets nationaux en lien
avec les différentes agences



SEVENTH FRAMEWORK PROGRAMME

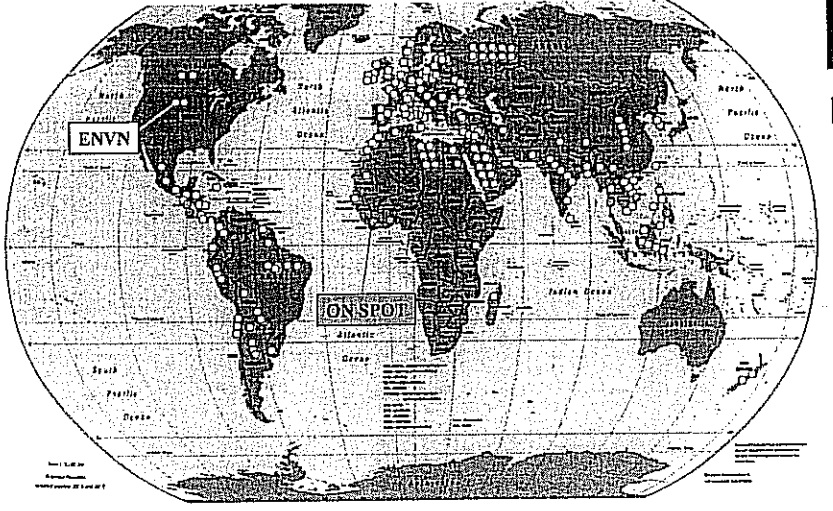
MARIE CURIE ACTIONS — PEOPLE

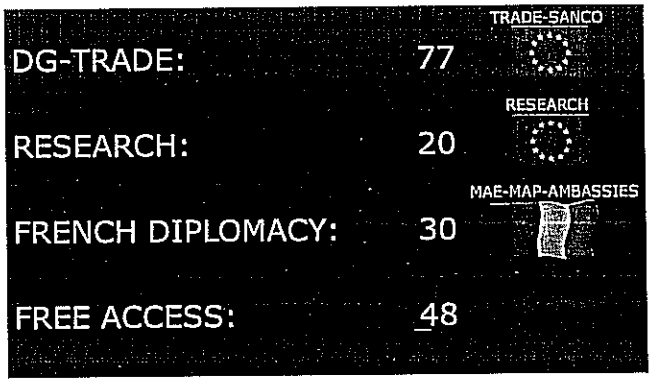
Implication forte dans les projets du 7ème PCRD et du 6ème PCRD

Une ingénierie pédagogique à visée **INTERNATIONALE**
pour la dissémination des connaissances générées par nos activités de recherche

SARAF

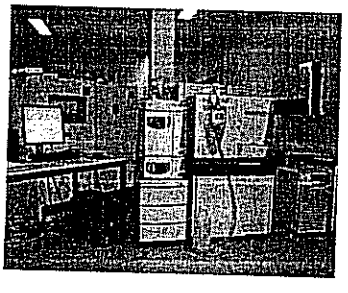
School for Advanced Residue Analysis in Food





- **ENVN : 180 participants > 2001**
  - 12 sessions dont
    - -3 sessions DG TRADE
    - -1 session DG RESEARCH
  - 68 pays dont 44 pays tiers
- **Sur site : 100 participants >2008**
  - 8 contrat DG TRADE
  - 2 semaines chacun
  - Am. centrale, Afrique, Asie, Océanie

LABERCA



LABoratoire d'Étude des Résidus et Contaminants dans les Aliments

École Nationale Vétérinaire de Nantes
Route de Gachet - BP 50707
44307 Nantes Cx 3
Tél : (33)2.40.68.78.80
Fax : (33)2.40.68.78.78

http://www.laberca.org

History

1979 Creation of « École Nationale Vétérinaire de Nantes ».

1980 1985 Development of a diagnostic activity in endocrinology applied first to cattle, then to pets. At the time, the Laboratory is known as **LDH** (Laboratoire des Dosages Hormonaux).

1986 First contacts with the DGAl (Direction Générale de l'Alimentation). The various skills acquired by the Laboratory in the fields of endocrinology and radioimmunology are exploited for the control of the use of anabolic substances in calves.

1989 The DGAl chooses to designate LDH as National Reference Laboratory (**LNR**) (European Directive 89/610/EC then 93/257/EC Directive). The Laboratory takes on the name **LDH/LNR** and separates from LDH which keeps their diagnostic activities.

1995 First **accreditation** of LDH/LNR to EN45001 standard.

2000 The Laboratory is nominated as National Reference Laboratory for the control of dioxins. It then becomes **LABERCA** (Laboratoire d'Etude des Résidus et Contaminants dans les Aliments).

2003 In July, the Laboratory becomes National Reference Laboratory for PAH in food. The research unit gets a seal of approval from **INRA** (ALIM H Department). In addition, the research activities led in the LABERCA receive their **first certification.**

2007 Publication of the hundredth article in a peer-reviewed international journal.

General Organisation

LABERCA

Presentation

The LABERCA is a **research unit** of the National Veterinary School of Nantes (ENVN) and belongs to the Ministry of Agriculture and Fishing. It is under the supervision of the Direction Générale de l'Enseignement et de la Recherche (**DGER**) and the Institut National de Recherche Agronomique (**INRA**) (Alim H Department) for its research activities in the field of food chemical safety. It is the **National Reference Laboratory** for dioxins, Polycyclic Aromatic Hydrocarbons (PAH) and growth promoters in cattle (Direction Générale de l'Alimentation - **DGAl**).

École Nationale Vétérinaire de Nantes

Organisation

The LABERCA is made up of 25 persons (teacher-researcher, research engineers and assistants, technicians and administrative staff). About ten master, PhD and post-graduate students come to complete the team. In order to meet the requirements of its supervising bodies, the LABERCA has created **2 Units**: the Growth Promoters Unit and the Contaminants Unit, backed up by a **team of researchers** (engineers and PhDs) and an advanced and comprehensive set of equipment.

INRA

Missions

For several years now, the LABERCA has been providing scientific and technical support to the Ministry of Agriculture (DGAl) and the European Commission (DG Trade and DG Sanco). In this respect, its main missions are as follows:

- **Methodological development**, essentially based on Mass Spectrometry under all its forms (low and high resolution, MS only or MS/MS, quadrupole or magnetic field based, coupled to liquid or gas chromatography) for the analysis of natural and xenobiotic hormonal substances at trace level in food matrices, fluids and biological tissues;
- **Technological transfer** of methods, developed and validated according to the applicable European criteria (Decision CE/2002/657), to a network of field laboratories;
- **Development of international relations** with the European Commission and the Community Reference Laboratories.

In addition to these scientific and technical support activities, research has been considerably developed within the LABERCA over the past ten years. The laboratory's research activities have been assessed and have obtained the support of DGER since 1995 and INRA since 2003.

LABoratoire d'Étude des Résidus et Contaminants dans les Aliments

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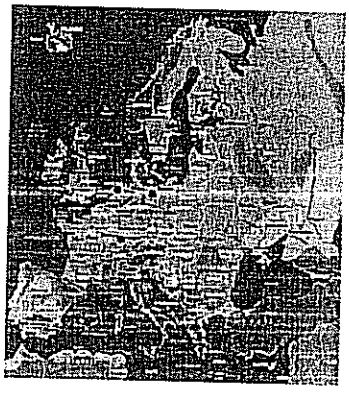
LABERCA

Reference Laboratory



LCRs européens compétents dans les domaines couverts par le LABERCA (Bilthoven, Berlin, Freiburg)

Laboratoires du réseau Promoteurs de croissance

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European level

In order to ensure **food safety** and thus maintain an efficient and consistent control system within the Union, the Commission has set up an overall provision of regulations (EC Directive 96/23) on the **control of residues and contaminants** in live animals and animal products. This monitoring system is based on a network of approved laboratories: Community Reference Laboratories (**CRLs**), National Reference Laboratories (**NRLs**) and **field laboratories.**
France has designated the **LABERCA** as **National Reference** Laboratory:

- In 1989 for group A (1 to 5) as defined in Annex I of EC Directive 96/23, ie. substances likely to have an anabolic effect and assimilated substances (stilbens, resorcylic acid lactones, ß-agonists, steroids and thyreostats) and group B(2f) (corticosteroids).

- In 2000 for group B (3f) as defined in Annex I of EC Directive 96/23, ie. polycyclic aromatic hydrocarbons (dioxins, PCDD/F, dioxin-like PCBs, PAHs...).

At a European level and within this network, the LABERCA is in contact with Community Reference Laboratories **RIVM** (Bilthoven, Netherlands) for residues in groups A1, 2, 3, 4 and B2f, **BfR** (Berlin, Germany) for residues in group A5 and **CVUA** (Freiburg, Germany) for residues in group B(3f).

National level

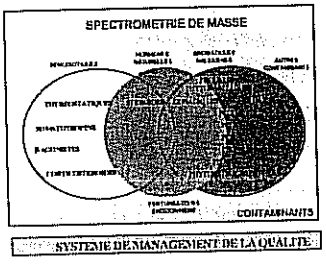
As National Reference Laboratory, the LABERCA is currently managing **three networks** of field laboratories:

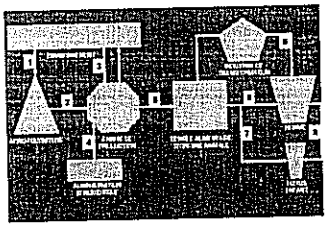
- The Growth Promoters network,
- The PCB and Dioxin network,
- The PAH network.

In this respect, the Laboratory must:

- **Coordinate** the work of the field laboratories in charge of residue analyses, in particular as regards methods.
- **Assist the competent authority** in the organisation of the residues control and monitoring plan.
- Periodically **organise interlaboratory assays** for each group of substances in order to evaluate the competence of the field laboratories.
- Ensure that national laboratories abide by the **set limits.**

LABERCA





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Research Activities

Summary

In a national and international context encouraging research in the field of **food safety**, and due to a greater awareness both from consumers and the authorities, the LABERCA's activities are focused on the study of the **chemical risk**. In this respect, two main categories of compounds can be identified, ie. on the one hand **residues** and on the other hand **contaminants**. Residues include **substances of natural origin** (among which gonadic steroid hormones, proteic hormones of somatotropin-type) and **xenobiotic substances** (among which anabolic steroids, corticosteroids, beta-agonists and thyreostats). Environmental contaminants comprise dioxins, PCBs, PBBs, PBDEs, PAHs or phytosanitary products. In these two groups, certain substances are suspected to have a disrupting activity on the endocrine system, which has been attracting the scientific community's attention for several years now, considering the direct consequences on consumers. Since 2003, the LABERCA has been giving more and more importance to this notion of **endocrine disruption**. The analysis of these molecules, most of the time present at trace level within complex biological matrices requires the use of ever more sensitive and specific identification techniques. **Mass spectrometry** under all its forms is therefore the laboratory's preference analytical tool when it comes to research projects in this general context. Research activities as a whole are being run according to our quality management system, which is **certified** to **ISO9001:2000** standard.

Period 2008-2011

Several **key actions** will be carried out over this period; the main expected results being the acquisition of a better knowledge of:

- the profile of environmental contaminants in farm animals feed;
- the transfer of these environmental and feed contaminants to farm animals and the consequence in terms of food contamination;
- Populations exposure at critical stages of their development (fœtus, new-born, pre-pubertal children) to endocrine-type contaminants (flame retardants, steroids, phytoestrogens);
- the consequence of certain industrial processes on food contamination (PAH, PFOA, PFOS);
- phase I/II metabolites of known (or yet unknown) families of growth promoters; their binding (integuments) and elimination (biological fluids) kinetics.

LABERCA

Quality Management

Accreditation and certification

cofrac
ACCREDITATION
COFRAC
N° 1-0549
Portée disponible
Sur www.cofrac.fr
ESSAIS

Initiated in 1992, the LABERCA's quality policy was first exclusively focused on **analytical testing services**. Under constant development, the LABERCA quality assurance system was later (2001) recognised true to the new applicable laboratory standard **ISO17025**.

In 2004, the LABERCA applied for an audit from the COFRAC to standard **ISO17025** with a **level 3 flexible range**. This new principle of flexible range implies that the laboratory is considered competent to adapt and set up, within the field covered by the standard range, any standardised or assimilated method and develop any other method which it will have had validated.
The general range concerning the growth promoters section is the following:
« **Monitoring of forbidden substances residues of growth promoters-type, corticosteroids, phytoestrogens in food, hair and biological matrices and fluids** ».
The general range concerning the contaminants section is the following:
« **Screening of organic contaminants:**
- **in food or animal feed;**
- **in beverages;**
- **in environmental matrices;**
- **in biological matrices of animal and human origin.** ».

MOODY
CERTIFICATION
FRANCE
ISO 9001-2000
N° d'enregistrement
2002212551
Gestion de projets de recherche :
Conception, conduite et valorisation

In addition, the LABERCA has initiated a quality validation process of its research activities. The Laboratory progressively turned towards the **ISO9001 version 2000** standard and worked on its quality management system in order to bring it up to the requirements of this standard. This considerable work bore fruit in March 2003 when the LABERCA was audited and approved to ISO9001v2000 regarding the **management of research projects: design, conducting and valorisation.**

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Training Activities

LABERCA

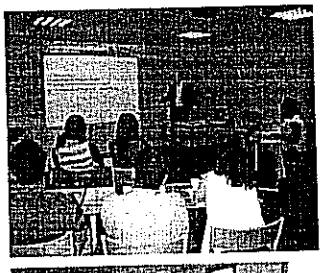
Higher Education

LABERCA's experience in the fields of analytical chemistry and food safety enabled teachers-researchers from the laboratory to take part in the training of the veterinary students, both at a basic and advanced stage of their courses. This teaching is particularly based on risk analysis methods (evaluation, management, communication). Besides, analytical chemistry (MS, RMN, IR, chromatography) is covered in order not only to reinforce the veterinary students' knowledge, but also in specialised studies for Master and continuing education students. As an example:

- ENVN:
- UV 51 « Basic scientific and legal tools and methods »
- UV 63 « Dangers for public health »
- UV 82 « Construction of quality and safety in food production chains »
- ENSV:
- « Control of residues and contaminants in food »
- ENITIAA:
- UV: « Food chemical and toxicological risk »
- ENSIA: UV « Physico-chemical food safety »
- Nantes University:
- M2R: Master in chemical and engineering sciences, with a speciality in chemistry of the living - chemical analysis and bio-products quality.
- M2P: Master in chemical and food engineering, with a speciality in food industry management and control.
- M2P: Master in chemical sciences and engineering, with a speciality in the analysis and control of industrial products.
- M2R: Master in chemical and food engineering, with a speciality in food and human nutrition sciences (SANH).

UNIVERSITÉ DE NANTES

SARAF



SARAF

In 2001, along with 5 European laboratories, the LABERCA implemented a training course (SARAF – School for Advanced Residue Analysis in Food), provided in English. This course is destined to international scientists willing to gain a high level of expertise in the field of residues and contaminants analysis in food. It accommodates two sessions every year of about twenty professionals each, originating mainly from third countries, for theoretical (2 weeks at the ENVN) and practical (2 to 3 weeks within the LABERCA or partner laboratories) sessions. The LABERCA can thus ensure officially (practical support to the Ministry of Agriculture and the Ministry of Foreign Affairs, DG SANCO, DG TRADE) and efficiently the transfer of the French know-how regarding the control of residues and contaminants in food.

Since 2004, the European Commission (DG TRADE) has annually selected the LABERCA, through a call for tenders, to organise a training on the physico-chemistry safety of food.

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Research News

LABERCA

The newsletter of the "Laboratoire d'Etude des Résidus et Contaminants dans les Aliments" of the Veterinary School of Nantes — *N°1 - June 2004*

Editorial

During its last direction council in January 2004, our laboratory highlighted a need for an improved internal and external communication, especially regarding its research activities.

Therefore, the idea of a new document was proposed to present briefly some of the running research projects and main results, in addition to the existing activity reports and publications.

So it is a real pleasure for me to open the first of these bi-annual newsletters. I thank its authors and designers, and I wish you a good and pleasant lecture.

François André

The LABERCA recognised at the European level

From the 10th to the 12th May at Noordwijkerhout (Netherlands), was held the 5th EuroResidue conference. Organised each 4 years by a committee linked to the Utrecht university, it includes all aspects of residues in food. The analytical, pharmacological, toxicological and regulation topics are especially covered. The contribution of the LABERCA was significant and remarked, through 1 plenary lecture, 2 oral communications and 8 posters. These presentations concerned both hyphenated technical aspects (ion suppression, isotope ratio mass spectrometry), and various applications (proteic hormones, endogenous and exogenous steroids, corticosteroids). An intense discussion was also engaged regarding the minimal required performance limits (MRPL) actually proposed in order to harmonise the methods of control in the EC.

For the first time since its first edition in 1990, this conference was closed with an award ceremony, with the objective to distinguish scientists who have marked the domain through a particularly significant contribution. The first award was then attributed to the Dr. Bruno Le BIZEC, head deputy of the LABERCA, for his reference work especially in the field of steroid hormones.

In Brief

• Continuous Education

The fourth session of the continuous education course "School for Advanced Residue Analysis in Food" (SARAF) will be organised from the 4th to the 15th October. All information on the Web site (http://www.saraf-educ.org).

• Quality assurance

The 21th April 2004, the quality assurance system developed for the "project management of research: organisation, control and valorisation", based on the ISO 9001 standard, was successfully audited for the second time.

Proteic hormones analysis: an on-going challenge

Growth hormones, also known as somatotropins, are proteins produced by the anterior pituitary gland. Biological effects of somatotropins are numerous and associated with growth, development and reproductive functions. Growth hormones are widely used outside Europe to stimulate milk production in dairy cows and as a general growth promoter in pigs. Human and equine growth hormones are thought to be widely abused in sports since the increase in muscle size and strength make them a viable alternative to anabolic steroids.

Recombinant DNA techniques allow the production of large quantities of recombinant growth hormones which may exhibit slightly different chemical structures from the pituitary somatotropin, by adding a number of amino acids on the N-terminal side (1 to 8). Recombinant bovine and porcine somatotropins from these sources, legally used in USA, are banned in EU. In order to anticipate a potential illegal use of these substances, specific and sensible methods are required to detect these molecules in biological fluids.

In this context, researches are carried out in LABERCA to detect the illegal administration of recombinant hormones. Identification of natural somatotropins and recombinant ones from different sources and species as well as discrimination in-between the different forms is now overcome through peptide-mapping method (LC-MS/MS). Present work is focused on the extraction and purification steps of these molecules from biological fluids in which they are found at the ppb level. Different approaches are developed which, for example, deal with immuno-affinity chromatography as well as two dimensional electrophoresis.

Endocrine disruptors: new exposure assessment studies

The endocrine disruption has been a research topic of growing concern for several years, both for scientists and public authorities, especially because of a close relation with food safety and consumer health. The LABERCA has been conducting for 2 years some projects dedicated to the development of efficient analytical methods for the identification of substances recognised as endocrine disruptors. A main objective of such developments is to ensure a robust basis for any further exposure assessment, and more generally risk assessment studies, that are actually missing at the national level. This first studies concerned compounds of natural (phytoestrogens) or synthetic (flame retardants) origin.

A first study was already devoted to a method development based on liquid chromatography-tandem mass spectrometry (LC-MS/MS), permitting the identification of 12 isoflavones, lignans and coumestans in milk samples, at concentration as low as 0.1-0.5 $ng.mL^{-1}$ (ppb). Extended data collection will be now engaged, the purpose being to be interested in the potential beneficial or negative effects of these ubiquitous molecules, especially for young children.

A second project, supported by the Environmental Sanitary Safety French Agency (AFSSE) and coordinated by the UMR INRA 3089 Xenobiotics laboratory, was initiated to evaluate the foetal exposure to the polybromodiphenylethers (PBDEs) and tetrabromobisphenol-A (TBBP-A). The LABERCA will ensure the analytical basis of this study, through a development based on high resolution mass spectrometry (GC-HRMS) realised through a university PhD thesis. This development will be also exploited in order to identify eventual metabolites of these compounds.

Research News

LABERCA

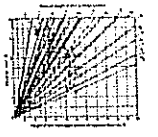
The newsletter of the "Laboratoire d'Etude des Résidus et Contaminants dans les Aliments" of the Veterinary School of Nantes — *N°1 - June 2004*

Analysis of Residues in hair: a promising improvement for the control

Urine, faeces, or tissue, are biological matrices used for many years in order to control the utilisation of xenobiotic agents in cattle. Hair represent an interesting alternative strategical matrix for revealing a misuse of steroids, due to an easy sample collection and a usual extended period of detectability. However, the mechanisms and incorporation kinetic of xenobiotics in the hair remained not fully understood and characterised. In this context, a project was initiated to study the fixation of médroxyprogesterone acetate (MPA), estradiol benzoate, and 17α-methyltestosterone in bovine hair.

An analytical development based on a specific purification associated to selective and sensitive measurement techniques (GC-MS/MS and LC-MS/MS) permitted to clearly decrease the limits of detection. The elimination kinetic was monitored during several months after intramuscular administration. Steroid residues were then detected during several months. Residues of estradiol esterified forms were also identified during several weeks. This observation permits to propose this matrix as a promising candidate to demonstrate an illegal misuse of natural hormones in cattle, the detection of the injected ester form revealing unambiguously an exogenous origin.

Mass spectrometry and statistics: a new analytical approach

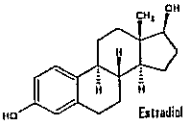


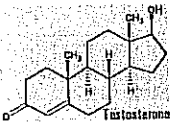
If the high interest of multivariate statistical techniques (MANOVA, PCA, LDA, classification,...) for the treatment of large data set is known for a long time, their utilisation in the field of analytical chemistry remains more recent. For several years, the LABERCA has been progressively introducing these techniques to analyse spectrometric data. The first applications were found very promising, and concerned the differentiation of stereoisomers (corticosteroids betamethasone and dexamethasone, androstan-diol steroids), on the basis of a global (MS in full scan mode) or specific (MS^2 in MRM mode) mass spectrometric fingerprint.

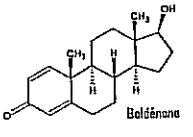
New projects are currently running using these techniques, with the objective to exploit their descriptive and explicative power, in order to investigate and explain the variability observed in term of contamination profile for the contaminant families constituted by different congeners such as dioxins.

This thematic is today the opportunity to collaborate with the UMR INRA Chemometry and Sensometry of the National School of Engineers for Agriculture and Food Industries (ENITIAA), Nantes, France.

The difficult case of endogenous hormones: new results for a European need







The research of xenobiotic anabolic steroids in biological matrices such as urine, tissue, or hair is today usually well controlled, in a context of food safety, human antidoping or horseracing control. The situation is clearly different when the purpose is to control the misuse of natural steroid hormones, in other words to distinguish the residue origin and ask the question: production by the organism or consequence of an exogenous compound administration ? Indeed, the administration of theses substances induces only minor change in their residual concentrations in tissue or excreta's. These variations are therefore usually considered as non significant compared to the natural physiological variations between and within specimen.

The research projects conducted at the LABERCA since 1997 in this field today lead to very promising results. The actual approach developed by the laboratory consists in the differentiation of the origin – endogenous or exogenous – on the basis of the $^{13}C/^{12}C$ ratio of metabolites and precursors of these steroids in bovine urine, using gas chromatography-isotope ratio mass spectrometry (GC-C-IRMS). The precursors, also named endogenous reference compounds (ERC), are characterised by an isotopic composition non modified by an administration of gonadic steroids such as testosterone, estradiol, progesterone, or nandrolone, that appears at the end of the metabolic pathways. These synthetic anabolic steroids have the particularity to present an isotopic composition decreased compared to the endogenous sources. At the opposite, the $^{13}C/^{12}C$ ratio of DHEA and 5-androstene-3β,17α-diol (ERC) reflects the endogenous isotopic composition in carbon, directly depending on the alimentation specific of each animal. Thus, the observation of a difference between precursors and metabolites in term of $^{13}C/^{12}C$ ratio permits to suspect an exogenous administration of gonadic steroid hormones in cattle, over several weeks after the treatment. These results are currently obtained in a European project of the 5th FP and by a university PhD thesis.

Last Publications

- Pinel G, André F and Le Bizec B. Discrimination of recombinant and pituitary-derived bovine, porcine and human growth hormones by peptide mass-mapping. Journal of Agricultural and Food Chemistry 2004;52(3):407-414.
- Antignac J-P., Cariou R., Le Bizec B. and André F. New data regarding phytoestrogens content in bovine milk. Food Chemistry 2004;87(2):275-281.
- Antignac J-P., Monteau F., Négriolli J., André F. and Le Bizec B. Application of hyphenated mass spectrometric techniques applied to the determination of corticosteroid residue in biological matrices. Chromatographia 2004;59:S13-S22.
- Le Bizec B, Maume D, Marchand P, Monteau F, Bichon E, André F. Review: control of anabolic steroid in breeding animals: mass spectrometry, a powerful analytical tool. Chromatographia, 2004;59:S3-S11.

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Research News

LABERCA

The newsletter of the "Laboratoire d'Etude des Résidus et Contaminants dans les Aliments" of the Veterinary School of Nantes *N°2 - December 2004*

Editorial

The first issue of the LABERCA research newsletter was favourably received. Here is the second issue; some aspects non treated before are presented.

It summarises some research results included in the official area of the laboratory (MAAPAR/DGAL), but also other ones around this scope, for example regarding PAH or pesticides.

With all my best wished for the new year 2005, I hope you will have a pleasant lecture.

François André

The LABERCA involved in the 6th FP

In the 6th FP, the laboratory will participate to a European research project (IP) since January 2005. This project called "BIOCOP", will start next January and will run over 5 years, involving about 40 partners. Its main objective is the development of new analytical strategies, based on the emerging techniques such as transcriptomic and proteomic, for screening of residue and contaminants in food. The current controls, which are based on a limited number of selected target analytes, today appears clearly limited in term of large scale screening.

Therefore, the proposed systemic and innovative new approach will consist to study the global effect of the concerned substances, rather than looking for their identification. Thus, the challenge will be to identify some relevant biomarkers (RNA or proteins) characteristic of these compounds. This project will concern various classes of molecules including toxins, pesticides, steroid hormones, quinolones, phytoestrogens, and heavy metals. The LABERCA will be responsible for the workpackage dedicated to phytoestrogens, and will be also actively involved in the workpackage related to growth promoting factors. The laboratory will be finally in charge of the scientific dissemination.

In Brief

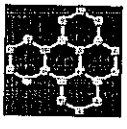
• Continuous Education

In answer to a request of the European Commission, a special SARAF session (School for Advanced Residue Analysis in Food) was organised from the 22nd November to the 3rd December 2004 (20 participants/15 nationalities)

• Quality assurance

LABERCA was invited to present its quality management system dedicated to its research activities (ISO 9001) through an oral communication in a congress organised by the Belgium accreditation body (Melle, 20th October 2004).

Polycyclic aromatic hydrocarbons: characterisation of the food intake



The Polycyclic Aromatic Hydrocarbons (PAH) are organic compounds constituted by two to seven benzene rings. For humans, the main ways of exposure to these substances are the ambient air (depending on various factors including urban traffic and industrialisation), as well as food intake through products contaminated by environmental sources and/or specific food processes such as smoking, roasting or torrefaction. For a non smoker individual, food remains the most important exposure vector to PAH, with global intake estimated to 3 µg/day. For a smoker, this value may be increased by a factor 2.

The French Agency for Food Safety (AFSSA) established in 2003 a list of 11 indicator PAH to evaluate the risk associated to these compounds through food intake. The National Institute for Industrial Environment and Risk (INERIS) also published in 2003 a report validating the AFSSA recommendations with the addition of a proposed virtually safe dose equal to 5 ng/kg/bw/day, based on Toxic Equivalent Factors (TEF).

An analytical method dedicated to the identification and quantification of these contaminants in various food products was developed, using gas chromatography coupled to mass spectrometry (GC-MS). This study is accompanied by a more basic study led in collaboration with the Ecole Nationale Supérieure d'Agronomie et des Insdustries Alimentaires (ENSAIA), with the objective to asses the transfer of PAH in ruminant from feed to milk. This project is expected to improve the knowledge regarding the metabolism of PAH and especially determine PAH markers in milk.

Phytosanitary products: some projects for a regional concern

Since 1999, the laboratory participated to 3 research projects involving regional partners (Bretagne and Pays de la Loire) in the field of phytosanitary products. The objective of the first project was to evaluate the transfer of some phytosanitary products especially used at the regional level, to food products from vegetal (carrots, spinach, apples, baby-food) and animal (milk, meat) origins. It has been demonstrated that all these samples were compliant according to the current regulation (observed concentrations far below the maximal residue limit).

The second project consisted to study the eventual contamination of marine molluscs by 3 herbicides families widely used at the regional level (phenylurea, triazins, aminophosphates). After an important analytical development, assays were realised on samples collected in various locations, supposed to be particularly exposed; no trace of contamination was observed. A complementary study realised on oysters incubated with diuron permitted to show that the concentration retrieved in oysters was 5 to 10 times higher than the concentration measured in water.

The last project actually running is dedicated to the two insecticides highlighted by bee keepers: fipronil and imidaclopride. The aim is on one hand to evaluate the contamination of surface water in South Vendée, and on the other hand to study the eventual transfer of fipronil from the maize seed to the milk. This last study is realised in collaboration with an experimental farm "Les Trinottières" in Maine et Loire.

Research News

LABERCA

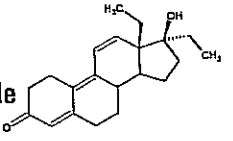
The newsletter of the "Laboratoire d'Etude des Résidus et Contaminants dans les Aliments" of the Veterinary School of Nantes — *N°2 · December 2004*

Thyreostats: a new strategy for the control of their illegal use

With their property to inhibit the natural production of thyroid hormones, thyreostats disturb the metabolism pathways under their direct dependence. As a result, an extracellular water retention as well as a reduction of the tractus transit may be expected through their illegal use in breeding animals, with alteration of the muscle quality. Moreover, the presence of thyreostat residues represent a carcinogenic and teratogenic risk. The extraction of these small, highly polar, and amphoteric molecules from biological sample, as well as their measurement represent a real analytical challenge.

Therefore, a innovative derivatisation reaction was developed. The interest of this chemical modification is first to stabilise all possible tautomeric forms characteristic of these molecules under a unique and less polar structure, which facilitate the extraction. Then, the addition of the derivatisation agent led to an increase of the molecular weight of the molecule, permitting to improve significantly the signal specificity. Finally, the obtained derivative presents a diagnostic signal with higher sensitivity and the possibility to be fragmented in tandem mass spectrometry (that was impossible on the original molecule). A validation is currently running on urine sample according to 2002/657/EC Decision.

Tetrahydrogestrinone (THG): the new anabolic steroid now detectable



Despite the intensity of the controls realised within the EU to survey the illegal use of steroid growth promoters, the number of non compliant samples reported in the main species of breeding animals is clearly decreasing since the end of the 90's. This observation may be explained by various factor, including the existence of new compounds belonging to the classical families of growth promoters, or the apparition of new classes of compounds. Tetrahydrogestrinone (THG) is one of these new substances recently discovered, revealed in 2003 by the USADA (U.S. Antidoping Agency).

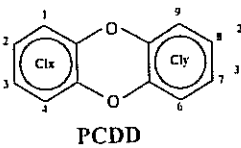
The LABERCA immediately launched a research project dedicated to the development of efficient method permitting the identification of this molecule based on liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS). The metabolism of this drug in bovine was also investigated after intra-muscular or oral administration. The results demonstrated that the more appropriate biomarker remains the non-modified parent drug, with minor proportion of conjugated forms, both in urine and faeces. The method was then applied to hundred urine samples (80% bovine and 20% porcine) coming from the French monitoring plan. No trace of THG was detected in these sample, at a decision limit (CCα) estimated to 0.1 ng.mL-1 (ppb).

Persistent organohalogenated contaminants: the analytical tool developed by the national reference laboratory today permits large scale risk exposure studies

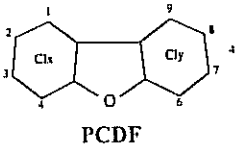
The LABERCA was designed in 2000 as National Reference Laboratory for the control and analysis of dioxins and related compounds in food. The competence acquired in the field of gas chromatography coupled to high resolution mass spectrometry (GC-HRMS) permitted to develop efficiently and rapidly some analytical methods for which it is today accredited (ISO 17025 standard). The missions of this unit (official survey programs, organisation of ring tests, trainning,...) are realised by a team of 5 individuals. Some research project were initiated since the creation of the unit. One of them, funded by Bretagne and Pays de la Loire and realised in collaboration with the Ecole de Chimie de Rennes (LARCIP) is now finalised.

The objective of this study was to improve the analytical competences at the regional level, as well as to collect data regarding the occurrence of these contaminants both in the sources of emission and in food products. Four industrial sites were monitored in 2003, in term of emission and evaluation of environmental rejections around the sites.

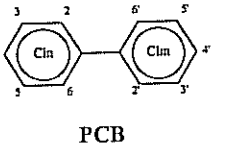
The collected samples included soil (as marker of long term exposure), grass (as marker of short term exposure), and milk from 10 farms selected considering their location from the source and position compared to winds. The results demonstrated than no sample presented a dioxin content higher than the authorised limit during the year, even if a clear seasonality phenomenon was revealed whatever the considered site. The examination of the full patterns given by the concentrations of the different analysed congeners demonstrated that an isolated measurement is insufficient to identify a source of pollution.



PCDD



PCDF



PCB

Last Publications

- Antignac J-P., De Wasch K., Monteau F., De Brabander H., André F. and Le Bizec B. The ion suppression phenomenon in liquid chromatography-mass spectrometry and its consequences in the field of residue analysis. Analytica Chimica Acta, *in press*.
- Meunier-Solère V, Maume D, André F and Le Bizec B. Pitfalls in trimethylsilylation of anabolic steroids: new derivatisation approach for residue at ultra-trace level. Journal of Chromatography B, *in press*.
- Pinel G, Buon R, Aviat F, Larre C, André-Fontaine G, André F, Le Bizec B. Assessment of Western blotting and 2D-electrophoresis methods on biological samples for the demonstration of its administration. Analytica Chimica Acta, *in press*.
- Rambaud L, Bichon E, Cesbron N, André F and Le Bizec B. Study of 17β-estradiol-3-benzoate, 17α-methyltestosterone and medroxyprogesterone acetate fixation in bovine hair. Analytica Chimica Acta, *in press*.

Research News

LABERCA

The newsletter of the "Laboratoire d'Etude des Résidus et Contaminants dans les Aliments" of the Veterinary School of Nantes — *N°3 - June 2005*

Editorial

I am pleased to present the 3rd issue of the LABERCA newsletter. Previous issues have been well appreciated, considering your reactions and regular sending requests.

The present issue presents some of our research projects having in common to improve the knowledge regarding environmental contaminants in food and feed.

I wish you a pleasant reading.

François André

The LABERCA and the 6th FP

The official launching meeting of the BIOCOP project was held in Brussels the 15th and 16th march 2005. As announced in our last newsletter issue, this integrated project of the 6th FP, coordinated by the Queen University of Belfast (Dr. Chris Elliott) is planned on 5 years and involves around 36 partners. Its main objective is the development of new analytical strategies for residue and contaminant in food, based on emerging techniques such as transcriptomic or proteomic. As responsible of the WP7 "endocrine disruptors", the LABERCA is working on the development of confirmatory methods (based on LC-MS/MS) for phytoestrogens in milk, cereals, and baby-food samples. The laboratory also participates actively to the WP8 "growth promoters", especially through the development of an direct identification method for peptidic biomarkers in plasma, susceptible to reveal the administration of anabolic steroids or corticosteroids. Finally, as responsible of the training activities organisation, the LABERCA will use its official teaching structure, i.e. SARAF (School for Advanced Residue Analysis in Food) in order to harmonise the content and the organisation of the different training courses and workshops proposed during the all project duration.

In Brief

• Continuous Education

The 6th session of the continuous education course SARAF (School for Advanced Residue Analysis in Food) will be organised from the 3rd to the 14th October 2005 (http://www.saraf-educ.org).

• Quality assurance

The certification of the quality management system developed by the laboratory for its research activities (according to the ISO 9001:2000 standard) was recognised and prolonged for the third time following the last audit the 28th April.

The isotopic approach (IRMS): new results for a 5th FP European project

The LABERCA is involved since January 2002 in one of the last 5th FP project – ISOSTER – dedicated to the development of innovative analytical methods for the demonstration of the illegal administration of steroid hormones in meet producing animals. Nine partners participate to this project, including laboratories in charge of anti-doping in sport (for example from Cologne, one World reference in this field) or in horseracing (from Newmarket, HFL), some national reference laboratories (NRL) specialised in chemical food safety (BfR, Berlin – TNO, Zeist – QUB, Belfast – CSL, York), as well as two of the main mass spectrometry companies providing IRMS instruments.

The measurement tools and the analytical strategy are now developed, and their robustness was demonstrated by a proficiency test involving ten invited laboratories. This method authorises the unambiguous discrimination in term of isotopic ratio ($^{13}C/^{12}C$) between precursors (or endogenous reference compounds such as DHEA or 5-androstenediol) and metabolites (etiocholanolone, androstanediol or 17α-estradiol) of the administered anabolic steroid.

This approach permits the unambiguous demonstration of steroid misuse up-to several weeks after the administration. Results have also demonstrated the influence of animal feed on the $^{13}C/^{12}C$ ratio of the monitored substances, but without affecting the diagnostic capability of the method. These results represent a major advance in the field of control, especially for the "double status" steroids, i.e. both endogenously secreted by the organism and potentially exogenously administrated.

Persistent organic pollutants (POP's): a large scale exposure assessment study in seafood

The LABERCA participates to a research project – POPINRA – coordinated by J-C Leblanc (National Institute for Agronomic research), which major objective was the determination of the occurrence of main POPs in various fish species and other seafood products. Because of their position in the food chain and their relatively high fat content (at least for certain fish species), these products are indeed recognised as particularly sensitive to these category of lipophilic environmental contaminants. Moreover, the investigation of the contamination profiles (relative proportions of the different monitored substances and congeners) may sometime be helpful in order to determine the contamination sources and the associated type of pollution.

For each sample, the monitored POPs included 35 molecules: 17 PCDD/PCDF congeners, 18 PCB congeners (12 characterised by toxicological properties similar to dioxins – "dioxin-like" PCB and 6 other presenting distinct effects – "indicator PCB"). The quantification of 7 brominated flame retardants (PBDE-28, 47, 99, 100, 153, 154 and 183) will be also performed on several samples.

This large scale and innovative study, coupled to simultaneous measurements for heavy metals, will undoubtedly lead to a very informative interpretation and a solid basis for further discussion. On the other hand, through the collection of 5565 data (number of measurement results estimated on the basis of 159 samples and 35 monitored analytes), this study will give a response to a need largely underlined at the European level regarding the risk assessment of these substances in France.

Research News

LABERCA

The newsletter of the "Laboratoire d'Etude des Résidus et Contaminants dans les Aliments" of the Veterinary School of Nantes — *N°3 - June 2005*

Growth hormone and Secretagogues: a new competence now well established

The anabolic properties of the growth hormone (GH, somatotropin) are potentially used in cattle or horseracing in order to improve performances. The LABERCA and the horseracing laboratory (LCH, Verrière le Buisson, France) are then concerned by this problematic. Since September 2004, a Cifre PdD thesis was initiated between the two laboratories. The main purpose of this shared research project is the demonstration of the illegal administration of recombinant growth hormones, especially in horse.

Behind the study of the growth hormones as such, the interest of the LABERCA is also focused on substances susceptible to induce and promote the natural secretion of somatotropins : the secretagogues. A master student was then in charge of this subject, with main objective to develop efficient analytical tool authorising the identification of these substances in biological matrices. The final report of this study was defended the 4th July 2005.

This "growth hormone" thematic of the LABERCA appears more and more recognised; a review presentation is planned to be prepared for the next International Symposium on Hormone and veterinary Drug Residue Analysis which will be held inAntwerpen (Belgium) next 16-19 May 2006.

Endocrine disruptors: Three classes of compounds particularly surveyed

Endocrine disruption is a growing concern for the scientific community for several years. Indeed, an increasing number of chemical substances are regularly pointed out because of their potential negative impact on human health, especially for the reproduction and development functions and particularly sensitive populations (foetus, young children). For this kind or residue and contaminants, it is now well established that food intake represent, with the environment, a major route of exposure to these substances. In this context, and according to his contract with the AlimH department of the National Institute for Agronomic Research (INRA), the LABERCA is interested since 2002 by three families of compounds for natural (steroid hormones, phytoestrogens) or synthetic origin (brominated flame retardants). Current researches focus on one hand to (re)-evaluate the endogenous production of natural sexual estrogens in the concerned population (prepubertal children), on the other hand to characterize the exposure to these substances (direct intake in case of foetus or food intake for young children), and finally to determine the hormonal activity associated to this intake. The expected results would probably permit both to a data set clearly missing at the national and European level, and to reach a better characterisation of the eventual danger associated to these compounds.

Insecticides (Fipronil and Imidaclopride) : toward the evaluation of human exposure

In front of the abnormal mortality of bees observed by the beekeepers professional organisations, a study was initiated by the LABERCA in order to determine the presence of Fipronil and Imidaclopride residues in honey. With main objective to anticipate potential phytosanitary crisis, the developed method was extended to various food matrices.

The first work focused on honey and milk samples, as representative of natural and baby-food products, respectively. Limits of quantification obtained after validation of the methods were found to be fit-for-purpose regarding the expected concentration, i.e. around 25 $ng.L^{-1}$ for fipronil and lower than 1 $\mu g.L^{-1}$ for chloronicotinic residues (imidaclopride, nitenpyram, thiamethoxam, acétamipride, thiaclopride and clothianidine) in milk. For honey, limits of quantification were found to be lower than 5 $\mu g.kg^{-1}$.

Theses methods were applied on a preliminary sample set consisting in various commercially collected products. Honey samples have been analysed, some of them (from importation) showed the presence of imidacopride and thiaclopride residues.

Boldenone : a new control strategy under investigation

Boldenone is an anabolic steroid characterised by its androgenic activity; its use is strictly forbidden in cattle within the EU. The control of its illegal use was based on the identification of 17α-boldenone in urine. Recent studies showed a possible but non systematic endogenous production of this steroid in bovine. A research project dedicated to the metabolism study of boldenone in cattle was started in LABERCA. Potential marker phase I metabolites were identified in urine (5β-androst-1-en-17α-ol-3-one (M2) and 5β-androst-1-en-17β-ol-3-one (M4)) as well as phase II metabolites such as 17β-boldenone sulphate and glucuronide.

Two analytical methods were developed in LABERCA, the first relying onto specific separation of free, glucoro- and sulphoconjugates, specific hydrolysis and GC-MS/MS (EI) analysis, the second onto the direct measurement of conjugates by LC-MS/MS (ESI). This last approach would constitute a major improvement and a definitive analytical tool for the demonstration of the illegal use of this anabolic steroid in cattle.

Last Publications

- Antignac J-P., Brosseaud A., Gaudin I., André F. and Le Bizec B. Analytical strategies for the direct mass spectrometric analysis of steroid and corticosteroid phase II metabolites. Steroids, 2005;70:205-216.
- Le Bizec B., Antignac J-P., Bertrand D., Qannari El M. and André F. Multidimensional statistical analysis applied to electron ionization mass spectra to determine steroid stereochemistry. Rapid Communication in Mass Spectrometry, 2005;19(4):509-518.
- Pinel G, Bichon E, Pouponneau K, Maume D, Andre F, Le Bizec B. Multi-residue method for the determination of thyrostats in urine samples using LC-electrospray-MS/MS after derivatisation with 3-iodo-benzyl-bromide. Journal of Chromatography A, 2005, *sous presse*.
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- Debrauwer L, Riu A, Jouahri M, Rathahao E, Jouanin I, Antignac JP, Cariou R, Le Bizec B, Zalko D. Probing new approaches using atmospheric pressure photoionization for the analysis of brominated flame retardants and their related degradation products by LC-MS. Journal of Chromatography A, 2005, 1082(1):98-109.

Research News

LABERCA

The newsletter of the "Laboratoire d'Etude des Résidus et Contaminants dans les Aliments" of the Veterinary School of Nantes *N°4 - January 2007*

Editorial

This 4th edition of LABERCA's Newsletter details some of the main happenings in our Laboratory during the end of 2005 and the year 2006.

This period has been particularly busy for all members of our team, in terms of research projects, paper submissions, organisation of trainings and events...

I leave you to discover the contents of our « Research News », We will be in touch soon with a new one!

Pf. Bruno Le Bizec

OAV and COFRAC audits

The LABERCA was audited during the OAV Mission, organised in France from 21 June to 1 July 2005, for the control of residues and contaminants in live animals and in food of animal origin. The OAV report on this mission showed no dysfunctioning of the LABERCA as National Reference Laboratory.

The LABERCA's accreditation audit pertaining to the laboratory's competence in terms of « design, development and validation » (flexible range) took place from 30 June to 1 July 2005. All activities were found compliant during this audit. The renewal and extension of type 3 flexible range accreditation for the LABERCA were granted for a period of **five years**, from **01/01/2006** to **31/12/2010**.

In Short

- **Change in GPU Management**

As from 19 January 2006, Yoann DECEUNINCK has been appointed as Manager of the LABERCA's Growth Promoters Unit.

Emmanuelle BICHON, who held this position for the previous two years, has now joined LABERCA's Technical Support and Innovation team.

PhD research: PhD defended and new PhDs

Three PhDs were successfully defended by the Laboratory's students between end 2005 and the beginning of 2006. Mrs Véronique Meunier-Solère presented her work on « the Study of natural steroid Androgens and Oestrogens in bovines: methodological contribution and metabolic inventory».

Miss Corinne Buisson defended her PhD work on « Developing an analytical strategy based on isotopic ratio Mass Spectrometry applied to the control of the fraudulous use of steroid hormones in cattle ».

Finally Mr Ronan Cariou presented the results of his PhD on the «Evaluation of the fœtus and newborn exposure to endocrine disrupters such as bromated flame retardants ».

All these studies enabled the Laboratory to reinforce its analytical experience in the field of residues and contaminants (ultra-trace measurement and use of innovative tools such as IRMS) and to broaden its scope of analysis to fields related to human health.

New PhD research work has now been initiated, in particular through « CIFRE » grants, a programme which enables a cooperation between private companies and public research institutions. Miss Blandine Destrez has started her work on the « Development of identification strategies of new active growth accelerating compounds in production animals » and Marie-Hélène Le Breton is working on a project related to the rbST growth hormone.

Polycyclic Aromatic Hydrocarbons : A new reference Activity

Polycyclic Aromatic Hydrocarbons (PAH) are organic molecules made up of 2 to 7 aromatic cycles and belong to the Persistent Organic Pollutants family (POP). They are carcinogenic, genotoxic and mutagene compounds which are inclined to bioaccumulation within the food chain. Food being the major source of exposure to these substances for Man, it is essential to be able to measure these molecules and their occurrence better, in order to get a better control of food contamination.

In this general context, the DGAl, French Food Directorate, appointed the LABERCA as National Reference Laboratory for the control of PAH in July 2005. In this respect, the laboratory's missions are numerous:

- Design and validate official measurement methods of PAH in food matrices (solid, liquid and oily).

- Participate to the Inter-Laboratory studies (EIL) organised by the European Commission in order to ensure the proper performance of the laboratory.

- Create a network of application laboratories and transfer the LABERCA's know-how to these structures in order to carry out the national control plans.

- Organise national Inter-Laboratory Studies to control the analytical capacity of the application laboratories and supply the results to the DGAl for them to deliver the agreements to the approved laboratories.

The objectives set by the DGAl have now been reached and a network of laboratories has been created to ensure the control of feedstuff in France as from 2006.

Research News

LABERCA

The newsletter of the "Laboratoire d'Etude des Résidus et Contaminants dans les Aliments" of the Veterinary School of Nantes — *N°4 – January 2007*

Educational Engineering: A rapidly expanding activity

SARAF is a Continuous Education Programme of the National Veterinary School of Nantes (ENVN) aimed at training executives from developing countries in modern methods of analysis of residues and contaminants in food. This concept was developed in 2001 and is now extremely successful. Entirely conducted in English by the most renowned European experts of the day, the programme consists of theoretical and practical courses alike. It allows participants to acquire up-to-date knowledge about the very latest technologies used in the National Reference Laboratories.

The SARAF programme was held for the seventh time from 11th to 23rd June 2006, at the specific request of the European Commission – DG-Trade. Twenty participants from 17 countries were invited to participate in this training programme; the training courses covered the following topics: regulatory bases of control, methods of risk evaluation, strategy in terms of sample preparation, measurement of samples through physicochemical techniques (essentially mass spectrometry) and, finally, the validation of measurement methods according to analytical decision 2002/657/EC.

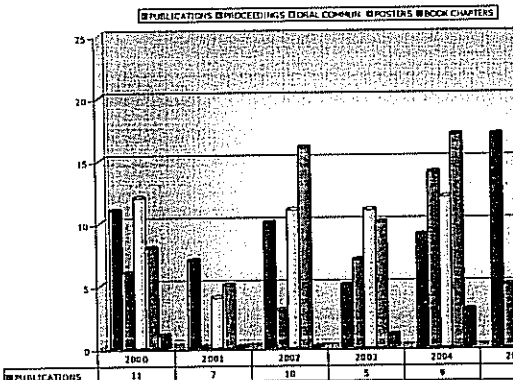
Two new SARAF training sessions will be organised in 2007, one in March (DG-Research) and the other in June (DG-Trade).

POPINRA: A successful project

This research project, initiated in the laboratory in 2005, aimed at determining the occurrence of the main POPs in sea produce. Certain species, through their position in the trophic chain, become a choice target when it comes to evaluating the chemical risk in terms of contaminants. The study of the contamination profiles (relative proportions of the various substances and congeners under screening) gives relevant information as to the sources and types of pollution involved. For each sample, a dioxins, furanes, PCB and other PBDE profile was established. This study generated a lot of data (measurement of 35 parameters in 159 samples) which served as a basis for the evaluation of the risk in the targeted consumers, who were « large » seafood consumers and thus particularly exposed.

The results showed that the most contaminated samples came from the fattest fish and the crustaceans. An increasing POP contamination gradient in the sea produce was observed from the south to the north of France. Whereas a correlation was established between the concentrations in dioxins and PCB, the bromated flame retardants (7 PBDE indicators), did not appear to follow the same rules. The results of this study were presented notably during the world POPs Congress in Oslo (August 2006).

Scientific valorisation: Evolution from 2000 to 2006



	2000	2001	2002	2003	2004	2005	2006
PUBLICATIONS	11	7	10	5	9	17	23
PROCEEDINGS	6	0	3	7	14	5	5
ORAL COMMUN.	12	4	11	11	12	12	15
POSTERS	6	5	16	10	17	5	22
BOOK CHAPTERS	1	0	0	1	1	0	4

JFSM 2006: A Mass Spectrometry Congress in Nantes

The Annual Congress of the Mass Spectrometry and Stable Isotopes French Societies was held from 11 to 14 September 2006 in Nantes Congress Centre. This Congress was organised by a joint Committee led by the LABERCA and including the Universities of Nantes and Angers as well as INRA and IFREMER.

270 participants, originating mainly from France, but also from Belgium, Algeria, Canada, Rumania, United Kingdom or Switzerland attended the Symposium, which dealt with scientific topics such as Identification / quantification of small molecules, Biology and –omic analyses, reactivity and theory, Innovation and new concepts and Isotopic analysis.

Conferences were given by some of the most renowned specialists of Mass Spectrometry and/or its applications. The Congress was greatly appreciated, both for the quality of its scientific conferences and the social interaction between participants.

Latest Publications

- Le Bizec B, Van Hoof N, Courtheyn D, Gaudin I, Van De Wiele M, Bichon E, De Brabander H and André F. Metabolism study of a new anabolic steroid in bovine: preliminary data on 19-norchloroteststerone acetate. J Steroid Biochem Molec Biol 2006;98(1):78-88.
- Antignac J-P., Marchand P., Gadé C., Matayron G., El Mostafa Qannari., Le Bizec B. and André F. Studying variations in the PCDD/PCDF profile across various food products using multivariate statistical analysis. Anal Bioanal Chem, 2006;384(1):271-279.
- Lutz S, Feidt C, Monteau F, Rychen G, Le Bizec B and Jurjanz S. Transfer of Polycyclic Aromatic Hydrocarbons and their major metabolites in milk after chronicle exposure to contaminated soil. Journal of Agriculture and Food Chemistry. 2006;54(1):263.
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- Buisson C, Heberstreit M, Preiss-Weigert A, Heinrich K, Fry H, Flenker U, Banneke S, Prevost S, André F, Schanzer W, Houghton E, and Le Bizec B. Application of stable carbon isotope analysis to the detection of administration of natural hormones to cattle: estrogen administration J Chromatogr A, 2005;1093(1-2):69-80.

Research News

LABERCA

TheNewsletter of the « Laboratoire d'Etude des Résidus et Contaminants dans les Aliments » of the Veterinary School of Nantes N°5 - June 2007

Editorial

The first six months of 2007 have been extremely eventful for the LABERCA. The members of our dynamic team have initiated research work in new areas and set up innovative projects and new partnerships.

I won't say more and leave you to discover this 5th issue of our Research Newsletter...

Have a good read and enjoy the summer.

Bruno LE BIZEC

Important Dates

Evaluation of LABERCA by INRA

1st December 2006: application for the renewal of the status of LABERCA as an INRA Research Unit.
27th February 2007: audit of LABERCA by 3 independent experts.
3rd May 2007: LABERCA's Director met the INRA Commission (ENGREF, Paris).

Evaluation of LABERCA by the DGER

15th December 2006: application for the renewal of the status of LABERCA as « programme support » DGER Unit.
6th September 2007: audit of LABERCA by 2 independent experts.

Evaluation of LABERCA's Quality Management System

23rd and 24th January 2007: audit of LABERCA according to the ISO17025 standard (surveillance audit, flexible type B range).
15th May 2007: Decision of the COFRAC Scientific Committee Meeting to maintain LABERCA's accreditation.

1st June 2007: audit of LABERCA according to standard ISO9001 (surveillance audit).
2nd July 2007: Decision of the MOODY Certification Committee to maintain LABERCA's certification.

In Short

▪ **Continuous education**
The 9th SARAF Training Session is now over and we are actively preparing for the 10th one to take place from 8th to 19th October 2007. Information on http://www.saraf-educ.org.

▪ **They will be defending their PhDs and HDR**
Ms F. Courant on 22/10/07
Mr L. Bailly-Chouriberry on 23/11/07
M. J.P. Antignac will be taking his **HDR** on 05/10/07 (a diploma allowing the candidate to run research work)

ADIPOTOX or the risks of slimming fast

The LABERCA is committed to the ADIPOTOX project, financed by the national agency for research (ANR) in the framework of the national food research programme (PNRA).
This project aims at studying the redistribution and toxicity of Persistent Organic Pollutants (POPs) during severe slimming phases in patients. These slimming phases will constitute a unique model for the study of POPs redistribution in the circulation and the analysis of the genetic and toxic consequences of this redistribution.
POPs include dioxins, PolyChloroBiphenyls (PCBs)*, several organochlorine pesticides, some Polycyclic Aromatic Hydrocarbons (PAH) as well as some brominated flame retardant compounds (PBDE, TBBPA, HBCD), which contaminate human beings, partly via the diet. These lipophilic compounds accumulate predominantly in the adipose tissue, which constitutes a constant "stock" of pollutants suspected to contribute to a chronic toxicity. During severe slimming phases, they could be redistributed brutally in the body, thus exerting their toxicity on exposed organs and tissues. This study will therefore focus on measuring the presence of several POPs in the adipose tissue of severely obese patients as well as the variations of these POPs within a period of three months following the beginning of the slimming protocol. The functional consequences of the redistribution of these POPs will also be evaluated.
ADIPOTOX will be carried out as a partnership between the LABERCA and two INSERM units (UMR S 747 and UMR S 755). The study will follow a clinical protocol led by the nutrition Department of Hôtel-Dieu (Paris).

Contact: marchand@vet-nantes.fr

AISQAL (Integrated Approach of food security and safety)

The research project « AISQAL » was selected following a bid for research projects from the Pays de la Loire Region in 2006. It is being coordinated by Dr. Monique AXELOS, of the « BIA » Unit of INRA in Nantes.

This study aims at providing the local industries with the necessary tools to guarantee the chemical and microbiological safety of food while safeguarding the associated food quality.

This 3-year project brings together eighteen teams from various research institutes (IFREMER, INSERM, CNRS and INRA), higher education establishments (ENV, ENITIAA, AUDENCIA, ESA) and Universities (Nantes, Angers and Laval).
The LABERCA is strongly involved in this project as regards chemical risk evaluation and will be contributing in the following three main areas:

- Evaluation of the consequence of industrial smoking processes on the qualitative and quantitative profile of PAHs;
- Development of a comprehensive dioxin measurement method in fish by studying dioxins binding to the AhR receptor and measuring the induced signal by quantitative PCR;
- Development of an ultra sensitive measurement method dedicated to perfluorinated contaminants (of PFOS- and PFOA-type) in food.

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Research News

LABERCA

TheNewsletter of the « Laboratoire d'Etude des Résidus et Contaminants dans les Aliments » of the Veterinary School of Nantes N°5 - June 2007

Growth hormone and secretagogs: A new skill to be reckoned with

One of the PhDs conducted at LABERCA on the subject of growth hormone and the identification of its fraudulous use on animals will soon be over. Initiated in 2004 in partnership with the French Horseracing Laboratory (Laboratoire des Courses Hippiques), this PhD allowed for the development of two innovative methods for the detection of the fraudulous use of the equine recombinant form of the hormone in the world of horseracing. The first of these methods, dedicated to screening, is an ELISA test which can detect, over a long period of time, the antibodies produced by the animal in response to an administration of the proteic hormone. The confirmation technique consists in an elaborated extraction/purification of the hormone present at trace level (fentomoles) in the animal blood as well as the identification by Liquid Chromatography coupled to Mass Spectrometry on a linear trap of a peptide which is specific to the protein recombinant form. For the first time, this method, validated as per the European Decision 2002/657/EC criteria, enabled to detect the hormone in treated animals. In parallel, work is also being carried out together with a food producing company for the development of a detection method of recombinant bovine growth hormone residues in dairy products.

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The isotopic approach (IRMS): The natural steroids control method now validated

The ISOSTER Project from the 5th PCRD Programme – dedicated to the development of innovative methods for the identification of an illegal use of natural steroid hormones on cattle was recently finalised and the outcome was considered very positive. Tools for measurement and an analytical strategy are now available and their robustness has been proved via interlaboratory assays involving more than ten international laboratories. Today, LABERCA is accredited by the COFRAC on this method according to ISO 17025 standard, type B flexible range.

In parallel, a second project aiming at developing a method for screening cortisol in urine by GC-C-IRMS has been initiated. The strategy is based on the measurement of isotopic deviations of DHEA as ERC on the one hand and 5ß-androstane-3,11,17-trione, which is the oxydation product of cortisol metabolites on the other hand. After intramuscular injection of hydrocortisone on a bovine, a depletion of the targeted metabolites $^{13}C/^{12}C$ was observed several days later. This approach is the first of its kind in this context. Work was published in RCM.

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A new ß-agonists screening method using the transcriptomic approach: towards a drastic change in screening strategies

The LABERCA is developing alternative « indirect high throughput » analytical methods to ensure the salubrity of food of animal origin and, if the case arises, to detect the use of new anabolic substances. The new so-called « indirect » analytical approaches do not aim at identifying the screened molecule as such, but rather the effect that the latter can produce on the organism. The indirect testing would allow for a rapid discrimination between compliant and suspect samples as well as a « high throughput » result. The LABERCA will be coordinating this project with AFSSA Ploufragran as their main partner.

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Water and health: Endocrine disrupters and other emerging substances

The Ministry of Health has asked the national water agencies to set up a control of endocrine disrupters in water destined to consumption. Two water agencies (Seine Normandie and Adour Garonne) are working together with the LABERCA, providing the laboratory with samples taken from sites of their choice. LABERCA has suggested a list of pertinent compounds and will be carrying out the analyses, ensuring analytical performances which allow for the detection of some hundreds picograms per litre. The analysis bears on the screening of natural and synthetic estrogens, androgens and progestagens. The first results have allowed us to reduce down the list of molecules to be screened and to envisage the search for other compounds of endocrine disrupters-type.

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Latest Articles

- H. DE BRABANDER, D. COURTHEYN, J.P. ANTIGNAC, G. PINEL, B. LE BIZEC B.Past and future of mass spectrometry in the analysis of illegal growth promotors for meat production. Invitation Journal of Mass Spectrometry, 2006.

G. PINEL, D. MAUME, Y. DECEUNINCK, F. ANDRE, B. LE BIZEC. Unambiguous identification of thiouracil residue in urine collected in non-treated bovine by tandem and high-resolution mass spectrometry. Rapid communication in Mass Spectrometry (2006) 20,3183-3187.

- G. PINEL, S. MATHIEU, N. CESBRON, D. MAUME, H.F. DE BRABANDER. Evidence that urinary excretion of thiouracil in adult bovine submitted to a cruciferous diet can give erroneous indications of the possible illegal use of thyrostats in meat production. Food Additives and Contaminants. (2006) 23(10):974-980.

- F. COURANT, JP. ANTIGNAC, D. MAUME, F. MONTEAU, AM. ANDERSSON, N. SKAKKEBAEK, F. ANDRE, B. LE BIZEC. Exposure assessment of prepubertal children to steroid endocrine disrupters. Analytical strategy for estrogens measurements in plasma at ultra-trace level. Analytica Chimica Acta. (2007) 586:105-114.

- B. VEYRAND, A. BROSSEAUD, L. SARCHER, V. VARLET, F. MONTEAU, P. MARCHAND, F. ANDRE, B. LE BIZEC. Innovative method for determination of 19 polycyclic aromatic hydrocarbons in food and oil samples using gas chromatography coupled to tandem mass spectrometry based on an isotope dilution approach. Journal of Chromatography A, 1149 (2007) 333-344.

- L. RAMBAUD, F. MONTEAU, Y. DECEUNINCK, E. BICHON, F. ANDRE, B. LE BIZEC. Development and validation of a multi-residue method for the detection of a wide range of anabolic steroids in hair using gas chromatography-tandem mass spectrometry. Analytica Chimica Acta. (2007)586:93-104.

Research News

LABERCA

The Newsletter of the « Laboratoire d'Etude des Résidus et Contaminants dans les Aliments », Veterinary School of Nantes

N°6 – December 2007

Editorial

Our 6th Newsletter not only summarizes some of the projects which we finalised at the end of this year but also introduces the research actions which will be initiated in 2008 in order to follow up on the same topics.
Our objectives remain the same and are perfectly in line with new partnerships and the integration of our team in the 7th FP.
I wish you a pleasant reading and a festive end of year.

Bruno LE BIZEC

SARAF and the European Commission

In 2006, LABERCA answered a call for tender from the European Commission for the organisation of engineering training cycles in eight countries (Ghana, The Philippines, Vietnam, Costa Rica, Papua New Guinea, Thailand, Honduras and Gambia). LABERCA was granted the contracts and contacted the European delegations in the countries concerned in order to define a training programme dedicated to their specific needs.

This programme is now being established and about ten experts in the analysis of residues and contaminants in food will be commissioned to these countries in 2008.

In 2007, LABERCA also won the call for tender from the European Commission for the organisation and implementation of a training programme on the analysis of residues and contaminants in food, dedicated to the reference laboratories of about twenty third country selected by the DG-Trade.

In this respect, a new SARAF Training Session will be organised in June 2008 in the National Veterinary School of Nantes.

In Short

- **Retirement**

Mr Daniel Maume will be retiring on 31 December 2007 after working for 17 years for LABERCA.

We wish him a long and happy retirement, which will no doubt be very active in the pursuit of his favorite pastimes.

Exposure characterisation of the prepubertal children to gonadal steroid hormones (PhD defended by Frédérique COURANT on 22/10/2007)

Global concerns have been raised in recent years over the potential adverse effects that may result from exposure to chemicals that have the capacity to interfere with the endocrine system. Our main purpose was to investigate to what extent food intake of steroid hormones can represent a risk for prepubertal children.

At first, a new analytical strategy for the measurement of steroid hormones at ultra-trace level in food and complex biological matrices was developed. Subsequently, steroid measurements in various food products (milk, egg and meat) were performed in order to evaluate the food intake in steroid hormones for this population.

Then, the quantification of the main androgens and estrogens in more than 120 serum samples from children aged 6-16 years gave access to the steroid plasmatic hormonal rates and consequently to the daily endogenous production of this population.

A first interpretation of these data in terms of risk assessment is also provided, in connection with existing CODEX, JECFA and FDA recommendations regarding the maximal acceptable daily intake for estradiol.

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DEER or the risks of endocrine disruption induced by certain residues and contaminants in Man

In 2008, the LABERCA will become part of a European Research Project from the 7th FP entitled « Developmental effects of environment on reproductive health » (DEER).

This project, coordinated by the University of Turku (Pf. J. Topari) gathers 9 partners coming from the most advanced teams on the subject (Pf. N. Skakkebaeck, Pf. R. Sharpe, Pf. S. Swan, Pf. B. Jegou, ...). Its general objective is to study the potential links between a chemical exposure at various early development stages (early puberty, infertility, « testicular dysgenesis syndrome ») and the LABERCA will be in charge of a work package whose task will be to develop and apply metabolomic approaches in order to characterise in a global manner the samples taken in the various population groups concerned, then to identify several relevant biomarkers for the pathologies studied.

The LABERCA will also take part in the characterisation of the contents of the various chemical residues and contaminants present in the biological samples of these populations.

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Research News

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N°6 – December 2007

Detection of recombinant equine growth hormone administration: antidoping application in horseraces

(PhD defended on 23 November 2007 by Ludovic BAILLY-CHOURIBERRY)

The growth hormone is a peptidic molecule with anabolic activity potentially used to improve horse athlectic efficiency and to increase the production profitability of farm animals. However, the use of this molecule is strictly forbidden by the races code and the European directives in force.

The control of its potential use constitutes an analytical challenge which many laboratories have attempted to tackle. The difficulty of this analysis lies mainly in the fact that the hormone of interest is present at trace level (µg. L^{-1},fmol.L^{-1}) in complex biological matrices such as blood, with a short half-life estimated to a few hours.

In this context, this PhD thesis work enabled the development of two innovative methods, one direct and one indirect, for the detection of recombinant equine growth hormone (reGH) illegal use in horses. A protocol of extraction/purification of the hormone in plasma leading to an enzyme digestion and the detection by LC-MS/MS of a specific peptide has enabled for the first time to reveal growth hormone in samples from animals treated with a preparation of hormones and to build a kinetic of elimination over 48 hours.
As for the indirect detection method, it was developed by ELISA to detect the production of anti-reGH antibodies consecutive to a treatment with reGH up to 5 months after treatment. This method thus allows to envisage immediate applications in the field of antidoping in horseraces and more generally in farm animals.

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Growth hormone and secretagogues: ... Continued

The regulations forbidding this moledule in the world of horseracing have now been very recently followed by sensitive and specific screening and confirmatory analytical methods. Nevertheless, the performance of the methods is limited by the short half-life span of this type of proteic substance, which does not allow an optimal detection in time of the fraudulent use of these molecules. It now seems obvious that a more global approach has to be implemented. Facing this very same problem and after several years of collaboration on the subject, the Laboratoire des Courses Hippiques (LCH) and the LABERCA have launched a new research work together by the means of a co-financed PhD. This innovative strategy will allow to set and compare the urine and blood biological prints of two groups of animals, one treated with equine recombining growth hormone and the other consisting of control animals. The setting up of such a study requires the use of specific and adequate tools such as high performance Liquid Chromatography coupled to High Resolution Mass Spectrometry on a brand new generation hybrid instrument (LTQ-Orbitrap).

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NUPEM

Starting in 2008, the LABERCA will participate in a regional research project coordinated by the Centre Régional en Nutrition Humaine (CRNH), whose subject is perinatal nutrition and the notion of metabolite profiling. The laboratory will be mostly involved in the development and application of general solutions of metabolomic-type aimed at characterising the potential impact of certain food constituants (polyinsaturated fatty acids, oligosaccharides, proteins and chemical pollutants) on newborns.

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Latest Publications

- G. PINEL, L. RAMBAUD, G. CACCIATORE, A. BERGWEFF, C. ELLIOTT, M. NIELEN and B. LE BIZEC. Elimination kinetic of 17ß-estradiol 3-benzoate and 17ß-nandrolone laureate esters metabolites in calves urine. Journal of Steroid Biochemistry and Molecular Biology 2008. Under press.
- E. BICHON, F. KIEKEN, N. CESBRON, F. MONTEAU, S. PREVOST, F. ANDRE and B. LE BIZEC. Developpment and application of stable carbon isotope analysis to the detection of cortisol administration in cattle. Rapid communications in Mass Spectrometry 2007;21:2613-2620.
- J.P. ANTIGNAC, R. CARIOU, D. MAUME, P. MARCHAND, F. MONTEAU, D. ZALKO, A. BERREBI, J.P. CRAVEDI, F. ANDRE and B. LE BIZEC. Exposure assessment of foetus and newborn to brominated flame retardants in France: preliminary date. Molecular Nutrition and Food Research 2008. Under press.
- L. BAILLY-CHOURIBERRY, E. CHU-VAN, G. PINEL, P. GARCIA, M.A. POPOT, G. ANDRE-FONTAINE, Y. BONNAIRE and B. LE BIZEC. Detection of a secondary biomarker of met-eGH as a strategy to screen for somatotropin misuse in horseracing. The Analyst 2008; 133: 270-276.
- H. NOPPE, B., B. LE BIZEC, K. VERHEYDEN and H. DE BRABANDER. Review: novel analytical methods for the determination of steroid hormones in eddible matrices. Journal of Mass Spectrometry, 2008. Under press.

HDR

Dr. Jean-Philippe ANTIGNAC has passed his « Habilitation à Diriger des Recherches » examination on 5th October 2007. Jean-Philippe has been working in LABERCA since 1998 and his research activities deal mainly with chemical food safety. Since 2003, the main focus of his work has been research topics aimed at the study of some specific chemical pollutants (endocrine disruptors), on the angle of their potentially adverse effects on man and his descendants, especially by developing new global approaches such as Mass-Spectrometry based metabolomics.

Research News

LABERCA

The newsletter of the Laboratoire d'Etude des Résidus et Contaminants dans les Aliments from the Veterinary School of Nantes

N°7 - January 2009

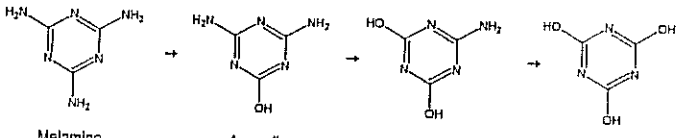
Edito

The end of the year 2008 has been particularly rich for LABERCA, first as National Reference Laboratory for food control through several sensitive dossiers (melamine in food product imported from South-East of Asia or persistent contaminants in river fishes), then as research unit (more than 20 articles published and/or in press, first valorisations in the field of metabolomics), and finally in terms of European funding (projects DEER and UNIQUECHECK, FP7) or knowledge dissemination (one SARAF extra session for DG research). Enjoy your reading.

Pf. Bruno Le Bizec

The melamine crisis

Following the human health problems reported from South-East of Asia in September 2008, in relation with the presence of melamine in food products such as milk and derivatives, the LABERCA was mandated by the French Ministry of Agriculture as referent structure in charge of organising the control of this chemical pollutant and its related metabolites (ammeline, ammelide, cyanuric acid).



Melamine — Ammeline — Ammelide — Cyanuric acid

The laboratory has developed and validated an appropriate analytical method for these compounds by gas chromatography coupled to tandem mass spectrometry (GC-MS/MS), disseminated on our website (http://www.laberca.org), and to several application laboratories in France for performing screening analyses, the confirmatory analyses being be performed by the LABERCA.

In Brief

▪ SARAF Certification

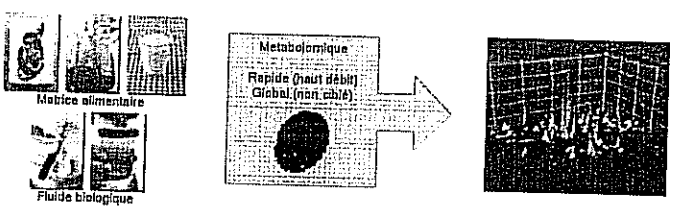
The continuous education stucture School for Advanced Residue Analysis in Food (SARAF) is now certified according to the ISO 9001 : 2000 standard.

▪ Visit of a Minister

In the frame of a visit to the national veterinary school of Nantes on 23th October 2008, the French Minister of Agriculture and Fishery Michel Barnier visited the LABERCA, a good opportunity to bring up the melamine crisis...

METABOLOMICS : a novel screening tool for illegal growth promoters in cattle

The development of metabolomic approaches based on liquid chromatography coupled to high resolution mass spectrometry is one research area of the laboratory which is supported by significant human and financial resources, and conducted through various research projects at regional, national, and European levels. Initiated since early 2007, and after a central phase consisting in establishing appropriate and necessary analytical procedures, these different studies today generate their first concrete results demonstrating the usefulness of this innovative approach for revealing some biomarkers signing an exposure to one or several xenobiotics.



The first valorisations of these works, related to the identification of illegal exposure to steroid hormones, corticosteroids, beta-agonists or growth hormones, are under publication.

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UNIQUE-CHECK : a multiple approaches project dedicated to the control of growth hormone illegal use and staff mobility

Marie Curie actions are a particularly popular and appreciated component among European research and development framework programs. Their initial orientation (simple financial support for mobility) progressively shifted towards a more complete tool for promoting researcher careers. In the frame of the FP7, Marie Curie actions have been grouped under the specific "people" program, the objective of which is to make Europe more attractive for the best scientists. Among the 5 defined thematics, the LABERCA with some of its European partners applied to the IAPP call to submit a collaborative project related to the growth hormone. One aim of this project will be to propose efficient and complementary tools and strategies for controlling this compound in cattle in general and in lactating cows in particular. This project was retained for funding, and is planned for 4 years from mid-2009. The second major objective will be to stimulate people mobility across several European laboratories in the field, and to promote the sharing of scientific knowledge generated within the project.

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Research News

LABERCA

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N°7 - January 2009

Perfluorinated contaminants : a PhD thesis for the study of these emerging chemical pollutants

Perfluorinated compounds (PFC), e.g. PFOS or PFOA, represent a class of emerging persistent organic pollutants (POPs). These synthetic molecules, characterised by anti-adhesive properties, are present in many manufactured products as well as in the environment and represent a potential risk for human health. The global purpose of this new PhD thesis initiated in September 2008 will be to develop appropriate analytical tools for measuring these compounds at trace level in complex biological matrices. A first objective will be to generate some data regarding their occurrence in selected food products, and a second objective will be to collect some information leading to a better understanding the contamination and transfer mechanisms in the scope of identifying some solutions for controlling and/or managing this risk.

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Natural hormones : a PhD thesis to develop new screening strategies in bovines

The endogenous presence of the main natural hormones (estradiol, testosterone, progestérone) in biological fluids and tissues make the compliant / non compliant decision difficult for the analysed samples. Indeed, the extremely wide variability observed in terms of concentration levels does not allow to determine some reference thresholds for these parameters. The global purpose of this new PhD thesis initiated in September 2008 will be to investigate and evaluate 3 complementary approaches expected to lead to one or more screening criteria permitting to reveal an abuse of natural hormones in bovine: a statistical analysis of quantitative data related to the main hormone and phase I metabolites monitored in the considered samples, and 2 metabolomic approaches (targeted and untargeted).

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ECDs Workshop : a new SARAF session dedicated to endocrine disruptors

A workshop dedicated to the "Analysis of estrogenic endocrine disruptors in food: state-of-the-art and future trends from specific spectrometric approaches to emerging global transcriptomic approaches" was organised by the LABERCA from 9th to 11th December 2008, through the continuous education structure School for Advanced Residue Analysis in Food. The main objective of this workshop was to disseminate and demonstrate to the scientific community the various developments and findings gathered on this subject within the BIOCOP integrated project (http://www.biocop.org) conducted in the frame of the 6th FP and coordinated by Pf. Chris Elliott (Queen's University, Belfast). This event, also in connexion with the CASCADE network of excellence (http://www.cascadenet.org), gathered 16 participants from France, Belgium, The Netherlands, Northern Ireland, Italy, Hungary and Sweden. Internationally renowned speakers from the most recognised and reference experts in the field such as Pr. Niels Skakkebaek (Rigshospitalet, Copenhagen, Denmark), Dr. Bernard Jegou (INSERM, Rennes, France) or Dr. Alessandra Roncaglioni (Mario Negri Institute, Milano, Italy) have first introduced the complex clinical, biological, and mechanistic aspects of this thematic, respectively. Major scientists involved in the BIOCOP project were then invited to present their own methodological developments including targeted mass spectrometric measurements for steroid hormones (Bruno Le Bizec, Frédérique Courant) or phytoestrogens (Jean-Philippe Antignac), global transcriptomic profiling applied to mycotoxins or phytoestrogens (Pf. Hanspeter Naegeli, Dr. Hans Gmuender, Dr. Katerina Lancova), and finally some preliminary results in the field of metabolomics applied to the investigation of metabolic disorders induced in animal (Dr. Gaud Pinel) or human (Dr. Frédérique Courant) consecutively to a chemical exposure. Discussions were also organised so as to encourage exchange and networking within the group.

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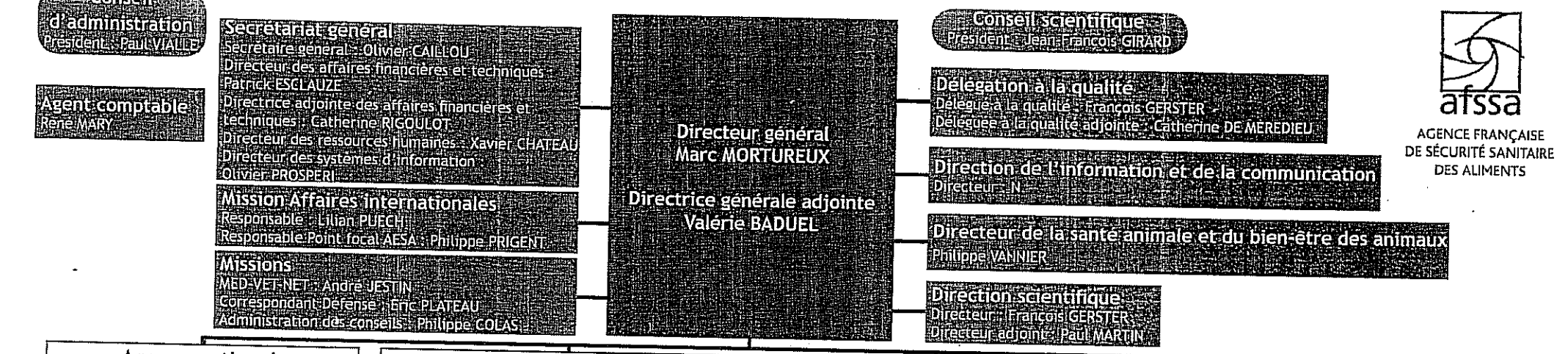
Last Publications

- Veyrand B., Venisseau A., Marchand P., Antignac JP. And Le Bizec B. Determination of toxaphene specific congeners in fish liver oil and feedingstuff using gas chromatography coupled to high resolution mass spectrometry. Journal of Chromatography B 2008;865:121-126.
- Courant F, Antignac J-P, Laille J, Monteau F, André F and Le Bizec B. Exposure assessment of prepubertal children to steroid endocrine disruptors. 2. Determination of steroid hormones in milk, egg and meat samples. Journal of Agricultural and Food Chemistry 2008;56(9):3176-3184.
- Bichon E, Richard CA and Le Bizec B. Development and validation of a method for fipronil residue determination in ovine plasma using 96 well-plate solid-phase extraction and gas chromatography-tandem mass spectrometry. Journal of Chromatography A 2008;1201:91-99.
- Antignac J-P, Cariou R, Zalko D, Berrebi A, Cravedi J-P, Maume D, Marchand P, Monteau F, Riu A, André F and Le Bizec B. Exposure assessment of French women and their newborn to brominated flame retardants. Determination of tri- to deca-bromodiphenylethers in maternal adipose tissue, serum, breast milk and cord serum. Environmental Pollution 2009;157(1):164-173.
- Le Breton MH, Rochereau-Roulet S, Pinel G, Bailly-Chouriberry L, Rychen G, Jurjanz S, Goldmann T and Le Bizec B. Direct determination of recombinant bovine somatotropin in plasma from a treated goat. Rapid Communication in Mass Spectrometry 2008;22:3130-3136.
- Destrez B, Pinel G, Monteau F, Lafont R and Le Bizec B. Detection and identification of 20-hydroxyecdysone metabolites in calf urine by LC- HRMSn measurements and establishment of their kinetics of elimination after 20E administration. Analytica Chimica Acta 2008, Sous Presse.
- Duffy E, Rambaud L, Le Bizec B and O'Keeffe M. Determination of hormonal growth promoters in bovine hair: comparison of LC-MS/MS and GC-MS/MS methods. Analytica Chimica Acta 2008, Sous presse.
- Bailly-Chouriberry L, Pinel G, Garcia P, Popot M.-A, Bonnaire Y, Le Bizec B. Identification of recombinant equine growth hormone in equine plasma by LC-MS/MS measurements as a confirmatory method for doping control. Analytical Chemistry 2008;80:8340-8347.



Agence nationale du médicament vétérinaire

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Directeur adjoint : Dominique MOUROT

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  - Unité Gestion et évaluation
  - Centre de pharmacovigilance vétérinaire de Lyon
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  - Unité Centre d'information sur la qualité des aliments (CIQUAL)
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  - Unité d'évaluation des risques biologiques
  - Unité d'évaluation des risques liés à l'alimentation et à la santé animales
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- **Unité d'évaluation sur la nutrition et les risques nutritionnels**
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  - Laboratoire d'études et de recherches sur les produits de la pêche - *Directeur : Pierre MALLE*
- **Site : Dozulé**
  - Laboratoire d'études et de recherches en pathologie équine
  Directrice : Claire LAUGIER
- **Site : Fougères**
  - Laboratoire d'études et de recherches sur les médicaments vétérinaires et les désinfectants - *Directeur : Pascal SANDERS*
- **Site : Lyon**
  - Laboratoire d'études et de recherches en pathologie bovine et hygiène des viandes - *Directeur : Didier CALAVAS*
  Directeur adjoint : Jean-Yves MADEC
- **Site : Maisons-Alfort**
  - Laboratoire d'études et de recherches en pathologie animale et zoonoses - *Directeur : Pascal BOIREAU*
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- **Site : Sophia-Antipolis**
  - Laboratoire d'études et de recherches sur les ruminants et les abeilles - *Directeur : Richard THIÉRY*

Organigramme des entités de l'Agence
novembre 2009

FICHE AUTO CONTROLE DE TEMPERATURE – VITRINES REFRIGEREES

RELEVE N° 1 - MATIN

MOIS DE :

RESPONSABLES DU RELEVE :

ENCEINTE REFRIGEREE N° :

HEURE DE DEGIVRAGE CONNUE :

NOM DU SITE : POISSONNERIE DOMAIN ET FILS

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Pour toute question complémentaire contactez HA 13 CONSEILS 115, rue de l'Abbé Groult 75015 - PARIS

Tél. : 01 48 28 33 40 - Email. : ha13@free.fr

RELEVE N° 2 - SOIR

MOIS DE :

RESPONSABLES DU RELEVE :

ENCEINTE REFRIGEREE N° :

HEURE DE DEGIVRAGE CONNUE :

NOM DU SITE : POISSONNERIE DOMAIN ET FILS

+ 12 ° C																													
+ 11 ° C																													
+ 10 ° C																													
+ 9 ° C																													
+ 8 ° C											INTERVENTION SERVICE ENTRETIEN																		
+ 7 ° C																													
+ 6 ° C																													
+ 5 ° C											TEMPERATURE MAXIMALE AUTORISEE																		
+ 4 ° C																													
+ 3 ° C																													
+ 2 ° C																													
+ 1 ° C																													
Jour du mois	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31

Les produits **sous signe officiel de la qualité et de l'origine** obéissent à des cahiers des charges précis, validés par les pouvoirs publics

Soumis à des contrôles rigoureux par des organismes indépendants, accrédités et agréés par l'Etat, ces produits sont le fruit de démarches de filières exigeantes, animées par des professionnels / partenaires impliqués, puisque engagés volontairement dans ces démarches de qualité.
Les produits sous signe officiel de la qualité et de l'origine couvrent de nombreux secteurs alimentaires.
Concernant les produits carnés, on compte à ce jour en France :

- **2 990 éleveurs et 100 entreprises d'abattage et/ou transformation enregistrés en Agriculture biologique ;**
- **33 500 éleveurs et 280 entreprises d'abattage et/ou transformation en Label rouge ;**
- **11 000 éleveurs et 68 entreprises d'abattage et/ou transformation répertoriés en démarche IGP ;**
- **400 éleveurs et 18 entreprises d'abattage et/ou transformation en démarche AOC.**

Un dispositif **encadré par les pouvoirs publics**

- **Ministère de l'Agriculture et de la Pêche:**
Il définit la politique en matière de qualité alimentaire et encadre en particulier le dispositif des signes d'identification de la qualité et de l'origine. Il assure la tutelle de l'INAO.
- **INAO** (Institut National de l'Origine et de la qualité) :
Il instruit les demandes de reconnaissances des AOC, IGP, STG et Label rouge. Il assure le suivi des règles relatives à l'Agriculture biologique et supervise les contrôles sur l'ensemble de ces signes.

Les viandes de **bœuf, veau porc, agneau** et leurs **produits tripiers** : quels **repères** pour bien **choisir** ?

Conseil Marketing - RCS Paris B 350 076 105 - Paris 09 - Création CEMOREVEUX - FEVRIER - 0908

Label Rouge · AB Agriculture Biologique · Agriculture Biologique · Appellation d'origine contrôlée AOC France · Appellation d'Origine Protégée · Indication Géographique Protégée · Spécialité Traditionnelle Garantie

Pour en savoir plus :

www.civ-viande.org Le Centre d'Information des Viandes est une association loi 1901.
Plate-forme d'échanges et d'information, le CIV réunit les professionnels de la filière viande, les pouvoirs publics, des scientifiques et des représentants d'associations.
Sa mission ? Contribuer à une meilleure connaissance, pour tous les publics, des viandes bovine, ovine, chevaline, porcine, des produits tripiers et de leurs filières.

www.label-viande.com
www.agriculture.gouv.fr
www.inao.gouv.fr
www.produitslaitiersetviandebio.com

CIV Centre d'Information des Viandes
64 rue Taitbout • 75009 Paris
www.civ-viande.org

Les signes officiels de la qualité et de l'origine, les seuls complètement encadrés par les pouvoirs publics.

CIV Centre d'Information des Viandes

Certains repères ont été instaurés pour aider le consommateur à faire son choix parmi les [illegible] de bœuf, de veau, de porc et d'agneau : ce sont les **signes d'identification de la qualité et de l'origine**. Au-delà de la qualité sanitaire ou de la traçabilité, valables pour toutes les viandes, ils attestent de caractéristiques spécifiques.

Français ou européens, ces signes sont encadrés par les pouvoirs publics et ont été mis en place autour de trois critères :

- une **qualité gustative supérieure**
- ou une **qualité de mode de production respectueux de l'environnement et du bien-être animal**
- ou une **qualité liée à l'origine**

Ces signes sont identifiés par des logos.

Qualité gustative supérieure

Label rouge

La vocation d'un produit Label rouge est de garantir un goût et des saveurs de qualité supérieure. Cette qualité gustative est déterminée par des analyses sensorielles effectuées chaque année auprès des consommateurs. Pour les espèces élevées en Label rouge, dans le respect de pratiques d'élevage traditionnelles, les critères du cahier des charges portent, entre autres, sur la sélection des animaux, sur leur alimentation et leur bien-être.

Propriété du ministère de l'Agriculture, le Label rouge est une démarche de filière associant les partenaires, de l'éleveur au distributeur dans le respect d'un cahier des charges strict. Cette démarche fait l'objet de contrôles complémentaires par des organismes certificateurs indépendants agréés par les pouvoirs publics.

Qualité attachée à un mode de production respectueux de l'environnement et du bien-être animal

Agriculture biologique

L'Agriculture biologique constitue un ensemble de pratiques agricoles respectueuses de l'environnement. Elle se distingue par son mode de production fondé sur la non-utilisation de produits chimiques de synthèse, le recyclage des matières organiques, la rotation des cultures et la lutte biologique.

Les produits d'origine biologique sont identifiés par le logo AB, propriété du ministère de l'Agriculture. Pour être commercialisé comme issu de l'Agriculture biologique, le produit doit avoir été contrôlé et certifié par un organisme certificateur indépendant agréé par les pouvoirs publics.

◀ Ce logo est l'équivalent européen du signe de qualité AB en France.

Qualité liée à l'origine

Appellation d'Origine Contrôlée (AOC)

Cette mention implique un lien étroit entre le produit, le terroir et le savoir-faire de l'homme. Le produit doit provenir d'une aire de production délimitée et répondre à un cahier des charges qui fait l'objet d'une procédure d'agrément conduisant à une reconnaissance officielle.

Appellation d'Origine Protégée (AOP)

Ce logo est l'équivalent européen du signe de qualité AOC en France.

Indication Géographique Protégée (IGP)

Cette mention, à l'échelle européenne, se traduit par une relation suffisante entre le produit et son origine pour lui conférer une caractéristique ou une réputation. Pour cela, seules certaines phases de production, transformation et élaboration doivent avoir lieu à l'intérieur de l'aire géographique délimitée.

Spécialité Traditionnelle Garantie (STG)

Cette mention, au niveau européen, ne fait pas référence à une origine géographique, mais aux caractéristiques traditionnelles du produit par sa composition ou son mode de production. Cette mention permet de protéger un nom caractérisant une recette ou un savoir-faire traditionnel.