

GROUNDWATER QUALITY MONITORING IN SPAIN

WATER QUALITY MONITORING IN SPAIN
Centro de Estudios Hidrográficos
CEDEX

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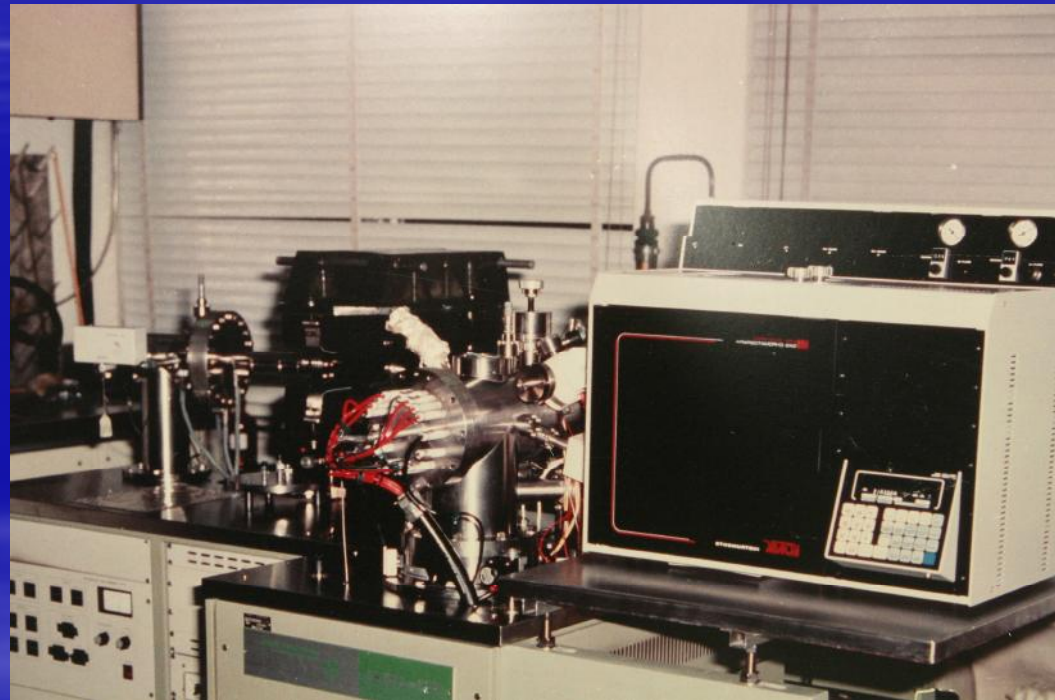


Background

- The Laboratory of Water Quality of the CHS (CEDEX), since its inception in the mid 70's, has been conducting studies of Quality and Pollution in water and other matrices related to the Water Environment (sludge, soil, sediments).
- For these studies advanced analytical techniques have been required, such as gas chromatography with different detectors and gas chromatography coupled with high resolution mass spectrometry (HRGC / HRMS).

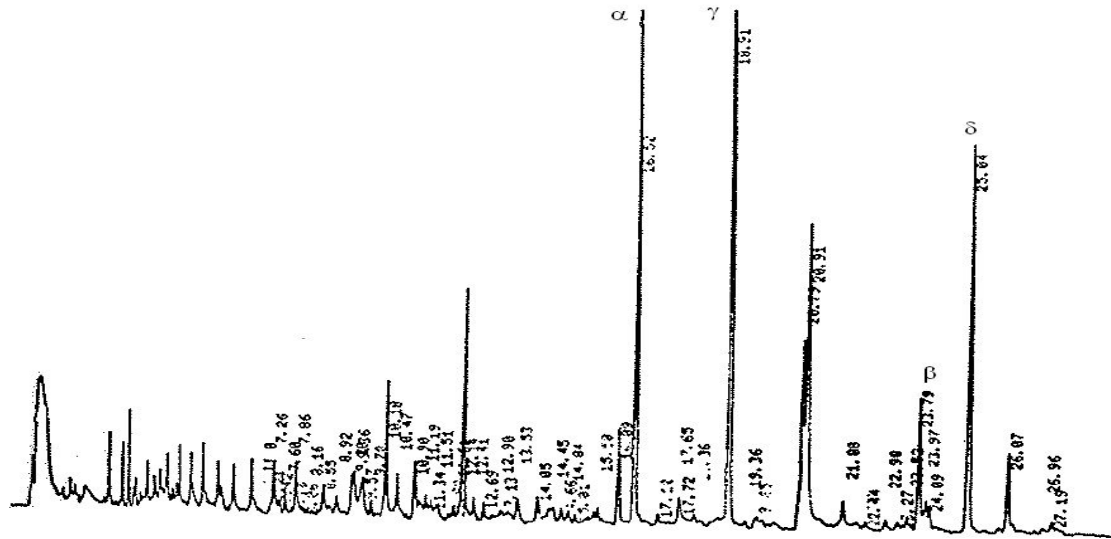
Background

- The **VG 70 – 250**, a high resolution mass spectrometer resolution was, with the CSIC of Barcelona, one of the first spectrometer in Spain dedicated to the study of organic pollutants in water.



This spectrophotometer, along with a gas chromatograph (Konik), was used to perform **specific studies** of pollution .

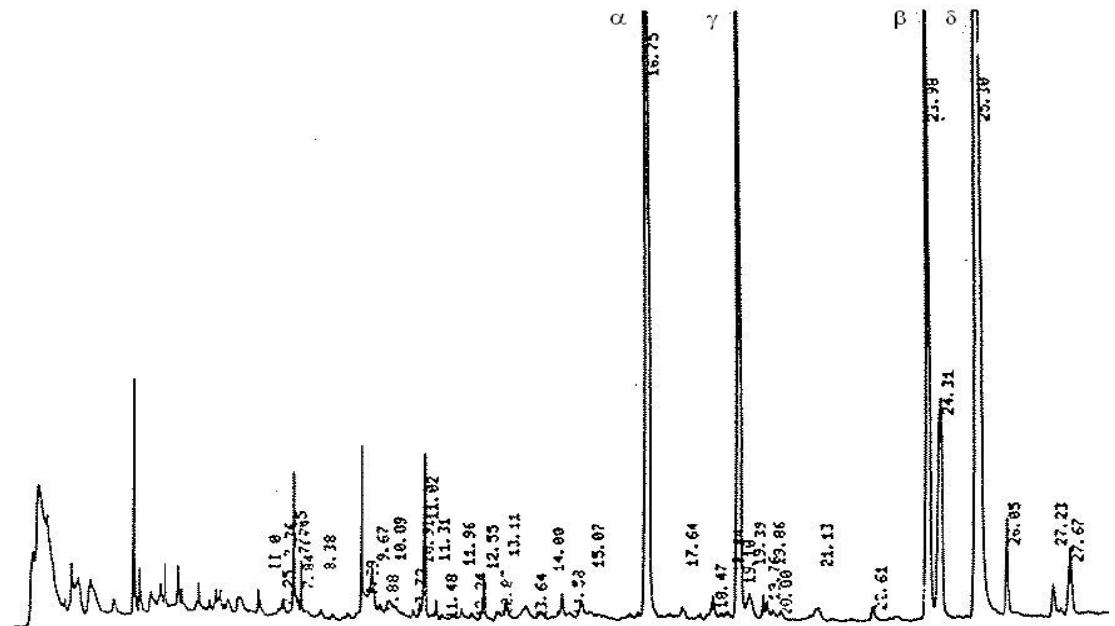
Rio Gállego Punto 8



...d
...es
...out in processes of
... Spain, such as:
...in of Rio **Gállego** in 1987,
...**nexane**.
... (carbamates) ...

- Studies of pesticide residues in **Palmerillas** (Almería)
- Pollution by ship fractions of samples seawater.
- Studies related to the

Vertido L. 3



Background

Specific studies: D. 76/464/CEE

- *"Progress in the study of dangerous substances as defined by the EEC":*
 - Made between 1988 and 1989 and requested by the DGOPH of the MOPU. It studied the presence of substances in **List I** and referred to in **List II**, belonging to **Directive 76/464/EEC**, from which emission limit values and environmental quality standards for various substances were established. This directive was transposed into Spanish legislation through various laws, Royal Decrees, etc.
 - The guidelines were derived from this first set out in **Annex IX of the WFD.**
 - Is carried out on samples taken in surface water and for various projects as the "Intensive management of the quality of reservoir water for supply to population in the river basin of Guadiana" (1993-1994).

Background

Specific studies

- *From the 80s studies on **groundwater** were initiated .*
 - Issue: since it is a hidden resource, for a long time attention was not focused on it, so there was no information and few studies were performed. The laboratory has always been committed to the study of such waters .
 - Specific studies were begun implementing policies that were subsequently listed in **Annexes VIII and IX of the WFD**
 - *"Evolution of the content of heavy metals, nutrients and organic micropollutants in groundwater Castellón de la Plana in collaboration with the Geological Survey" (1980-1991).*
 - *"Contamination of groundwater in the province of Valencia and its supply to urban population " .*
 - *"Organic micropollutants in the water of the hydrogeological units 04.04 West Mancha and 04.06 Campo de Montiel" .*

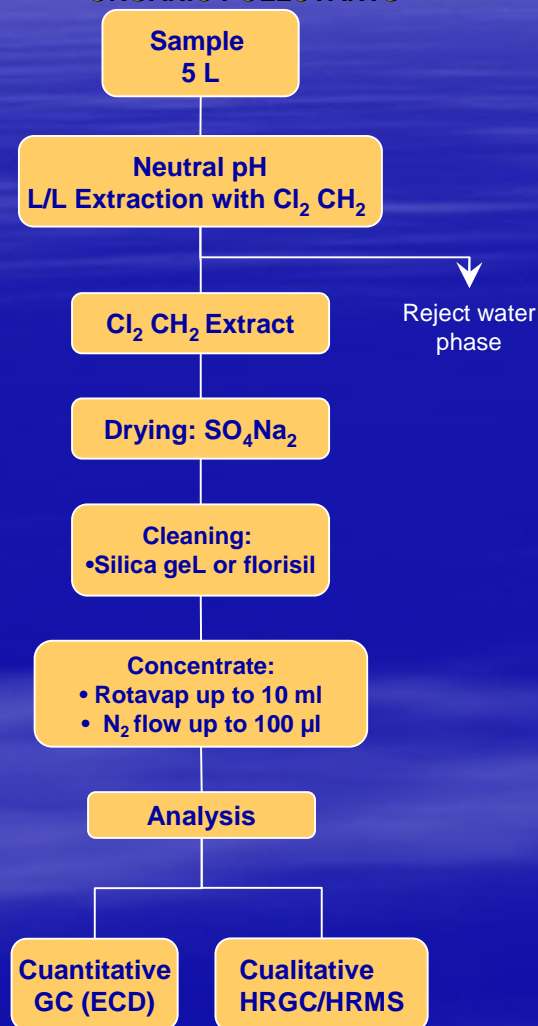
Background

General studies

- There have been **general studies** on groundwater, prior to **WFD** and since 1991 to date, through management agreements with DGOHCA, current DGA, whose resulting data were sent to the Ministry.
- All data that the laboratory has from the 90's are being introduced in the **HIDRO** database of the Centre for Hydrographic Studies .
- Data from these studies have also been taken into account to carry out the **design** of quality control **networks**.
- **Analytical** and **protocols** used:

General studies (1987 – 2000)

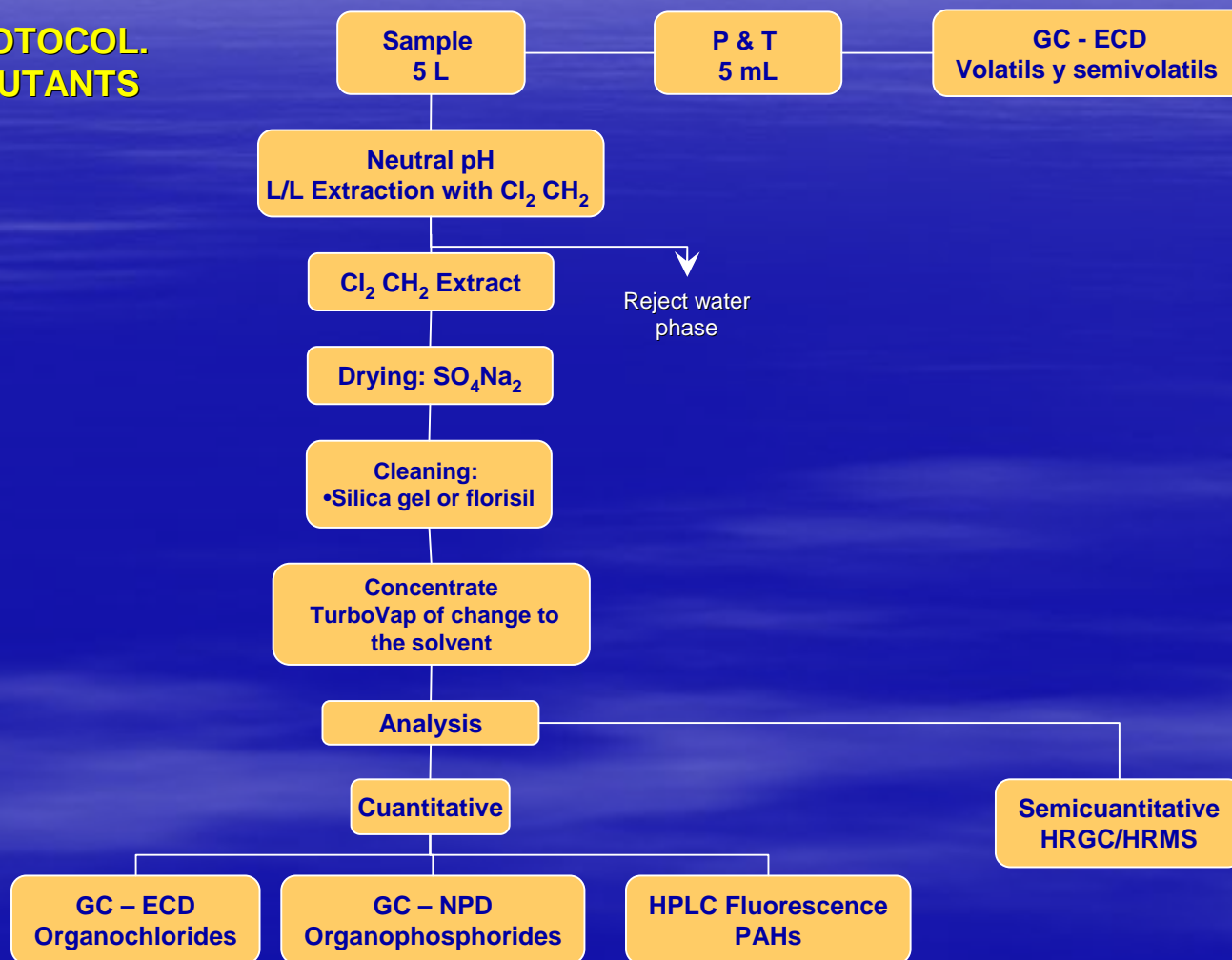
ANALYTICAL PROTOCOL . ORGANIC POLLUTANTS



In situ	Principal ions	Additional parameters	Metals
pH Conductivity Temperature	Chlorides Sulphates Carbonates Bicarbonates Calcium Magnesium Sodium Potassium Nitrates	Total phosphorus Kjeldhal Nitrogen	Cadmium Copper Iron Chromium Manganese Lead Mercury Arsenic Boron
Pollutants organic			
<ul style="list-style-type: none"> ▪ Semicuantitative screening (MS) - EPA 625 Method ▪ Compounds with EC directive: <ul style="list-style-type: none"> <li style="width: 50%;">– Carbon tetrachloride <li style="width: 50%;">– Chloroform <li style="width: 50%;">– Hexachlorocyclohexane <li style="width: 50%;">– Hexachlorobenzene <li style="width: 50%;">– DDT, DDD and DDE <li style="width: 50%;">– Hexachlorobutadiene <li style="width: 50%;">– Pentachlorophenol <li style="width: 50%;">– 1,2-Dichloroethane <li style="width: 50%;">– Aldrin <li style="width: 50%;">– Trichlorobenzenes <li style="width: 50%;">– Dieldrin <li style="width: 50%;">– Trichloroethylene <li style="width: 50%;">– Endrin <li style="width: 50%;">– Tetrachloroethylene <li style="width: 50%;">– Isodrin 			

General studies (1987 – 2000)

ANALITICAL PROTOCOL. ORGANIC POLLUTANTS



General studies (1987 – 2000)

In situ	Principal Ions	Additional parameters	Metals
pH Conductivity Temperature	Clorides Sulphates Carbonates Bicarbonates Calcium Magnesium Sodium Potassium	total phosphorus Kjeldhal Nitrogen Suspended Solids Nitrates Ammonium Fluorides COD	Cadmium Copper Iron Chrome Manganese Lead Mercury Arsenic Boron Zinc Nickel

Organico Pollutants

▪ Semiquantitative screening (MS) - EPA 625 Method

▪ Volatile and semivolatile: (P&T/GC-ECD) (EPA 624)

- Chloroform
- Carbon Tetrachloride
- 1,2- dichloroethane
- Trichloroethylene
- 1,3,5 – Trichlorobenzene
- 1,2,4 – Trichlorobenzene
- 1,2,3 – Trichlorobenzene
- Hexachlorobutadiene

▪ OCs pesticides: (GC-ECD) (EPA 608/8080)

- Hexachlorobenzene
- α -hexachlorocyclohexane
- β - hexachlorocyclohexane
- γ - hexachlorocyclohexane
- λ - hexachlorocyclohexane
- Heptachlor epoxide
- Heptachlor
- Aldrin
- Dieldrin
- Endrin
- Endrin aldehyde
- Endosulfan sulphate
- Endosulfan I
- Endosulfan II
- op´DDE
- pp´DDE
- op´DDD
- pp´DDD
- op´DDT
- pp´DDT

▪ Organophosphate pesticides : (GC-NPD) (EPA 507/8140/8141)

- Demeton – S
- Diazinon
- Disulfoton
- Methyl parathion
- Parathion
- Malathion
- Ethion
- Azinphos methyl

▪ Polycyclic aromatic hydrocarbons : (HPLC – Fluorescence)

- Fluorantheno
- Benzo [a] pyrene
- Benzo [b] fluoranthene
- Benzo [k] fluoranthene
- Benzo [g,h,i] perylene
- Indeno [1,2,3-c,d] pyrene

General studies



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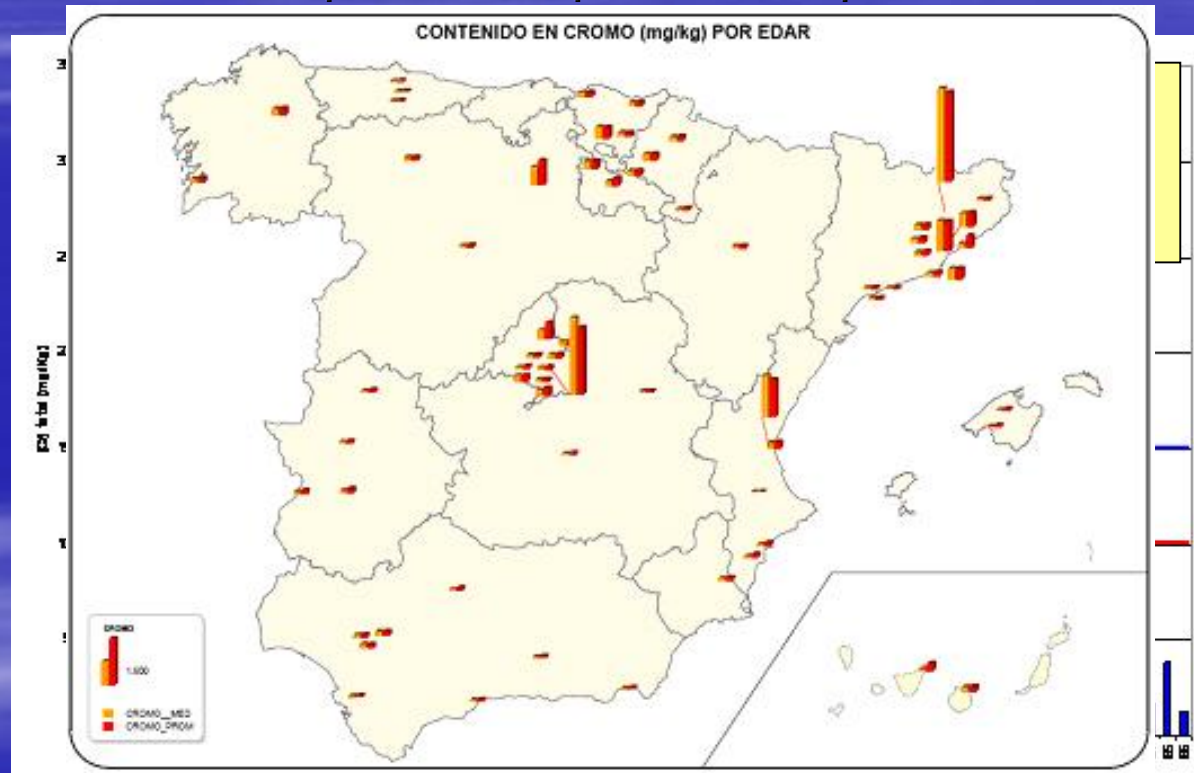
Other studies

- Years 2004-2006 - **Programme of characterization of sewage sludge generated in Spain** - Study of the composition of sludge from wastewater treatment plants in Spain. Parameters:

		Agronómics Parameters	
		Analytical Method	
Organics	Metals		
		pH	
		Conductivity	
		Total carbon	
		Oxydizable organic matter	
		Nitrogen (total)	
AOX		Microvolumetric analysis (discontinuous method) DIN 38409- H14 (1985) and UNE-EN 1485 (1977)	
	Cd	Microwave extraction and analysis by optical ICP	
	Cr	Microwave extraction and analysis by optical ICP	
LAS	Ni	HPLC/FID (horizontal project) by optical ICP	
		Microwave extraction and analysis by optical ICP	
		P (assimilable)	
DEHP	Hg	GC/MS (NERI (2003) y Planas et al. (2002))	
		absorption spectrophotometry	
NPE		GC/MS (NERI (2003) y Planas et al. (2002))	
	Pb	Microwave extraction and analysis by optical ICP	
PAH	Zn	GC/MS (NERI (2003) y Planas et al. (2002))	
		Microwave extraction and analysis by optical ICP	
PCB	Cu	GC/MS (NERI (2003) y Planas et al. (2002))	
		Microwave extraction and analysis by optical ICP	
PCDD/F		V (total)	
		GC/HRMS (EPA 1613) (Method 1613)	
		K (assimilable)	
PBDE		HRGC/MS (Draft ISO/DIS 22032) (Draft ISO/DIS 22032. ISO TC 147/SC 2)	
		Fe (total)	
		Fe (assimilable)	
		Na (total)	
		Na (assimilable)	

Other studies

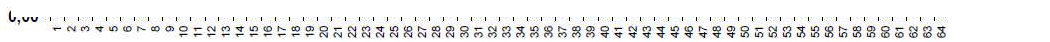
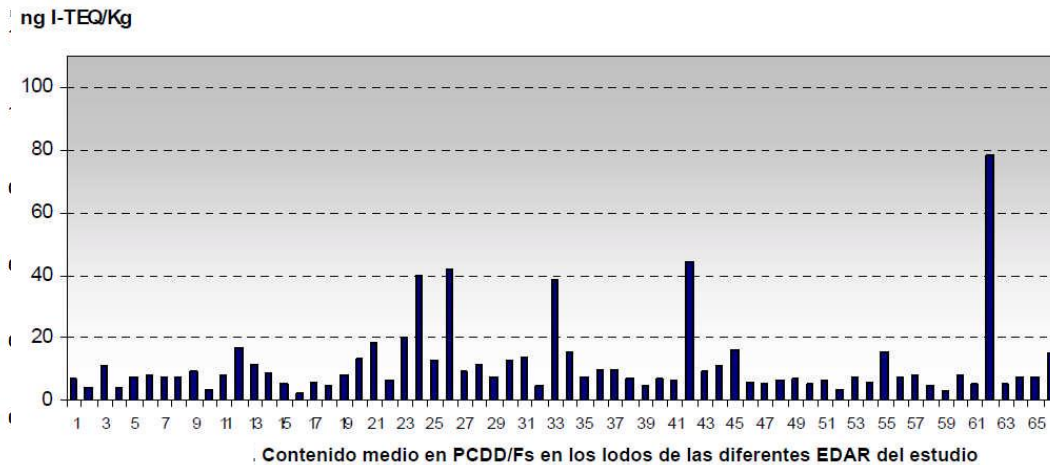
- Years 2004-2006 - **Programme of characterization of sewage sludge generated in Spain** - Study of the composition of sludge from wastewater treatment plants in Spain. Examples :



Geographical distribution of total Cr content in the sludge of different wastewater treatment plants studied

Other studies

- Years 2004-2006 - **Programme of characterization of sewage sludge generated in Spain** - Study of the composition of sludge from wastewater treatment plants in Spain. Organic parameters:



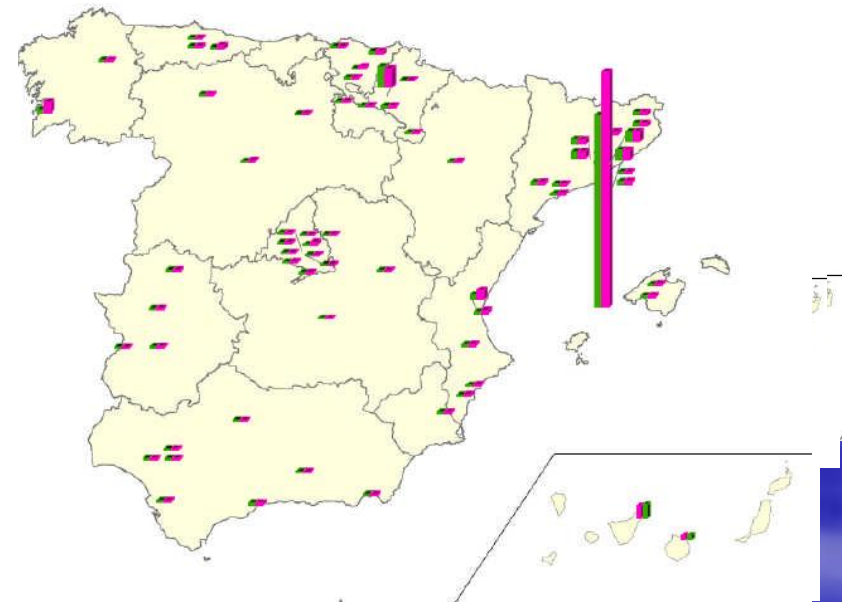
■ Media anual — Propuesta Direct
 Contenido medio de PCBs en los lodos de las diferentes EDAR del estudio
 Contenido medio de LAS en los lodos de las diferentes EDAR del estudio



MEDIANA Y MEDIA DEL CONTENIDO EN PCBs (mg/kg)



MEDIANA Y MEDIA DEL CONTENIDO EN DIOXINAS Y FURANOS EXPRESADO COMO I-TEQ (pg/g)



Other studies

- Photosynthetic pigment analysis – Calibration of remote sensing sensor .

Elution order	Pigments	Pigments	Algal Group or process
1	Carotenoids	Peridinin	Dinoflagellates
2	Carotenoids	19'-butanoyloxyfucoxanthin	Some prymnesiophytes, one chrysophyte, several dinoflagellates
3	Carotenoids	Fucoxanthin	Diatoms: prymnesiophytes, chrysophytes, raphidophytes, several dinoflagellates
4	Carotenoids	19'-hexanoyloxyfucoxanthin	Prymnesiophytes, several dinoflagellates
5	Carotenoids	Neoxanthin	Green algae: chlorophytes, prasinophytes, euglenophytes
6	Carotenoids	Prasinoxanthin	Some Prasinophytes
7	Carotenoids	Violaxanthin	Green algae: chlorophytes, prasinophytes, eustigmatophytes
8	Carotenoids	Alloxanthin	Cryptophytes
9		Myxoxantophyll	
10	Carotenoids	Lutein	Green algae: chlorophytes, prasinophytes
11	Carotenoids	Zeaxanthin	Cyanophytes: prochlorophytes, rhodophytes, chlorophytes, eustigmatophytes (minor pigment)
12		Canthaxanthin	
13	Chlorophylls	Chlorophyll b	Green algae: chlorophytes, prasinophytes, euglenophytes
14	Chlorophylls	Chlorophyll a	All photosynthetic microalgae (except Prochlorophytes)
15	Chlorophyll degradation product	Pheophytin A	Zooplankton faecal pellets, sediments

Chromatograms of studied pigments

WFD and quality monitoring network

Legal Framework

Directive 2000/60/EC:

- **Art. 8:** EEMM ensure the establishment of monitoring programs of water status in order to have a coherent and comprehensive overview of water status within each river basin. In the case of groundwater monitoring **programs** it includes **chemical and quantitative** status.
- In **protected areas**, programs are supplemented by the specifications of the standard under which it was established.
- **Anexo V.2.4:** Monitoring of the chemical status of groundwater
 - **Monitoring network of groundwater (surveillance and operative control).**
 - Aim, selection of points, selection of parameters and control frequency

WFD and quality monitoring network

- **Surveillance monitoring program:** Surveillance Network
- **Operational Control Program:** Operative Network
- **Control of protected areas program:**
 - Water used for drinking water catchment
 - Nutrient-sensitive areas: Directive 91/676/EEC
 - Other possible protected areas
- **Other programs of control:**
 - Specific pollution network (prevent or limit pollutant inputs)
- **Other information exchange obligations:**
 - Network WISE-SOE: Groundwater quality

WFD and quality monitoring network

Conventions:

- *"Conducting studies on the quality of groundwater" Basins: Duero, Guadalquivir, Segura, South and Júcar. (2001 - 2005) (Key CEDEX: 44-400-1-078)*
- *"Technical assistance, research and technological development in the areas of competence of the Directorate General of Water" (2007 - 2011) (Key CEDEX: 44-406-1-105 / 44-406-1-106 / 44-409-5 - 001). Factsheet: "Implementation of analytical determinations at checkpoints in networks of groundwater quality.« Basins: Duero, Norte, Júcar, Segura and Ebro.*

Network adaptation for groundwater monitoring. Criteria for the definition of surveillance networks (cont.)

Determination of network density

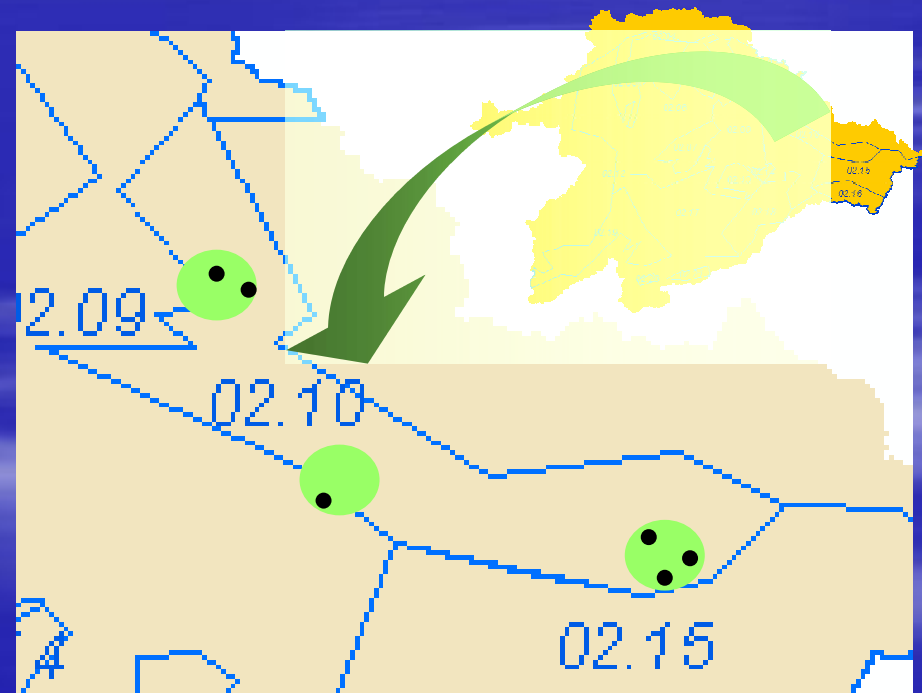
INTRINSIC CRITERIA:

- Carbonated surfaced → percentage of the UH
- Nature → presence and protection
- Relief → average slope
- Geology → relative complexity
- Aquifer type → free or confined
- Available resources → entries
- Reservations → stored volume
- Side inputs → underground connection
- Side outputs → underground connection

PRESSURE CRITERIA:

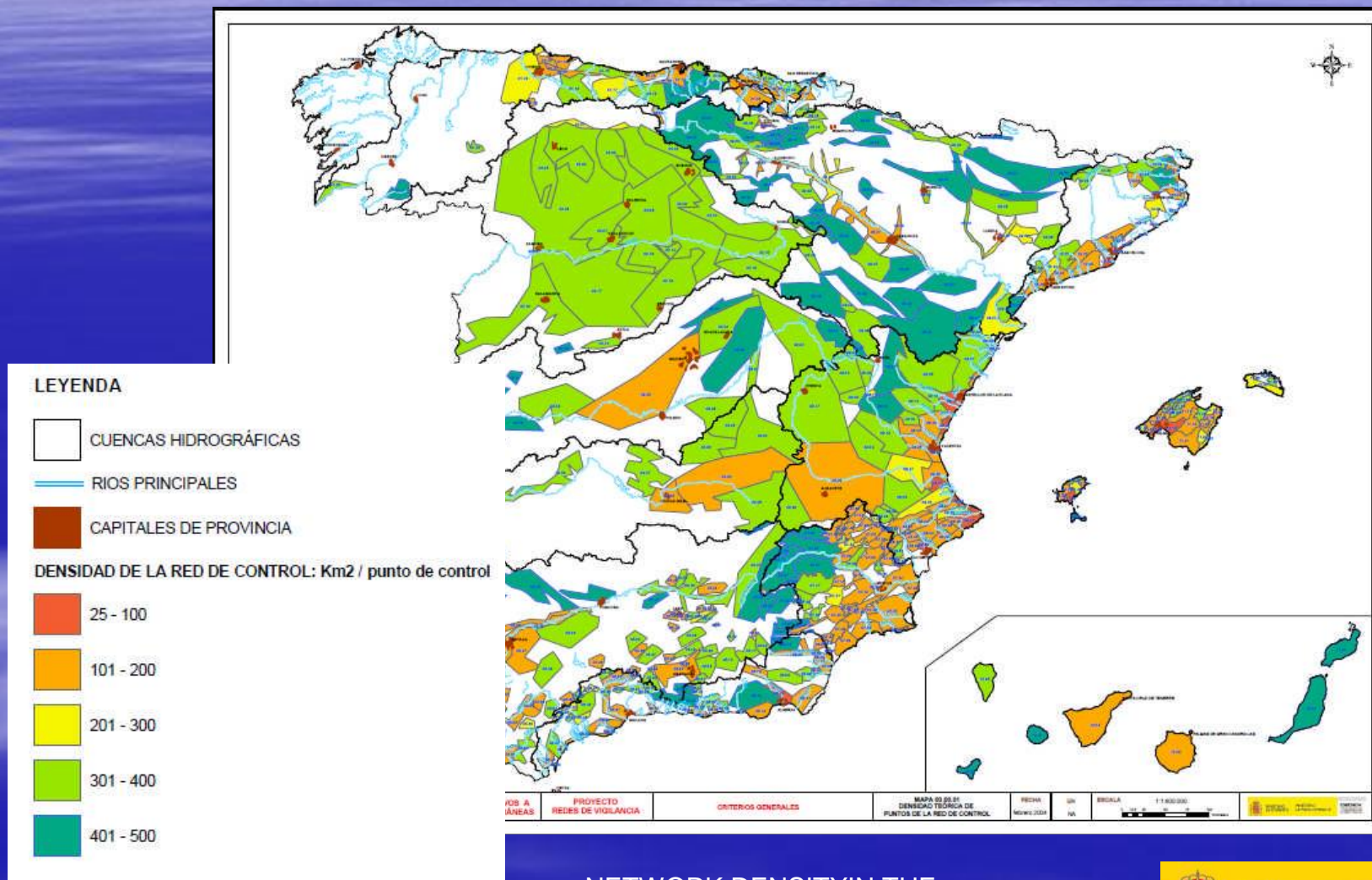
- Level of Operation → pumping regarding inputs
- Land for cultivation → percentage of land use
- Land for Industry → percentage of land use
- Mines, dumps and landfills → percentage use
- Population → population density

- Scale of regional work.
- Proposal of most favorable areas
- Proposal of points within areas
- Areas and control points must follow specific criteria



Network adaptation for groundwater monitoring. Criteria for the definition of surveillance networks (cont.)

Estimation of network density

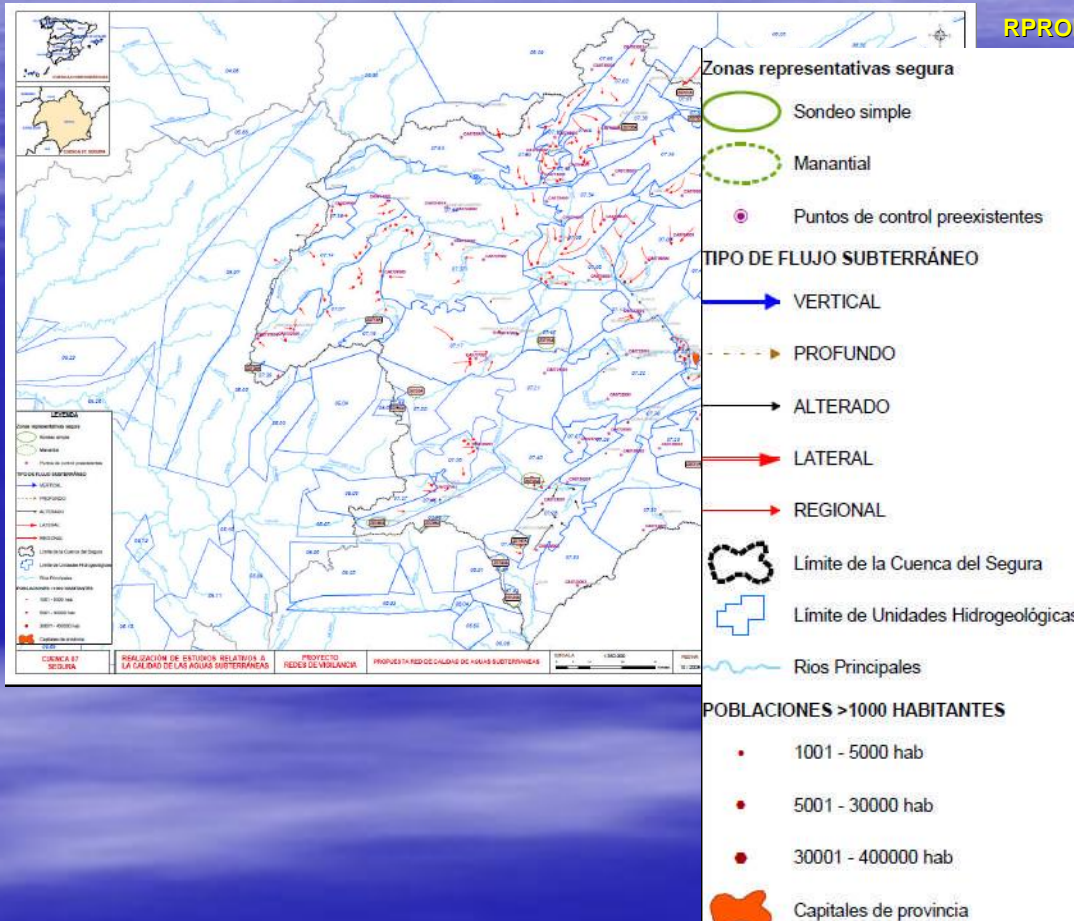


NETWORK DENSITY IN THE
INTERCOMMUNITY BASINS

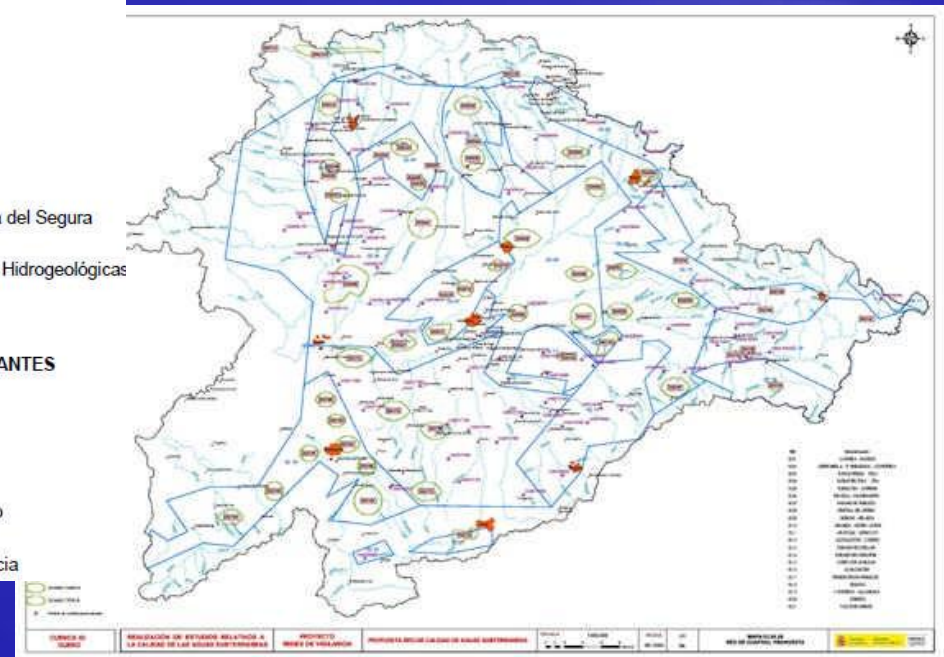
Proposed Surveillance network

PROPOSED SURVEILLANCE NETWORK

PROPOSED SURVEILLANCE NETWORK IN THE SEGURA BASIN



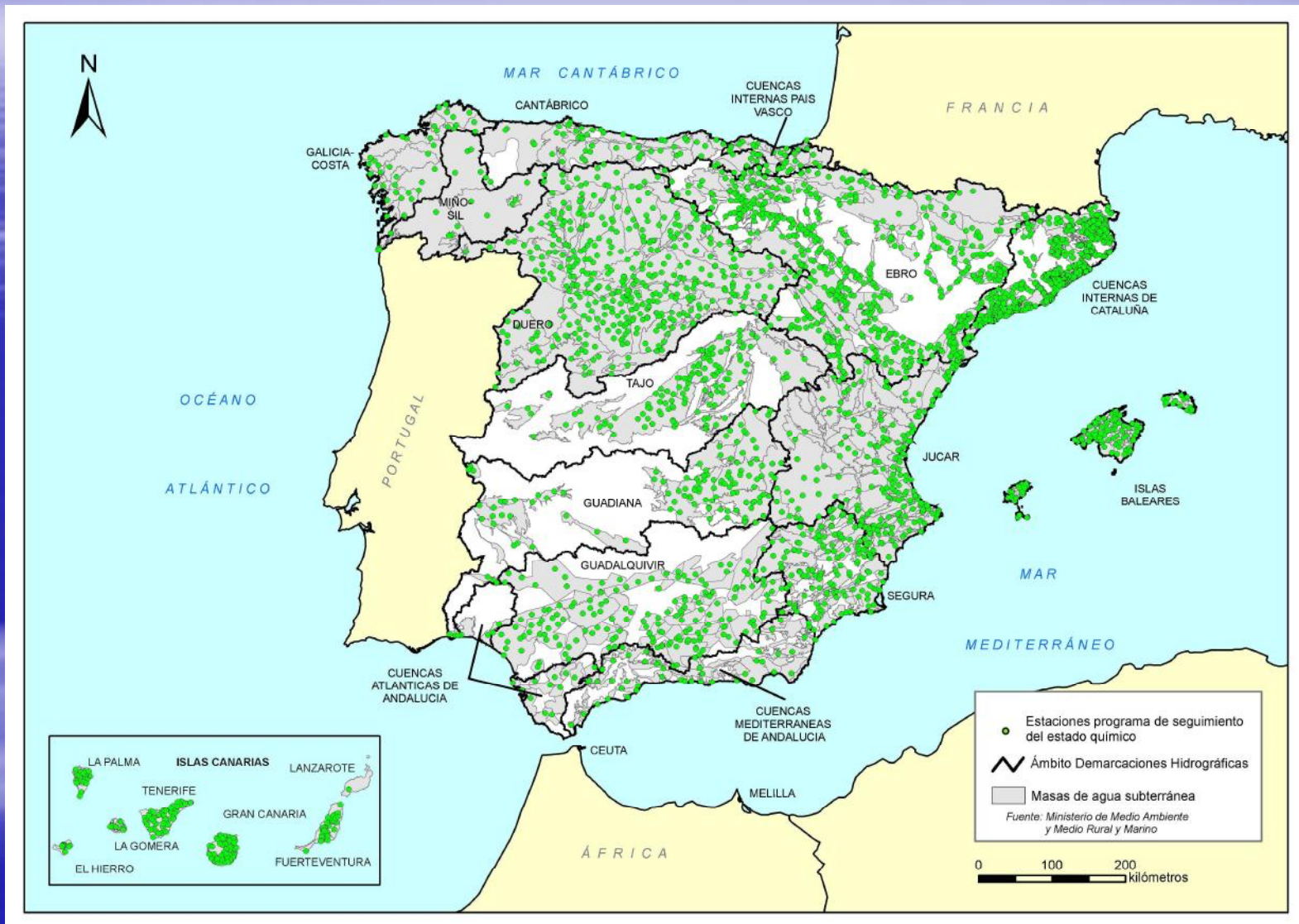
PROPOSED SURVEILLANCE NETWORK IN THE DUERO BASIN



Chemical status monitoring network

Intecommunity Demographic catchment areas	Program for chemical status monitoring	Water mass area (km ²)	Chemical status stations (n/100 km ²)	Surveillance	Operative	Protected areas
MIÑO-LIMIA	15	17.605	0,09	15	0	0
NORTE	78	17.273	0,45	78	0	28
DUERO	433	76.503	0,57	343	84	0
TAJO	126	22.311	0,56	126	6	40
GUADIANA	120	22.113	0,54	120	25	55
GUADALQUIVIR	171	34.754	0,49	133	62	64
SEGURA	145	15.025	0,97	119	4	26
JÚCAR	219	40.573	0,54	219	62	113
EBRO	616	54.858	1,12	616	188	147
TOTAL	1.923	301.014	0,64	1.769	431	473
Intecommunity Demographic catchment areas	Program for chemical status monitoring	Water mass area (km ²)	Chemical status stations (n/100 km ²)	Surveillance	Operative	Protected areas
C.M. ANDALUZA	49	10.254	0,48	49	30	36
C.A. ANDALUZA	15	4.974	0,3	15	7	0
CATALUÑA	904	9.551	9,47	500	705	0
GALICIA	41	13.133	0,31	41	0	0
PAÍS VASCO	17	2.268	0,75	15	4	10
BALEARES	113	4.196	2,69	113	67	63
CANARIAS	204	7.437	2,74	135	69	0
TOTAL	1.343	51.813	2,59	868	882	109
TOTAL ESPAÑA	3266	352.827	0,93	2.637	1.313	582

Chemical status monitoring network

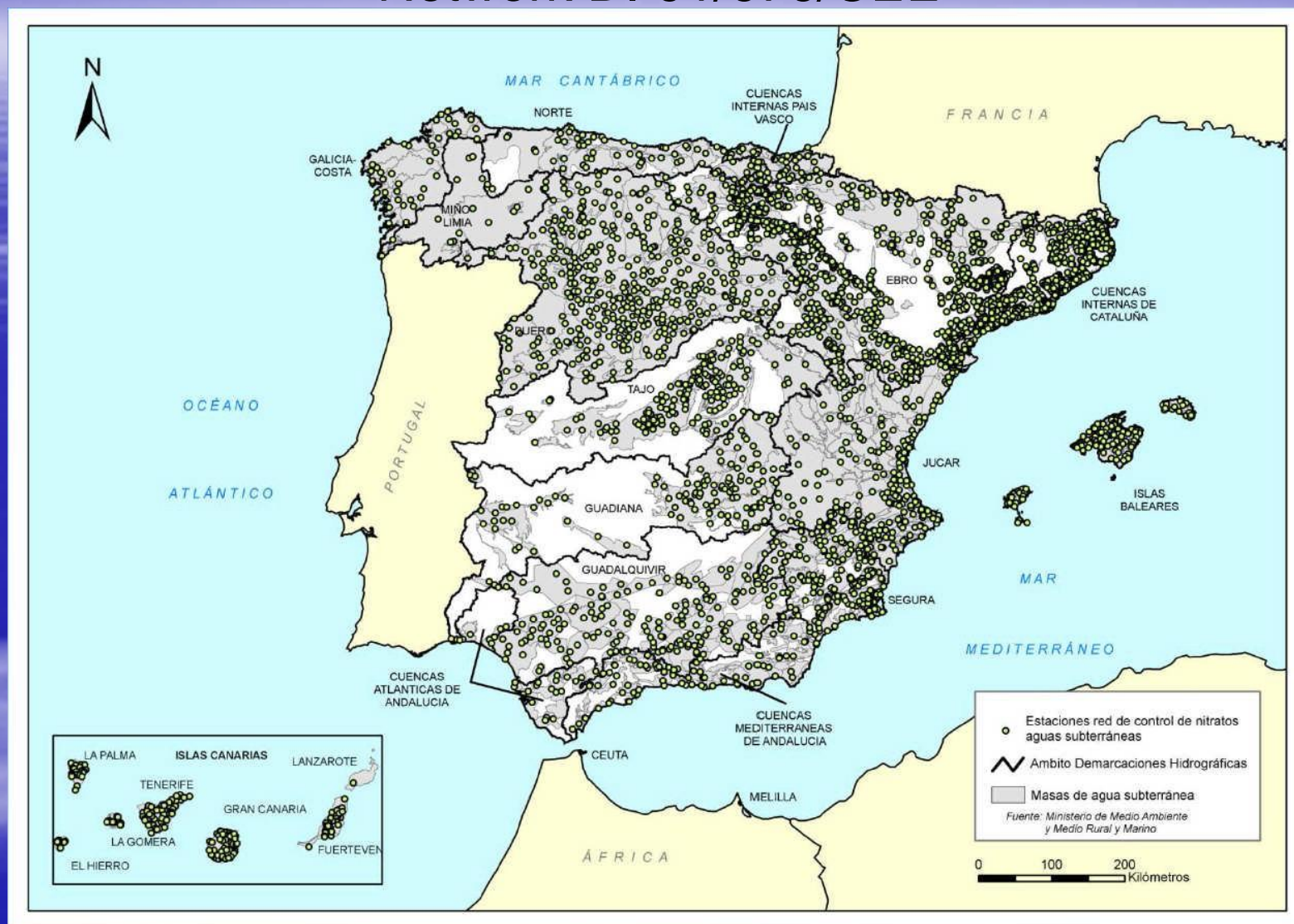


Chemical status monitoring network

Network D. 91/676/CEE

Intercommunity Demographic catchment areas	Program for chemical status monitoring	Stations Network Directive 91/676/CEE	Water mass area (km ²)	Density Chemical status stations (n/100 km ²)	Density stations Network Directive 91/676/CEE	Protected areas
MIÑO-LIMIA	15	15	17.605	0,09	0.09	0
NORTE	78	78	17.273	0,45	0.45	28
DUERO	433	437	76.503	0,57	0.57	0
TAJO	126	163	22.311	0,56	0.73	40
GUADIANA	120	120	22.113	0,54	0.54	55
GUADALQUIVIR	171	171	34.754	0,49	0.49	64
SEGURA	145	257	15.025	0,97	1.71	26
JÚCAR	219	214	40.573	0,54	0.53	113
EBRO	616	1272	54.858	1,12	2.32	147
TOTAL	1.923	2727	301.014	1.769	0.91	473
Intercommunity Demographic catchment areas	Program for chemical status monitoring	Stations Network Directive 91/676/CEE	Water mass area (km ²)	Density Chemical status stations (n/100 km ²)	Density stations Network Directive 91/676/CEE	Protected areas
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CATALUÑA	904	937	9.551	9,47	9.81	0
GALICIA	41	41	13.133	0,31	0.31	0
PAÍS VASCO	17	15	2.268	0,75	0.66	10
BALEARES	113	113	4.196	2,69	2.69	63
CANARIAS	204	97	7.437	2,74	1.30	0
TOTAL	1.343	1365	51.813	2,59	2.63	109
TOTAL ESPAÑA	3266	1092	352.827	2.637	1.16	582

Chemical status monitoring network Network D. 91/676/CEE



Chemical Status Monitoring Network: analyzed parameters in the network.

WFD (2000/60/CE) and DAS (2006/118/CE)

In situ	Principal Ions	Additional Parameters	Metals	Other pollutants
<p>pH Conductivity Dissolved oxygen Oxygen Nitrate Ammonium</p>	<p>Chlorides Sulphates Bicarbonates Calcium Magnesium Sodium Potassium Silica</p>	<p>Nitrates Nitrites Ammonium COD Totals Cyanides Fluorides</p>	<p>Iron Manganese Arsenic Mercury Cadmium Chrome Copper Lead Zinc Nickel Beryllium Cobalt Selenium Vanadium Barium</p>	<p>Pesticides Trichloroethylene Tetrachloroethylene ...</p>

SURVEILLANCE NETWORK

PARAMETERS ANALYZED IN THE LABORATORY.

MMA. EECC – 1/06 Technical Instruction

Principal Components		
Parameter	Standard	Method
Chlorides	UNE-EN ISO 77041:2002	Volumetric
	UNE-EN ISO 10304-1:1995	Ion Chromatography
Sulphates	UNE-EN ISO 10304-1:1995	Ion Chromatography
Bicarbonates	UNE-EN ISO 9963-1:1994, 1996	Volumetric
Carbonates		
Calcium	UNE-EN ISO 11885:1998	ICP-EEA
Magnesium		
Sodium		
Potassium		
Sílica	Standard Methods 19 th . Method 4500-Si F	Spectrophotometry

Surveillance network: parameters analyzed (cont.)

Additional compounds		
Parameter	Standard	Method
Nitrates	Standard Methods 19 th . Method 4500-NO ₃ B	Spectrophotometry
Ammonium	Standard Methods 19 th . Method 4500-NH ₃ C	Spectrophotometry
Nitrites	UNE-EN ISO 26777:1994	Spectrophotometry
	UNE-EN ISO 10304-1:1995	Ion Chromatography
Fluorides	Standard Methods 19 th . Method 4500-F D	Colorimetry
	UNE-EN ISO 10304-1:1995	Ion Chromatography
Cyanides	EPA 335.3	Automatized Colorimetry
Oxidability to dichromate	EPA Method 410.2	Colorimetry

Surveillance network: parameters analyzed (cont.)

Metals		
Parameter	Standard	Method
Mercury	EPA Methods 7473	Atomic absorption spectrometry
Chromium VI	Standard Methods 19 th . Method 3500-Cr D	Espectrophotometry
Arsenic	UNE-EN ISO 11885:1998	ICP- OES
Barium		
Berilium		
Cadmium		
Zinc		
Cobalt		
Copper		
Chromium (total)		
Iron		
Manganese		
Nickel		
Lead		
Selenium		
Vanadium		

Surveillance network: parameters analyzed (cont.)

Water Frame Directive Priority Substances		
Parameter	Standard	Method
Alachlor	EPA-507/8140/8141	GC/NPD
Atrazine		
Chlorfenvinphos		
Chlorpiriphos		
Simazine		
Trifluralin		
Antracene	EPA-550.1	HPLC/Fluorescence
Fluoranthene		
Naftalene		
Poliaromatic Hydrocarbons (Σ)		
Benzene	EPA-8000/8020	GC/FID
Brominated Diphenyll ethers	EPA-8000/8080	GC/ECD
C ₁₀₋₁₃ -chloroalcanes		
Endosulfan (Σ isomers α , β y sulphate)		
Hexachlorobencene		
Hexachlorocyclohexane (Σ isomers α , β , γ y δ)		
Pentachlorobenzene	EPA-8000/8080	GC/ECD
Pentachlorophenol		

Surveillance network: parameters analyzed (cont.)

Water Frame Directive Priority Substances (cont.)		
Parameter	Standard	Method
1,2-dichloroetano	EPA-5030B/8010B	P&T/GC/ECD
Dichlorometano		
Hexaclorobutadieno		
Triclorobenceno (Σ 1,2,3; 1,2,4 y 1,3,5)		
Triclorometano		
Di(2-etilhexil)ftalato (DEHP)	EPA-8270	GC/MS
Nonylfenoles (solo para p-nonylfenol)		
Octilfenoles (solo para p-ter-octilfenol)		
Compuestos de butilo de estaño (Σ mono, di, tri y tetra)		
Diuron	EPA-619	HPLC/Diodo-Array
Isoproturon		
Cadmio	UNE-EN ISO 11885:1998	ICP-EEA
Niquel		
Plomo		
Mercurio	Standard Methods 19 th .Method 3500-Hg	Cold Steam

Surveillance network: parameters analyzed (cont.)

Preferential Substances		
Parameter	Standard	Method
Atrazine	EPA-507/8140/8141	GC/NPD
Simazine		
Metolachlor		
Terbutylazine		
Benzene	EPA-8000/8020	GC/FID
Ethylbenzene		
Toluene		
Xylene (Σ orto, meta y para)		
Chlorobenzene	EPA-5030B/8010B	P&T/GC/ECD
Dichlorobenzene (Σ orto, meta y para)		
1,1,1-Trichloroethane		
Naphtalene	EPA-550.1	HPLC/Fluorescence
Butyltin Compounds (Σ mono, di, tri y tetra)	EPA-8270	GC/MS
Cyanides	EPA 335.3	Automatized Colorimetry
Fluorides	Standard Methods 19 th . Method 4500-F- D	Colorimetry
	UNE-EN ISO 10304-1:1995	Ion Chromatography
Chromium VI	Standard Methods 19 th .Method 3500-Cr D	Espectrophotometry
Arsenic	UNE-EN ISO 11885:1998	ICP-EEA
Cupper		
Chromium (total)		
Nickel		
Lead		
Selenium		
Zinc		

Current lines of work

- Emerging substances:

“Environmental contaminants that have recently been introduced into the environment, so that previously were not considered dangerous to himself, or substances that so far we know very little or nothing and whose detection has been made possible by development of new and more sensitive methods of analysis, both chemical and biological”

Current lines of work: Emerging substances

Industrial and domestic products

Insecticides	Polycyclic aromatic hidrocarbons (fossil fuels)
Diazinon	Naphtalene
Carbaryl	Phenantrene
Chlorpyrifos	Anthracene
<i>cis</i> -chlordane	Fluoranthene
N, N-diethyltoluamide (DEET)	Pyrene
Lindane	Benzo (1) pyrene
Methyl parathion	Antioxidant
Dieldrin	2,6-di-tert-butylphenol
Plasticizers	5-methyl-1H-benzotriazole
<i>bis</i> (2-ethylhexyl) adipate	Butylatedhydroxyanisole (BHA)
Ethanol-2-butoxy-phosphate	Butylatedhydroxytoluene (BHT)
<i>bis</i> (2-ethylhexyl) pftalate	2,6-di-tert-butyl-p-benzoquinone
Diethyl phtalate	Others
Triphenyl phosphate	Tetrachloroethylene (solvent)
Detergent metabolites	Phenol (desinfectant)
<i>p</i> -nonylphenol	1,4-dichlorobenzene (fumigation)
monoethoxylate nonylphenol (NPEO1)	Acetophenone (fragance)
diethoxylate nonylphenol (NPEO2)	<i>p</i> -cresol (wood preservative)
monoethoxylate Octylphenol (OPEO1)	Phtalic anhydride (used in plastic)
diethoxylate Octylphenol (OPEO2)	Bisphenol A (used in polimer)
Fire retardants	Triclosane (antimicrobial desinfectant)
Tri (2-chloroethyl) phosphate	
Tri (dichlorisopropyl) phosphate	

Current lines of work: Emerging substances

Steroids Hormones	
Biogenics	Pharmaceuticals
17 <i>b</i> -estradiol	17 etinilestradiol (ovulation inhibitor)
17 <i>un</i> estradiol-	Mestranol (ovulation inhibitor)
Estrone	19-noretisterone (ovulation inhibitor)
Estriol	Equilenine (terapia de reemplazo hormonal)
Testosterone	Equilin (terapia de reemplazo hormonal)
Progesterone	Steroles
<i>cis</i> -Androsterone	Cholesterol (fecal indicator)
	3 <i>b</i> -coprostanol (carnivore fecal indicator)
	Estigmastanol (plants sterols)

Current lines of work: Emerging substances

Veterinary and medical antibiotics

Tetracyclines

Chlortetracycline

Doxicycline

Oxytetracycline

Tetracycline

Fluoroquinolones

Ciprofloxacin

Enrofloxacin

Norfloxacin

Sarafloxacin

Macrolides

Erytromycin-H₂O (metabolite)

Tylosine

Roxitromycin

Sulfonamides

Sulfachlorpyridazine

Sulfamerazine

Sulfametazine

Sulfathiazole

Sulfadimethoxine

Sulfamethiazole

Sulfamethoxazole

Others

Lincomycin

Trimethoprim

Carbadox

Virginiamycin



MINISTERIO
DE FOMENTO

MINISTERIO DE
MEDIO AMBIENTE

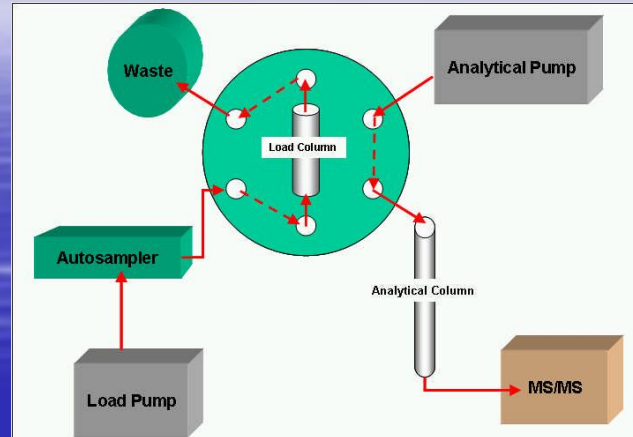
CEDEX
CENTRO DE ESTUDIOS
Y EXPERIMENTACIÓN
DE OBRAS PÚBLICAS

Current lines of work: Emerging substances

Medicines	
Metformin (antidiabetic)	Paracetamol (analgesic)
Cimetidine (antiacid)	Ibuprofen (anti-inflammatory, analgesic)
Ranitidina (antiacid)	Codeine (analgesic)
Enalapril (antihypertensive)	Caffeine (stimulant)
Digoxin	1,7-dimethylxanthine (caffeine metabolite)
Diltiazem (antihypertensive)	Cotinine (nicotine metabolite)
Fluoxetine (antidepressant)	
Paroxetine (antidepressant, anxiolytic)	
Warfarin (anticoagulant)	
Salbutamol (antiasthmatic)	
Gemfibrozil (antihyperlipidemic)	
Dehydronifedipine (antianginal metabolite)	
Digoxigenin (digoxin metabolite)	

Current lines of work: Emerging substances

LC – MS/MS



On-line Preconcentration

E-Quan system

Load Column: Hypersil Gold (C18)

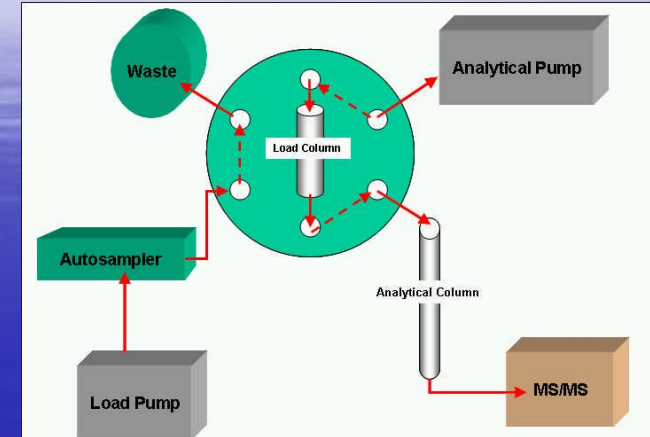
20 x 2.1 mm, 12 μ m

Mobile Phase. A: H₂O + 0.08% formic acid

B: ACN + 0.08% formic acid

Flow: 1 mL min⁻¹

Volume Injection: 1 mL



Chromatographic conditions

Analytical Column: Hypersil Gold (C18)

50 x 2.1 mm, 3 μ m

Mobile Phase. A: H₂O + 0.08% formic acid

B: ACN + 0.08% formic acid

Flow: 500 μ L min⁻¹

Mass Spectrometry (QqQ)

TSQ Quantum Discovery Max

ElectroSpray Spray Voltage: 3000 V

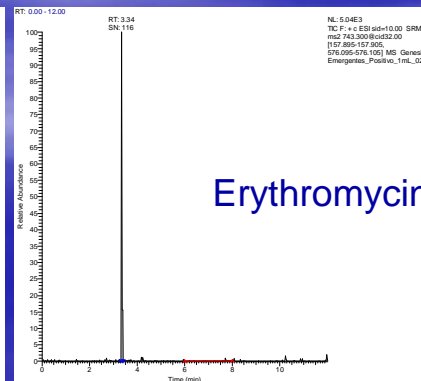
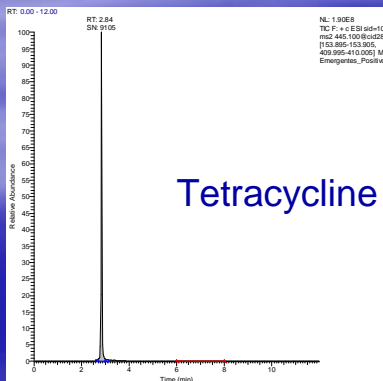
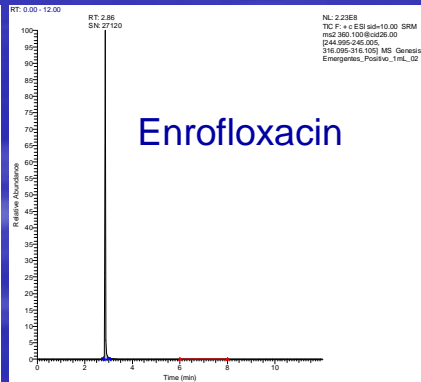
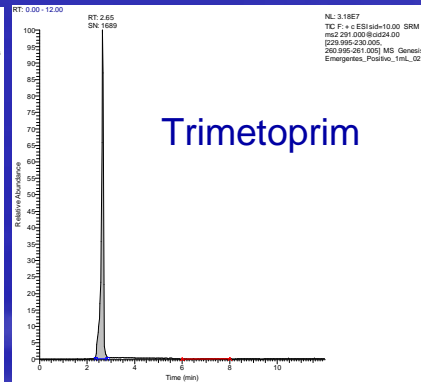
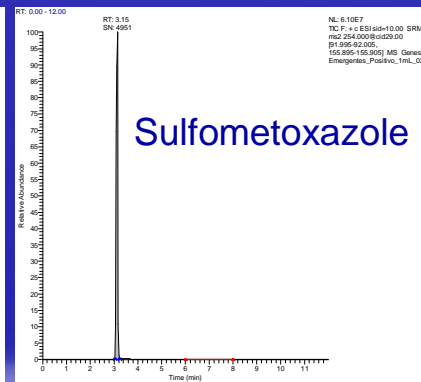
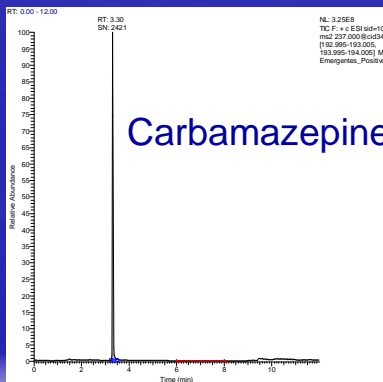
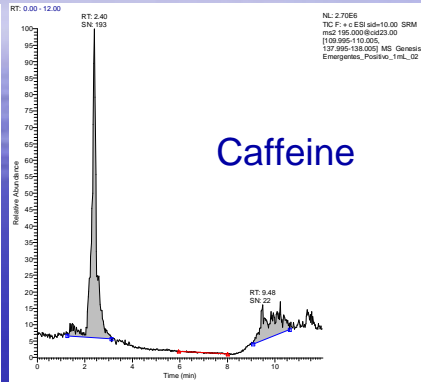
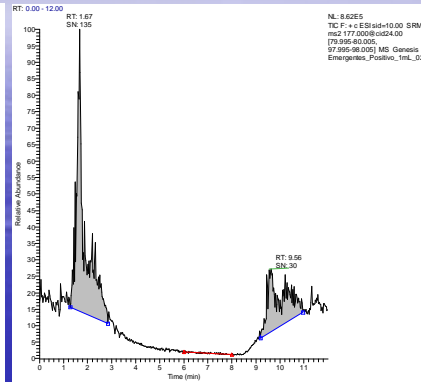
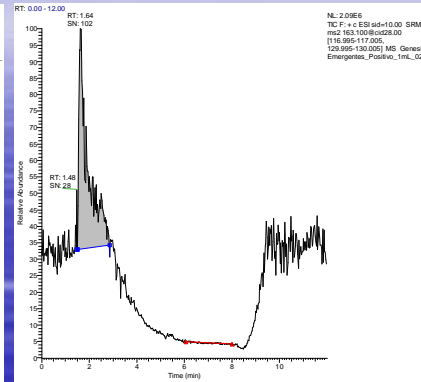
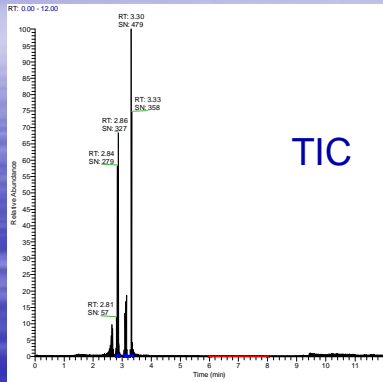
Sheat Gas Pressure: 30 u.a.

Auxiliar Gas Pressure: 5 u.a.

Ion Transfer Tube Temp: 300°C

Collision Gas (Ar): 1.5 mTorr

Current lines of work: Emerging substances



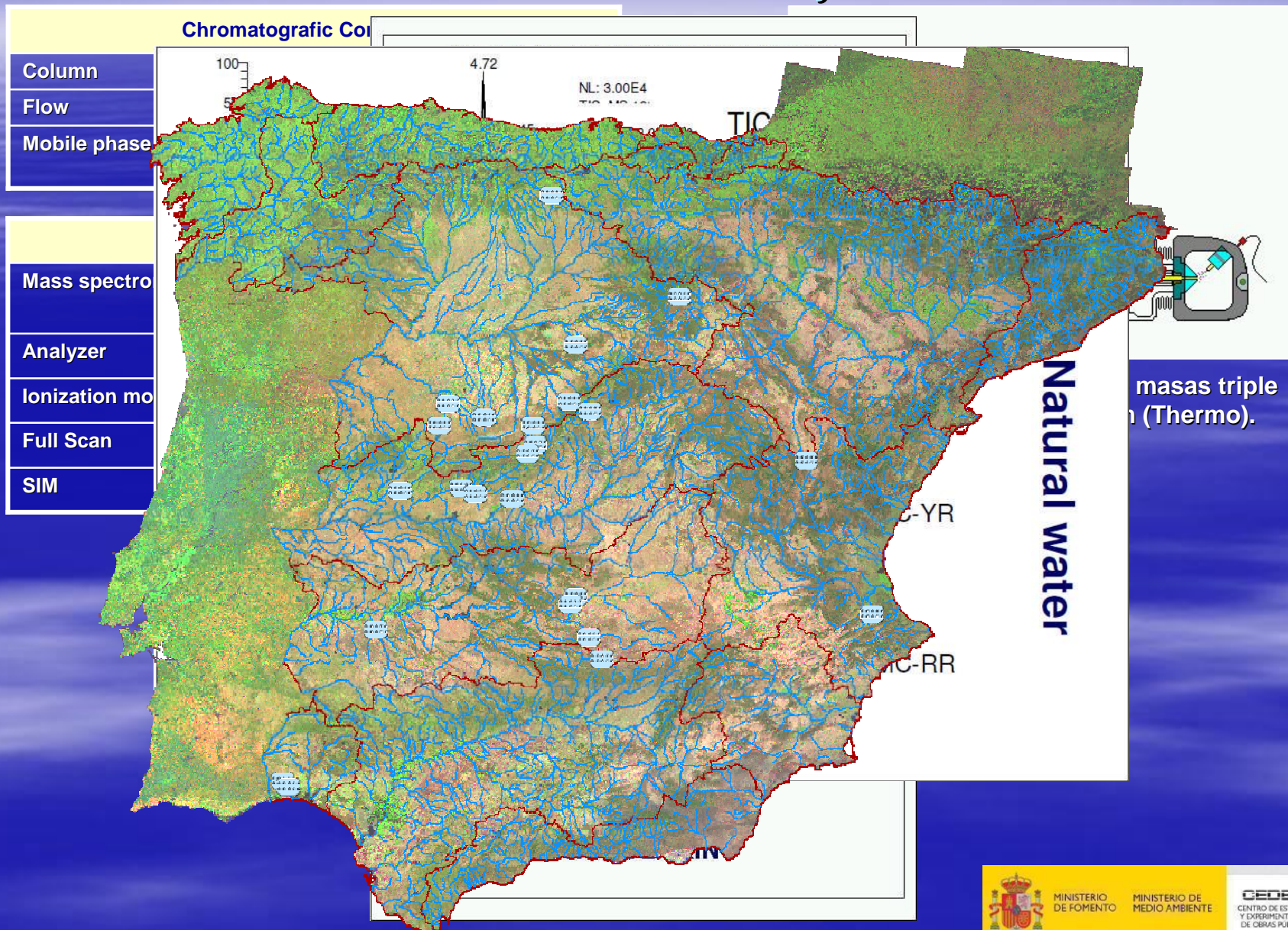
Current lines of work: Emerging substances

Algal toxins	
Cyanotoxins	Individual substances:
	Microcystin-LR
	Microcystin-RR Microcystin-YR

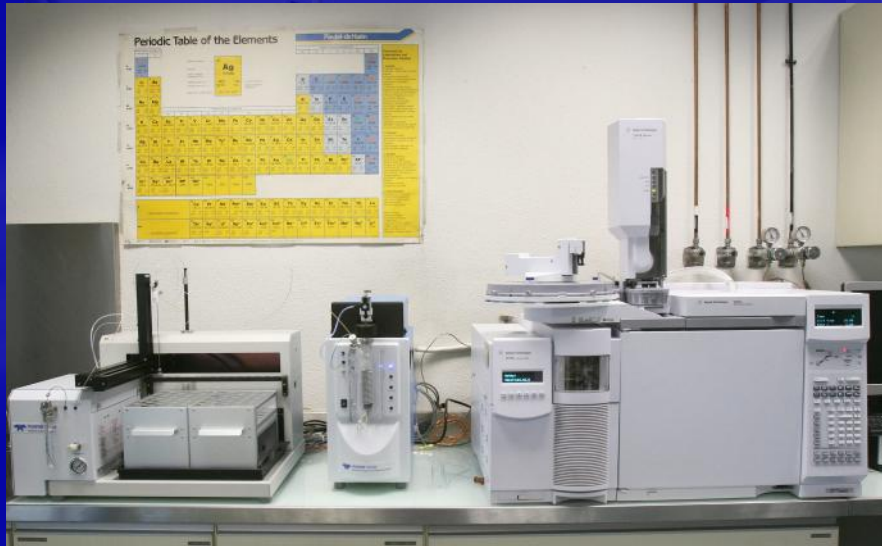
■ Cyanotoxins:

- Microcystins are a family of more than seventy hepatotoxic peptides produced by different genera of cyanobacteria (Microcystis, Anabaena, Planktothrix, Oscillatoria and Nostoc), present in surface waters as blooms.
- The "Blooms" of cyanobacteria are able to produce toxins that can cause serious health problems in water for human consumption. Its high frequency of apparition, its wide distribution and some poisoning episodes have led to consider them a serious risk worldwide. In the case of Spain, studies in recent years indicate that the most abundant toxins in the reservoirs are the microcystins (MCs), and has been estimated that over 15% of them, presented in levels of MCs ever totaling over 1 mg / L, limit value recommended by the World Health Organization (WHO) for MC-LR in drinking water.
- Spain included this same value for microcystins in DR 140/2003 (Dir. 98/83/EC) where the health criteria for water quality for human consumption are established.

Current lines of word : Cyanotoxins



Laboratory Structure



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**¡THANK YOU VERY
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