ORGANOCHLORINE POLLUTION OF THE EBRO RIVER, SPAIN: PRELIMINARY SURVEY OF AN INDUSTRIAL POINT SOURCE

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Technical Note 02/93

INTRODUCTION

The ERKIMIA Company located at Flix (Fig 1) is the only facility known to have produced polychlorinated biphenyls (PCBs) in Spain. According to De Voogt and Brinkman (1989), the plant was owned by a consortium of SA CROS and Rhône-Poulenc. PCB technical mixtures were produced under French licence with the names of Pyralene or Fenochlor. Marketing of the products was undertaken by Rhône-Poulenc España. ERCROS SA is the current owner of the plant which in turn is 40% owned by the Kuwaiti Investment Organisation (Romano pers. comm.). Other interests include a Spanish bank and a variety of small investors. It is also unclear whether production of PCBs has been discontinued since De Voogt & Brinkman (1989), Ayres (1987) and Montañes *et al.* (1990) all suggest that production was still continuing at the time their articles were published. In addition to PCB manufacture, Montañes *et al.* (1990) note that hexachlorobenzene manufacture was also carried out at the plant while Jones & Zabel (1988) identify the site as one of three producing hexachloroethane in Europe.

Total production of PCBs between 1955-84 is reported as 28,964 tonnes (de Voogt & Brinkman 1989). Between 1980-83, annual production of hexachlorobenzene at Flix was reported as 1000 tonnes per year (Montañes *et al.* 1990). At present the plant is reported to be manufacturing 120,000 tonnes of chlorine per annum using the mercury cell process. 70,000 tonnes per annum of chlorinated solvents are manufactured annually together with 1,500 tonnes per annum of monochloroacetic acid and a variety of inorganic chlorine compounds.

The Flix site is located some 80km from where the Ebro system debauches to the Mediterranean Sea. The River Ebro is the third largest river flowing into the Mediterranean with an averaged yearly flow of 550 cubic metres per second and drains an area of 80,000 km² (UNEP *et al.* 1984). As pointed out by Fowler (1986) several oceanographic and geographic characteristics of the Mediterranean Sea render it vulnerable to inputs of anthropogenic pollutants. Specifically, the sea is relatively shallow and semi-enclosed with only limited water exchange at the Straits of Gibraltar and the Black Sea. Thus the major water loss from the sea is by evaporation, so persistent pollutants tend to accumulate rather than be removed by flushing. A general lack of tides, coupled with weak coastal currents act to reduce dispersion of pollutants. The construction of dams on major rivers reduces seasonal extreme freshwater flows. Finally, the high species diversity in the Mediterranean is vulnerable to pollution insult and likely to be slow to recover from anthropogenic perturbation.

Accordingly, knowledge of point sources of anthropogenic contaminants into the Mediterranean Sea is useful both to assess potential threats and to evaluate possibilities for remediation of past and continuing impacts. Currently, direct process discharges from Flix to the River Ebro take place through 15 individual pipelines. This study reports the results from a preliminary analytical survey of the chemical content of effluents

discharged from the Flix site and sediments close to the discharge points. Some sediment analyses conducted specifically for PCBs and organochlorine pesticides are also reported. The implications of the results are discussed.

METHODS AND MATERIALS

Samples of effluent and river water were obtained from the locations indicated in Table 1. Sediment samples were obtained from close to the pipeline in each case.

SAMPLE No:	Process/location	Type	Pipe Ref.
MI2047; MI2092	shore	R,S	-
MI2048; MI2093	phosphate plant	E,S	-
MI2049; MI2094	PER plant	E,S	29
MI2050; MI2095	PER plant	E,S	-
MI2051; MI2096	PER plant	E,S	-
MI2052; MI2097	Tank farm	E,S	13
MI2053; MI2098	Tank farm	E,S	16
MI2054; MI2099	N/K	E,S	-
MI2055; MI2100	N/K	E,S	12a
MI2056; MI2101	N/K	E,S	11
MI2057; MI2102	N/K	E,S	-
MI2058; MI2103	N/K	E,S	10
MI2059; MI2104	N/K	E,S	17
MI2060; MI2105	N/K	E,S	9
MI2061; MI2106	N/K	E,S	6

Table 1: Process origin and company pipe reference for effluent and paired sediment samples taken at Flix, Spain, 25/5/92.
R: River water, E: Effluent, S: Sediment, N/K: Not Known

a) Qualitative GC/MS Screen

Analysis of extractable organic components was carried out on whole effluent collected from the point of discharge in pre-cleaned glass bottles. Some samples of river water were obtained from close to the point of discharge. After addition of an internal standard of deuterated naphthalene at 200μg per litre the sample was sequentially extracted into hexane. All solvents used were of glass-distilled grade and checked for impurities. The sample was into 5ml of the existing pH of the sample. It was then acidified with nitric acid to pH 2 and extracted again into a further 5ml of hexane. The extracts were combined and 1μl analysed by gas chromatography/mass spectrometry (GC/MS).

Wet sediments, collected in Nalgene bottles were subsampled after thorough mixing. Between 10 and 20g were then transferred into a pre-cleaned 100ml Schott bottle and 30ml of a 1:1 hexane/acetone solvent mix added together with an internal standard of deuterated naphthalene to give a concentration of 0.4 mg/kg. The samples

were then placed in an ultrasonic bath and extracted for two hours. The solvent extract was then filtered over glass wool and 15ml decanted and evaporated down under nitrogen to a final volume of 2ml. 1μl of extract was then analysed by GC/MS.

The samples were injected in splitless mode using a Hewlett-Packard 7673 autoinjector. Gas chromatography was carried out using a Hewlett-Packard 5890 gas chromatograph fitted with a HP-5890 mass selective detector. The chromatography column was a DB5 of 30m length, 0.25mm internal diameter and phase thickness 0.25μm. Peak identification was carried out using computer based probability based matching (PBM) techniques using the US National Bureau of Standards Spectral Library. Matches of >90% probability were recorded together with tentative identifications where matches <90% but >50% probability were obtained.

b) Quantitative pesticide and PCB analysis

Approximately 0.7g of sample was homogenised using a mixture of acid washed sand and anhydrous sodium sulphate as a grinding agent. Both reagents were previously baked at 700°C for five hours. Homogenates were allowed to soak overnight in a 1:1 mixture of acetone and hexane, both of pesticide analysis residue grade. Samples were extracted with repeated washings of solvent mixture into a final volume of 50ml. Extracts were shaken and allowed to stand overnight after which 25ml were pipetted into a preweighed universal flask and evaporated to constant dry weight giving the quantity of hexane/acetone soluble material in the sample. The hexane/acetone soluble fraction was then redissolved in 5ml of hexane and shaken occasionally for one hour.

Sample clean up was carried out by passing 1ml of extract through a packed column containing aluminium oxide previously held at 800°C for four hours and de-activated by tumbling for one hour after the addition of 5% distilled water. The column was eluted with 1ml aliquots of hexane until 5ml of cleaned up extract was obtained. Analysis was performed on a Varian 3400 GC fitted with a 30m DB210 capillary column temperature programmed to 190°C. Detection was by electron capture detector. Identification and quantification was carried out by comparison with a standard pesticide mixture and a PCB standard of Aroclor 1254 together with individual PCB congeners. The following pesticides and derivatives were determined: hexachlorobenzene, dieldrin, gamma-hexachlorocyclohexane (lindane), DDT and the DDT metabolites TDE and DDE. PCBs were determined for all samples. Recoveries were tested on spiked samples. Detection limits for the organochlorines were: hexachlorobenzene and gamma-hexachlorocyclohexane: 0.0016mg/kg; DDE: 0.0019mg/kg; DDT: 0.0026mg/kg; TDE: 0.0023mg/kg; dieldrin (HEOD): 0.0017mg/kg; PCBs: 0.05mg/kg. Individual PCB congener detection limits ranged between 0.0014 and 0.0224 mg/kg. The results for the three sediment samples analysed to date are reported below.

Heavy metal analysis

Analysis of the effluents for a selection of heavy metals was carried out using a Varian Liberty 100 ICP AES (Inductively coupled plasma atomic emission spectrometer) under computer control, using certified standards from MBH Analytical Ltd. UK. Prior to analysis, the effluents were acidified by addition of 2% analytical grade nitric acid. Sediment samples were not analysed for metals. Detection limits for all metals analysed were 0.02mg/kg.

RESULTS

a) Qualitative GC/MS screening analysis

The number of compounds isolated by GC/MS screening analysis together with the numbers of positively and tentatively identified compounds is shown below in Table 2. Identification was carried out on the basis of comparison of spectral traces with those held in the US National Bureau of Standards Library.

SAMPLE No:	Compounds Isolated		Compounds Identified	
	E	S	E	S
MI2047; MI2092	0	3	N/A	0
MI2048; MI2093	5	52	3 [0]	8 [3]
MI2049; MI2094	0	12	N/A	12 [1]
MI2050; MI2095	0	12	N/A	6 [0]
MI2051; MI2096	1	11	1	2 [1]
MI2052; MI2097	2	13	1	7 [2]
MI2053; MI2098	2	38	1	15 [10]
MI2054; MI2099	3	39	2	13 [11]
MI2055; MI2100	16	24	9 [4]	11 [2]
MI2056; MI2101	0	56	N/A	9 [6]
MI2057; MI2102	0	80	N/A	21 [26]
MI2058; MI2103	8	65	2 [3]	22 [10]
MI2059; MI2104	5	20	2 [1]	5 [4]
MI2060; MI2105	5	21	2 [1]	10 [4]
MI2061; MI2106	0	82	N/A	49 [4]

Table 2: Compounds isolated from effluents (E) and sediments (S) sampled in the vicinity of the Flix plant. Compounds identified to a probability >90% are shown. Compounds identified to <90% but greater than 50% probability are shown in square brackets. N/A: not analysed

The list contained in Appendix 1 shows all compounds identified to greater than 90% for each effluent and sediment sample.

b) Quantitative PCB and pesticide analysis

Results of the quantitative PCB and pesticide analysis conducted on three selected sediments are given in Table 3. Results of congener specific analyses are represented in Figure 2.

Sample No:	HCB	τ-HCH	p,p'-DDE	HEOD	p,p'-TDE	p,p'-DDT	ΣPCBs
MI2103	1.766	n/d	0.299	0.111	0.619	19.708	21.66
MI2094	30.367	n/d	1.701	0.098	4.375	16.348	35.40
MI2106	9.776	n/d	n/d	0.404	85.247	305.179	639.73

Table 3: Results of the quantitative analysis carried out for PCBs and pesticides in selected sediments sampled around the Flix site. All figures are reported as mg/kg dry weight. Water contents of samples were: MI2103: 63.5%; MI2094: 45.7%; MI2106: 68.9%.

c) Metal analysis

Results of metals analyses for effluents are recorded in Table 4. Mercury values have not yet been determined. Metal analyses for sediment samples have not been carried out.

SAMPLE No:	Ni	Mn	Fe	Cr	Zn	Cu	Cd	Co	Pb	Ti
MI2047	n/d	0.02	0.25	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2048	0.53	0.85	0.07	n/d	1.36	0.09	0.49	n/d	n/d	n/d
MI2049	n/d	n/d	0.05	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2050	n/d	n/d	0.05	n/d	n/d	n/d	n/d	n/d	0.03	n/d
MI2051	n/d	n/d	0.07	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2052	n/d	n/d	0.32	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2053	n/d	0.08	2.62	0.08	0.13	0.06	n/d	n/d	n/d	0.05
MI2054	0.17	0.54	30.0	1.15	1.43	0.55	n/d	n/d	0.63	0.15
MI2055	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2056	n/d	n/d	0.10	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2057	n/d	n/d	0.08	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2058	n/d	n/d	0.18	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2059	n/d	n/d	0.06	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2060	n/d	n/d	0.48	n/d	n/d	n/d	n/d	n/d	n/d	n/d
MI2061	n/d	n/d	0.08	n/d	n/d	n/d	n/d	n/d	n/d	n/d

Table 4: Results of metal analyses determined for effluents sampled from the Flix site expressed as mg/l. n/d: not detected

DISCUSSION

a) Effluent discharges

From the results tabulated above and the list of chemicals identified in each sample contained in Appendix 1 it is clear that the Flix operations are an important point source of organochlorine chemicals to the River Ebro system. Metal contaminants do not appear to be of great concern over the site as a whole with the exception of the process sampled in sample MI2048 which contained appreciable levels of nickel, zinc, copper and cadmium. This sample was obtained from an effluent discharged from the phosphate processing area of the site and the metal contamination most probably arises either as a result of the processing of phosphate rock, or the solvent purification of phosphoric acid. The process needs clarifying. Although phosphate rock processing effluent has not been set an emission limit value under EC Directive 83/513/EEC, this *per se* does not release member states from their obligation under Directive 76/464/EEC to set emission standards for these discharges. In all other industrial sectors, as of 1.1.89, emission standards have been set at 0.2mg/l cadmium (Cd) for effluent discharged, calculated on a monthly flow weighted average concentration.

Hexachlorobutadiene (HCBD) was found in effluent samples MI2059 and MI2060. This chemical is limited under the terms of EC Directive 83/347/EEC effective from 1990. Production of perchloroethylene and carbon tetrachloride by exhaustive perchlorination processes produce HCBD as a by-product. Such discharges are limited to 3.0mg/l on an average basis with an annual mean water quality objective of 0.1μg/l and a standstill imposed upon concentrations in sediments, molluscs, shellfish and fish.

Hexachlorobenzene (HCB) is similarly produced as a by-product of perchloroethylene and carbon tetrachloride manufacture and is regulated under the terms of EC Directive 88/347/EEC. This was found in effluent MI2051. Limit values are set according to whether discharges arise as a result of HCB production and processing or as a by-product of solvent manufacture. Limits effective from 1990 in the case of HCB production are 1mg/l as a monthly average or 2mg/l as a maximum daily average. For solvent manufacture the limits are 1.5 and 3.0mg/l respectively for monthly and daily averages. Standstill values have been specified for sediments, molluscs, shellfish and fish. Quality objectives in receiving waters are set at 0.03μg/l as of 1990 on an annual mean basis.

In the case of both HCBD and HCB, quantitative determinations were not carried out but both substances clearly require monitoring both at the point of discharge and in the receiving environment. Data should be made available for public inspection.

Hexachloroethane was found in effluent samples MI2053 and MI2054.

Jones & Zabel (1989) note that Flix is one of three plants producing this chemical commercially in Europe. It is produced by the chlorination of perchloroethylene, chlorobutadiene and 1,2-dichloroethane and hence at the Flix site is again likely to be produced as part of the chlorinated solvents manufacturing process.

b) Sediment contamination

The results of the sediment analyses suggest that the Flix plant is responsible for extensive and serious contamination of the Ebro River. A wide variety of chlorinated chemicals have been isolated including PCBs, chlorinated benzenes, hexachlorobutadiene, hexachloroethane and DDT together with its degradation products.

The qualitative GC/MS screening technique used in the sediment analyses has a relatively high detection limit and the isolation of persistent pesticides and PCBs without a clean-up stage in the analysis was highly unusual. Accordingly, quantitative determinations were carried out on the three most obviously contaminated sediments. Overall, the chlorinated compounds present resemble those found in the sediments of a receiving water for the effluents of a solvents plant in the UK reported by Johnston *et al.* (1991). The complexity of the sediment analytical traces is illustrated by Figure 3 which was derived from an extract of sample MI2106.

These quantitative analyses of the sediments show extremely high levels of PCBs. Under Dutch legislation for contaminated soils (van Gemert *et al.* 1988), levels of total extractable organic chlorine above 8mg/kg require further investigation while above 80mg/kg a clean up programme is required to remediate the contamination. MacDonald *et al* (1992) have compiled sediment quality guidelines for the protection of aquatic life derived by various national authorities. The Netherlands National Institute of Public Health has recommended limits for extractable organically bound chlorine (EOCL) in sediments of 5.5mg/kg as a target value, 7mg/kg as a directive value, 20mg/kg as a limit value for the classification of freshwater and dredged sediments. PCB congeners 28, 52, 101, 138, 153 and 180 are targeted to individual levels of 0.004mg/kg in sediments with a directive value of 0.03mg/kg. A sum target value has been recommended for PCBs of 0.02mg/kg. These values are broadly comparable to other national guideline levels.

On the basis of the values reported here for the sum of PCBs and concentration of individual congeners, the sediments impacted by the Flix operations represent a severe contamination problem which, on the basis of various derived sediment quality criterion values, is capable of impacting aquatic species. Similar considerations apply to the levels of DDT and its metabolites which exceed most of the derived criteria listed by MacDonald *et al.* (1992) and which are designed to protect aquatic species.

The results also bring into question whether inputs of the persistent organochlorines from the site into the River Ebro are

continuing. Montañes *et al.* (1990) note that in their survey of organochlorines in dissolved and particulate phases at the mouth of the Ebro, levels of p,p-DDT were generally higher than those of p,p-DDE and suggested that this indicated recent use or mobilisation of technical DDT.

The Flix site appears to be a major point source of DDT into the system. The reasons for this are unclear but may be related to contract waste incineration operations. In addition, relatively high levels of HEOD, the active ingredient of dieldrin, were also found, suggesting a local input of this pesticide. The congener specific analyses for the PCBs, recorded in Figure 2, are also suggestive of recent input of PCBs to the Ebro from the site. Sample MI2094 contained an extremely high level of PCB congener 18. This is a relatively persistent congener, substituted in the 2,2' and 5 position with chlorine (Ballschmiter *et al.* 1989). Nonetheless, it would not normally be expected to exceed levels of congener 153. Similar considerations apply to the high level of PCB 101 found in sample MI2106.

In addition, the congener profiles differ substantially between sites over a relatively small geographic area. This difference between sites is also reflected in the suite of organochlorines present in the sediment. The sediments sampled in the vicinity of the pipelines seem to reflect the integrated history of discharges. If this is the case the substantial differences, both in levels and congener profiles, argue that these chemicals have been discharged recently and that discharge may be continuing. Moreover, Montañes *et al.* 1990 note that the congener profile of PCBs at the mouth of the Ebro at the time of their survey were similar to that of the technical formulation Aroclor 1260. The congener profiles obtained from these three samples do not bear this resemblance.

Taken together the congener profiles for the three samples suggest that there has been recent input of PCBs into the system. Currently work is underway to confirm the peak in sample MI2094 is in fact due to PCB 18 and not an interfering chlorinated analyte, as there is a wide spectrum of contaminants of this nature present in the sediments. Further evidence of recent input of PCBs is provided by Porte *et al.* (1992) who note that while the closure of irrigation channels may explain the seasonal cycling of PCBs in mosquito fish in rice growing areas the data could also suggest a chronic input of PCBs into the Ebro Delta.

The findings of this preliminary survey seem to confirm the assertion by Montañes *et al.* (1990) that the Flix site is the likely source of PCBs and hexachlorobenzene entering the Mediterranean Sea through the Ebro Delta. Moreover, on the basis of the analytical findings it seems probable that inputs of DDT also originate from this site. The evidence also suggests that these inputs may be continuing or are at least of very recent origin.

c) Potential Ecosystem effects

At 22.66 - 639.73mg/kg, the concentrations of PCBs determined in this study in sediments from the vicinity of the Flix plant are among the highest recorded in the Mediterranean area. Data for marine sediments near the sewage outfalls of Marseilles show levels up to 16mg/kg dry weight while near Athens up to 0.8mg/kg PCBs have been found. Sediments near Nice, Naples and Augusta have been found to contain 1.165 mg/kg, 3.2mg/kg and 0.46mg/kg, respectively (UNEP 1990). Hence the Flix area represents a significant "hotspot" of contamination despite being some 80km inland. The transport of organochlorine contaminants to the deltaic region is reflected in the high levels of PCBs reported in the mussel *Mytilus galloprovincialis* by Fowler (1986). At the time of the analysis in 1970 the range was between 0.4 and 1.8 mg/kg wet weight. In 1980, normalised values of between 0.34 and 0.22mg/kg were reported. Albaigés *et al.* (1987) reported levels of PCBs and DDT in the whole bodies of fish. They concluded that the levels found in fish near the Ebro indicated that this area had high concentrations of organochlorines in relation to other areas in the Western Mediterranean. The seasonal dynamics of the river, together with seasonal utilisation of the water for irrigation purposes also has implications. Porte *et al.* (1992) note that around 80% of the Ebro Delta is intensively farmed for rice. Irrigation channels are opened from April to December. When these channels are closed fish populations are restricted to small pond areas where they are predated upon by egrets, herons and gulls. This plays an important role in the transfer of pollutants through the food web.

CONCLUSIONS AND RECOMMENDED FUTURE RESEARCH

This preliminary survey around the Flix site has shown the area to be a major point source of organochlorine contaminants into the Ebro River and Delta, a potentially sensitive ecosystem. These chemicals are toxic, persistent and known to bioaccumulate and in order to prevent their mobilisation, urgent attention should be given to a remediation programme to decontaminate the sediments. At the same time, a survey of PCB and other organochlorine contamination is required in the system to assess the degree to which sediment contamination has spread to the wider ecosystem. Particular attention should be given to the monitoring of species consumed as food and to tertiary consumers in the system. As a matter of urgency, the continuing fluxes of these chemicals from the site itself into the system needs to be investigated and addressed.

ACKNOWLEDGEMENT

Quantitative organochlorine analyses of sediments were carried out by J. Wright.

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APPENDIX 1

Chemicals isolated from effluents and sediments taken at Flix, Spain and identified to a probability greater than 90% using the GC/MS screening method detailed in Methods and Materials. Results are reported for paired sediment-effluent samples taken from the same location with the effluent contaminants listed first.

LAB CODE: MI2047; MI2092

None

LAB CODE: MI2048; MI2093

1,2-benzenedicarboxylic acid, dibutyl ester; heptacosane; pentacosane

hexachloroethane; hexachlorobutadiene; pentachlorobenzene; hexachlorbenzene; 1,2,3,4-tetrachloro-5-(dichloromethylene)1,3-cyclopentadiene; pentachloro(trichloroethenyl)-benzene; octachloronaphthalene;

LAB CODE: MI2049; MI2094

None;

hexachlorobutadiene; 1,2,4-trichlorobenzene; 1,2,3,4-tetrachlorobenzene; hexachlorobenzene; elemental sulphur; pentachloro(trichloroethenyl)-benzene; benzene; DDT; octachloronaphthalene; PCB136; PCB209;

LAB CODE MI2050; MI2095

None;

Hexachlorobutadiene; pentachlorobenzene; hexachlorobenzene; pentachloro(trichloroethenyl)-benzene; octachloronaphthalene, PCB209

LAB CODE MI2051; MI2096

Hexachlorobenzene

Hexachlorobenzene; eicosane,

LAB CODE MI2052; MI2097

1,3-cyclopentadiene;

tetrachloroethene; hexachloroethane; hexachlorobutadiene; hexachlorobenzene; 1,2,3,4-tetrachloro-5-(dichloromethylene)-1,3-cyclopentadiene; pentachloro(trichloroethenyl)-benzene.

LAB CODE MI2053; MI2098

hexachlorethane

tetrachloroethane; chlorobenzene; hexachlorethane; hexachlorobutadiene; tridecane; tetradecane; pentadecane; hexadecane; 1,2,3,4-tetrachloro-5-(dichloromethylene)-1,3-cyclopentadiene; octadecane; nonadecane; eicosane; heptadecanenitrile; heneicosane

LAB CODE MI2054; MI2099

hexachloroethane; 1,1,2,2-tetrachloroethane

tetrachloroethene; hexachloroethane; undecane; dodecane; hexachlorobutadiene; tridecane; tetradecane; hexadecane; hexachlorobenzene; octadecane; nonadecane; octadecanenitrile; methylbis(phenylmethyl)benzene.

LAB CODE MI2055; MI2100

1,1,2,2-tetrachloroethane, 1,3-dichlorobenzene; 1,2-dichlorobenzene; docosane; tricosane; octadecanoic acid, butyl ester; tetracosane; pentacosane

1,1,2,2-tetrachloroethene; 1,1,2,2-tetrachloroethane; 1,1,3,4-tetrachloro-1,3-butadiene; pentachloro-1,2-butadiene; dodecane; hexachlorobutadiene; tridecane; tetradecane; pentachlorobenzene; hexachlorobenzene; molecular sulphur.

LAB CODE MI2056; MI2101

None;

undecane; dodecane; tridecane; tetradecane; pentadecane; eicosane; octadecane; molecular sulphur; nonadecanenitrile.

LAB CODE MI2057; MI2102

None;

decane; undecane; dodecane; hexachlorobutadiene; 3,6-deimethyl-undecane; 2-butyl-1,1,3-trimethyl-cyclohexane; 1-butyl-2-propyl-cyclopentane; 3-methyl-dodecane; 7-tetradecene; tridecane; tetradecane; nonacosane; pentadecane; octacosane; PCB 21; nonadecane; elemental sulphur; 1,2-benzenedicarboxylic acid,

bis(2-ethylhexyl) ester.

LAB CODE MI2058; MI2103

1,4-dichlorobenzene; tricosane

chlorobenzene; decahydro-1,6-dimethylnaphthalene,; decahydro-2,3-dimethylnaphthalene; 1-heptadecene; 3-octadecene; PCB28; molecular sulphur; pentachloro(trichloroethenyl)benzene; PCB75; PCB72; PCB101; 1-chloro-2-[2,2-dichloro-1-(4-chlorophenyl)ethenyl)-benzene; p,p'DDD; methyl-bis(phenylmethyl)benzene; PCB136; hexachlorobenzene, PCB141; PCB180; PCB209.

LAB CODE MI2059; MI2104

1,2-dichlorobenzene; hexachlorobutadiene;

2,5-dimethylheptane; 1,3-dimethylbenzene; hexachlorobenzene; Hexachlorocyclohexane;

LAB CODE MI2060; MI2105

1,2-dichlorobenzene; hexachlorobutadiene.

hexachlorobenzene; 1,1'-(chlorethynylidene)bis[4]-chlorobenzene; 1-chloro-2-[2,2-dichloro-1-(4-chlorophenyl)ethenylbenzene; m,p'-DDD; 1,1'-(2,2,2-trichloroethylidene)bis [4] chlorobenzene.

LAB CODE MI2061; MI2106

None;

tetrachloroethene; chlorobenzene; bromobenzene; 1-chloro-2-methyl benzene; 1,3-dichlorobenzene; 2,4-dichloro-1-methyl benzene; 1,2,4-trichlorobenzene; hexachlorobutadiene; 1,4-dichloro-2,5-dimethylbenzene; tetrachlorothiophene; 2,4,5-trichlorotoluene; 1,2,4-trichloro-3-methylbenzene; 1,2,4,5-tetrachlorbenzene;1,4-dichloro-2-(2-chloroethyl)-benzene; 2-ethenylnaphthalene; 1,2-dichloro-4-(1-chloroethyl)-benzene; 2,4-dichloro-1-(2-chloroethenyl)-benzene; 1,2-dimethylcyclooctane; pentachlorobenzene; 1,3,5-trichloro-2,4,6-trimethylbenzene; PCB1; PCB4; PCB7; 3-hexadecene; hexachlorocyclohexane; hexachlorobenzene; PCB15; 1,1'-methylenebis(4-chloro)benzene; PCB24; PCB70; PCB80; PCB75; Pentachlorobiphenyl; 1,1'-(chloroethynylidene)bis-4-chlorobenzene; 1-chloro-2-[2,2-dichloro-(4-chlorophenyl)ethenylbenzene; p,p'-DDD; o,p'-DDT; p,p'-DDD; 1,1'sulfonylbis-4-chlorobenzene; 1,1'-(2,2,2-trichloroethylidene)benzene.