Technical note 07/97

Heavy Metal and Organic Screen Analysis of Wastewaters and River Sediments Associated with Chemical Plants in Usti nad Labem, Neratovice and Pardubice, Czech Republic, March 1997.

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Introduction

In March 1997 samples of wastewater were collected from three chemical plants discharging into the river Labe: the Spolchemie chemical plant, Usti nad Labem, which discharges to the Labe river via the Bilina river; Spolana Neratovice PVC, which discharges directly to the Labe river; and the Synthesia chemical plant based at Pardubice, which also discharges waste effluents directly to the Labe. Samples of river sediment were collected from points upstream and downstream from the chemical outfalls to assess the impact and migration of pollutants from these sites.

Thirteen samples were collected (eight sediment samples and five waste effluents). They were stored and transported to the Greenpeace Research Laboratory, University of Exeter, UK, in analytically clean glass bottles (previously rinsed with nitric acid, to remove heavy metal residues, and pentane to remove organic residues). The samples were kept cold during transit, and refrigerated immediately on arrival. Heavy metals were determined quantitatively using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) and organic compounds were identified qualitatively using Gas Chromatography Mass Spectrometry (GC-MS) screening. Details of all samples and sampling sites are given in appendices 1 and 2.

Analytical methodology

Organic Screen Analysis

All solvents were of High Purity Grade (PRAG or low haloform).Glassware used in extraction and cleaning up procedures was cleaned in detergent, rinsed with tap and deionized water, dried in the oven overnight at 105^0C and rinsed tree times with low haloform pentane.

1) Water Samples

Prior to the extraction cool samples were spiked with deuterated (d8) naphthalene as an internal standard at a concentration of 150ug/l. 20ml of pentane was added, and the sample agitated for 2 hours on a bottle roller to maximise contact between solvent and sample.

After separation of the phases, the solvent extract was filtered through hydrophobic phase separator filter and collected in precleaned reagent tube. The aqueous sample was acidified to pH 2 with 10% nitric acid, a second portion of 20ml pentane was added and the extraction procedure repeated. Finally, both extracts from the sample were combined and evaporated to a volume about 3ml.

The concentrated extract was cleaned Florisil, eluted with 95:5 mixture of pentane:toluene, evaporated down to a volume 2ml under a stream of clean nitrogen, and 1-bromonaphthalene was added as a marker.

2) Sediment samples

Approximately 30g (fresh weight) of the sample were transferred to a clean 100ml glass bottle. The sample was spiked with deuterated (d8) naphthalene as an internal standard at a concentration of 4.7mg/kg. 15ml of pentane and 5ml of acetone were added, and the sample was sonicated for 2 hours.

The extract was decanted, filtered through a precleaned hydrophobic phase separator filter and collected in a reagent tube. The sample was acidified to pH 2 with 10% nitric acid and a second portion of the solvents was added followed by above procedure.

The clean up procedure was the same as described above for the water samples.

3) chromatographic analysis

Samples were analysed by Hewlett Packard (HP) 5890 Series II gas chromatograph, interfaced with a HP ChemStation data system, and linked to a HP 5972 Mass Selective Detector operated in scan mode. The identification of the compounds carried out by computer matching against a HP Wiley 138 library of 136 000 mass spectra. Results are reported as a list of those compounds reliably and tentatively identified. Match qualities of 90% or greater are assumed to give reliable identifications; tentative identification refers to qualities between 51% and 90%. Analytes yielding match qualities of 50% or less are assumed to be unidentified.

Heavy Metal Analysis

Analytical grade reagents and analytically clean glassware were used throughout sample preparation and analytical procedures.

1) Water Samples

All samples were preserved in 10% v/v nitric acid on arrival. 50ml was transferred to a 120ml Teflon microwave vessel and digested using the same procedure and programming conditions as mentioned above. After cooling to ambient temperature, samples were filtered into 50ml volumetric flasks, diluted with deionised water, and up to a volume of 50ml, and mixed. A quality control standard (internally prepared at a concentration of 8.0 mg/l) and a blank were prepared with the batch of five samples. Both having the same acid matrix (10% v/v nitric acid) as the samples.

2) Sediment samples

Samples of river sediments were dried in an oven for 48 hours, until dry weight readings became constant. They were then crushed using a pestle and mortar until homogenous, and sieved through a 2mm mesh. 0.5g of sample was weighed into a 120ml Teflon microwave vessel fitted with a screw cap and pressure relief valve. To this 10ml of deionised water was added, followed by 7.5ml of concentrated hydrochloric acid and 2.5ml of concentrated nitric acid. The vessels were then sealed, placed on a rotating table in a microwave oven (model MDS-2000, CEM Corp.), and allowed to digest for one hour at full power (630W).

After cooling to ambient temperature, the digests were filtered into volumetric flasks, diluted with deionised water, made up to a volume of 50ml, and mixed. A Certified Reference Material: PACS-1 (trace elements in marine sediment, certified by the National Research Council Canada) and a blank were prepared with the batch of eight samples. Both having the same acid matrix (15% v/v hydrochloric acid, 5% v/v nitric acid) as the samples.

Following preparation the samples were analysed using ICP-AES, using a Varian Liberty-100 Sequential Spectrometer. The following metals were determined directly: manganese (Mn), chromium (Cr), zinc (Zn), copper (Cu), lead (Pb), nickel (Ni), cobalt (Co) and cadmium (Cd). A multi-element instrument calibration standard was prepared at a concentration of 10mg/l (again matrix matched to the samples), and the calibration itself was validated using a quality control standard, prepared from different reagent stock, at 8.0 mg/l. Samples exceeding the calibration range were diluted appropriately, in duplicate, and re-analysed. The spectrometer was re-calibrated after twenty determinations to adjust for fluctuations in sensitivity. All other instrument and laboratory quality control procedures were adhered to.

Mercury (Hg) was determined using Cold Vapour Generation ICP-AES. Hg (ii) was reduced to Hg (0) i.e. a vapour, following reduction by sodium borohydride (0.5% w/v) and hydrochloric acid (10 Molar). The vapour was carried in a stream of argon into the spectrometer. Two calibration standards were prepared, at 10ug/l and 100 ug/l (matrix matched to samples). Samples exceeding the range were diluted and re-analysed. The quality control standard, from different reagent stock, was prepared at 80 ug/l. The spectrometer was re-calibrated after every 10 samples, and each new calibration validated using the quality control standard as mentioned above.

Results

See appendices 1 and 2 for full samples descriptions, results, Certified Reference Material and quality control data.

Discussion

Organic Screen Analysis

<u>1. Spolana Neratovice PVC plant</u>

5 samples were analysed in the association of Spolana PVC plant: one waste water taken from the plant discharge canal (MI7011), two sediments collected 1.2-1.5km upstream of the

plant (MI7008 and MI7012), and two sediments collected 1.5-2.7km downstream of the plant (MI7009 and MI7010).

One chloroorganic compound was reliably identified in the waste water sample - 1,2,4-trichlorobenzene. A commercial grade (mixture of trichlorobenzenes) is used to combat termites and been found to cause irrigation to eyes and mucous membranes (Merck, 1989).

Two isomers of dichlorobenzenes were identified in the sediment samples, collected downstream from the plant. 1,2-dichlorobenzene (o-DCB) is used as a solvent for waxes, gums, resins, tars, rubbers, oils; insecticide for termites and locust borers; fumigant; as heat transfer medium; as intermediate in the manufacture of dyes. o-DCB can cause injury to the liver and kidneys. High concentration cause central nervous system depression (Merck, 1989) 1,4-dichlorobenzene (p-DCB) is widely using as a space deodoriser, for moth control and as a chemical intermediate in the production of PPS resins. p-DCB, as well as other chlorobenzenes, is released to the environment during both its manufacture and use. Heavily polluted and/or industrialised areas tend to have the highest concentration of dichlorobenzenes in air and water, including surface water, groundwater, and drinking water. p-DCB is reported to cause headaches and dizziness, toxic effects in the liver and kidney, to increase rates of cancer among experimental animals and to result in birth defects (USPHS, 1989).

Two representatives of the class of polycyclic aromatic hydrocarbons (PAH) were tentatively identified in the samples MI7009 and MI7010: fluoranthene and pyrene. PAHs can cause harm to human health. Report in human shows that individuals exposed by breathing or skin contact for long periods of time to mixture of PAHs and other compounds can develop cancer (USPHS, 1990a).

2,6-bis(1,1-dimethylethyl)-4-methylphenol was identified in the sample MI7010. This compound, also known as butylated hydroxytoluene (BHT), is frequently employed as an antioxidant in food products and in the production of plastics and other petrochemical products. It is also manufactured as an antiskinning agent in paints, varnishes and other surface finishes, and as an antioxidant. Its use in food has been associated with certain allergic reactions (Dean *et al*. 1986) and there is also some evidence that BHT can act as a promoter of liver cancer, in combination with carcinogenic substances, through induction of abnormal liver metabolism (Williams *et al*. 1986).

The samples MI7008 and MI7012 collected upstream from the plant have shown generally the same group of the compounds. In addition, hexachlorobutadiene was reliably identified in the sample MI7008.

Data regarding the effects of exposure to hexachlorobutadiene on humans are sparse. Serum bile acids were increased in workers exposed to vapour concentrations of 0.005-0.02 ppm. These effects could not be attributed to hexachlorobutadiene alone because the workers were also potentially exposed to other chemicals (carbon tetrachloride and perchloroethylene) Although there are studies available on the systemic (respiratory) effects following inhalation, data are not sufficient to identify a reliable NOAEL value by this route. Much of the available data involve oral exposures in rats. The primary health effect associated with intermediate-duration and chronic-duration oral exposures to hexachlorobutadiene is kidney damage, which occurred in this organ at doses of 0.2 to 20 mg/kg/day. The liver was affected to a lesser extent and effects occurred at doses of 6.3 or 15.6 mg/kg/day. Acute dermal studies, although limited, confirm the toxic effects of hexachlorobutadiene on these organs. Hexachlorobutadiene did not adversely affect reproduction in animals except at high doses (150 mg/kg/day for 10 weeks). Although there was some evidence of fetotoxicity in animals after inhalation (10 ppm) or oral (15 mg/kg/day) exposure, embryolethality and teratogenicity were not detected. Oral studies in animals indicate that hexachlorobutadiene may increase the risk of renal cancer at dose levels of 20 mg/kg/day. The effects of hexachlorobutadiene are most pronounced after repeated chronic exposure to low doses, suggesting that effects are cumulative. For this reason, there is greater concern for populations living near hazardous waste sites, where exposure to low levels may occur for long periods of time, than for acute exposure scenarios. Lethality may be of concern in humans following exposure to high doses of hexachlorobutadiene (USPHS 1994a).

2. Spolchemie chemical plant

Three samples - two sediments (MI7007 upstream from plant, MI7006 - downstream) and one waste water (MI7005) - were analyzed in association with Spolchemie chemical plant.

Waste water sample has shown a wide range of long chain alkylbenzenes reliably and tentatively identified. The source of these is unknown, though they may be a product of the degradation of the linear alkylbenzene sulphonate (LAS) detergents. The alkylbenzenes are highly resistant to degradation and may accumulate in sediments (Preston & Raymundo 1993).

Two PAHs - naphthalene and its chlorinated derivative - as well as 2,4-bis(1,1-dimethylethyl)phenol were identified in sample MI7005. The PAHs are briefly discussed above.

Sediment samples from this site have shown significant difference in the compounds identified. Additionally to several PAHs found in both samples, the sample MI7006 taken downstream of the plant contained o,p’-DDT and p,p’-DDD. DDT, a cheap and effective insecticide, was widely used in the past, and even sprayed directly onto people. Technical grade DDT is a mixture of p,p’-DDT and, to a lesser extent, o,p’-DDT, with traces of other analogues. Dichlorodiphenyldichloroethane (p,p’-DDD) is a metabolite of p,p’-DDT which occuring through the process of dehalogenation in the environment as well as in human body. Poisoning by DDT may occur by ingestion or by absorption through skin or respiratory tract. Acute poisoning causes tremors of head and neck muscles, tonic and clonic convulsions, cardiac or respiratory failure, death. The estimated oral fatal dose is 500mg/kg body weight of solid material. Solvents such as kerosene increase toxicity. Death occurs in 2 to 24 hours. Chronic exposure to DDT and its metabolites causes hepatic damage, central nervous system degeneration, agranulocytosis, dermatitis, weakness, convulsions, coma, death (Merck, 1989).

Another chloroorganic compound reliably identified in the sediment sample MI7006 is hexachlorobenzene (HCB). HCB is not currently manufactured as a commercial product, and evidence indicates that it has not been commercially produced since the late 1970s. However, some hexachlorobenzene is produced as a by-product or impurity in the manufacture of chlorinated solvents (i.e., tetrachloroethylene, trichloroethylene, carbon

tetrachloride), other chlorinated compounds (i.e., vinyl chloride), and several pesticides, including pentachloronitrobenzene, chlorothalonil, dacthal, picloram, and pentachlorophenol. Current releases to soils may occur through land disposal of hexachlorobenzene-containing wastes, from discharges from manufactured facilities, and from use of currently registered pesticides containing small amounts of hexachlorobenzene. Being one of the most persistent environmental pollutants, hexachlorobenzene bioaccumulates in the environment, in animals, and humans. Traces of hexachlorobenzene have been found in almost all people tested.
Historically, hexachlorobenzene was released to soils directly through its application as a fungicide on seed grains. Reports on oral human exposure and oral studies in animals demonstrate that porphyria, cutaneous lesions, neurological effects, altered enzyme levels, and changes in morphology of the liver are principal toxic manifestations of oral exposure to hexachlorobenzene. Adverse effects on the kidneys and immune system, as well as reproductive and developmental effects, have also been reported. These effects are manifested as alteration in the histology of immune organs and function, abnormal development of the fetus, and alteration of reproductive and developmental parameters, particularly ovarian histology and lactation and pup viability indices. Animal studies also show that oral hexachlorobenzene exposure can cause cancer of the liver, kidney, lungs, lymphatic system, blood, and thyroid (USPHS 1994b)

3. Synthesia Chemical plant.

5 samples were analyzed associated with Synthesia Chemical plant: three waste water samples (MI7013, MI7015 and MI7016) and two sediment samples (MI7014 downstream of plant, MI7017 - upstream).

Dichlorobenzenes (see toxicity data above) were reliably identified in both sediment samples.

All three waste water sample have shown similar picture of di- and trichlorinated benzenes with addition of dichlorotoluene, identified only in the samples MI7013 and MI7015. There is little information about the toxicity of the chlorinated toluenes available, but toluene itself is potential to cause adverse health effects in exposed people. Toluene is used as a component of gasoline, both as a nonisolated component and through addition of the isolated product into gasoline to improve octane rating. The minor part of the toluene produced, as well as other alkyl-benzenes, are used in solvents paints, inks, adhesives, cleaning agents, and for chemical extraction and in the chemical synthesis of organic chemicals. The most important health concern for humans from exposure to toluene is its harmful effects on the nervous system. Short-term exposure to high amount of toluene results in lightheadedness and euphoria and in some cases death. Long-term exposure to low and moderate amount of toluene has caused slight effects on kidneys in some people. Long term exposure to high amount of toluene by intentional abuse has been linked with permanent brain damage (USPHS, 1990b).

The Synthesia chemical plant manufactures a wide range of organic nitrogenated and chlorinated compounds. The chlorinated benzenes and closely related compounds, dichlorotoluenes, were found in high concentrations in the wastewater from this facility. These compounds are of particular environmental importance. Trichlorobenzene is listed on Annex 1A of the North Sea Ministerial Declaration (MINDEC 1990) as a priority pollutant for both air and water. The North Sea Ministers committed their governments to reducing inputs of the 36 priority pollutants on Annex 1A to half of 1985 levels. The three dichlorobenzenes which were also detected in the effluents are on Annex 1D of the Ministerial declaration indicating that they should be considered for prioritisation in the future.

The chlorobenzenes were detected in all effluent samples from this facility and also in all sediments collected in the vicinity of the plant. This points to extensive contamination of the local environment by these pollutants. Chlorobenzenes were also found in sediments upstream of the plant. This could indicate another source of input or atmospheric transport and subsequent deposition of emissions from Synthesia. This warrants further investigation.

The situation at Neratovice is complex. In the past, extensive organochlorine contamination was identified as a result of chlorophenol production (Zemek and Kocan 1991). Even if cleanup measures have been initiated, it is inevitable that some contamination will remain. Consequently, it is difficult to be certain whether sediment contamination in this region results form the discharges analysed here or other sources. One of the sediments upstream of the discharge canal sampled contained hexachlorobutadiene and dichlorobenzene. The effluent from the Spolana PVC plant here analysed contained trichlorobenzene, but no other organochlorines. This is highly unusual since wastewaters from PVC manufacturing facilites tend to contain numerous toxic organochlorines (Johnston *et al.* 1994) and the chlorinated dioxin and furans are also inevitably produced (Stringer *et al.*1995). Again further investigation of this whole site should be undertaken to identify the sources of this contamination.

The effluent from Spolchemie, as from the other plants, contained a toxic organochlorine element. This did not appear to reflect the contamination of the sediment but it must be remembered that facilities of this type frequently operate on a batch basis, manufacturing a specific chemical only for a short time. The effluent, therefore, may not give a full picture of the release pattern from this site. In the sediment downstream of the plant, though not at the upstream site, hexachlorobenzene and *o,p*-DDT were identified to better than 90% and *p,p*-DDD was tentatively identified. This is a very interesting finding in that *o,p*-DDT has a short half-life and is therefore rarely found in environmental samples. This result would therefore indicate that there is a current or recent source of this highly toxic pesticide to the Labe River.

In conclusion, there is widespread pollution of the section of the Labe river that we have investigated. Primary concerns must focus on the organochlorines, which are being released into the system at a number of points. because of the generally toxic and persistent nature of this class of compounds, stringent measures should be taken to minimise their input. In the longer term, however, ceasing to produce and use these chemicals is the preferred solution.

Heavy Metal Analysis

1. Spolana Neratovice PVC plant

Five samples were collected from this site. One sample of wastewater, collected from an effluent canal discharging into the Labe river, and four river sediments (two upstream and 2 downstream from the plant). The wastewater contained detectable levels of Mn, Zn and Cu.

Again ug/l (ppb) levels are found, which will not have deleterious effects on the surrounding aquatic environment. Analysis of the upstream sediments shows slightly elevated levels of Mn, Zn, Cu, Pb and Cd to be present. Similar levels are found in both samples. The downstream sediments also contain slightly elevated levels of Mn, Zn, Cu, Pb and Cd. Again similar concentrations are found in both samples. All four samples contain similar levels of heavy metals, thus the slight elevations above published background data observed, cannot be sourced to the PVC plant.

2. Spolchemie Chemical Plant

Two river sediments and one waste effluent sample were collected from this site. Liquid waste is discharged via an effluent canal to the Bilina river, which then discharges into the Labe. Wastewater from this effluent canal was collected, along with two river sediment samples, upstream and downstream of the plant.

Only Mn and Cu were present at detectable levels in the effluent sample, 190 and 30 ug/l (i.e. parts per billion) respectively, and these levels are unlikely to have a deleterious effect on the aquatic environment. However heavy metals were found at slightly elevated levels in the sediments. Sediments will always contain some level of heavy metal ions, levels very much dependant on the geology of the area as well as anthropogenic inputs.

Analysis of sediment provides an excellent picture of the extent of pollution within a defined area. Water, due to fluctuations in emission and flow, follows a less well defined pattern. Heavy metals will bind predominantly to suspended material and finally accumulate in the sediment.

Similar concentrations of the metals determined are found in both samples. The following concentration trend is observed:

$$Mn > Zn > Cu > Cr, Pb, Ni > Co > Cd$$

On comparison with background data (Salomons and Forstner, 1984; Bryan and Langston, 1991), it appears that levels of Cr, Zn, Cu, Pb and Cd are slightly elevated. However as there are no significant difference between levels detected in the upstream and downstream samples, we cannot assume the elevation is due to discharges from the Spolchemie plant. Also, as negligible levels of heavy metals were found in the effluent sample, it is more likely that the concentrations found are indicative of the natural geology in the area, or that the levels reflect past industrial activity in the area. Regarding the present wastewater discharge from Spolchemie, our analysis leads us to draw the conclusion that either effective effluent treatment systems are in place within the plant or, more probably, that heavy metal compounds are not being manufactured or utilised there.

3. Synthesia Chemical Plant

Five samples were collected from this site. Three samples of wastewater, and two samples of river sediment, upstream and downstream of the plant’s effluent canals. Two effluent canals were sampled. One of which (ref. canal no. 2) receives waste effluent from the plant as well as treated sewage. The effluent from canal 1 was found to contain detectable levels of Mn, Cr, Zn, Cu, Pb and Ni. Of all the wastewaters sampled this is the most contaminated, and the levels found lead us to conclude that heavy metal compounds are being manufactured or utilised by the plant. Levels found in canal 2 are lower. Mn, Cr, Zn and Cu are still detected, however the effluent from the plant is obviously being diluted by the treated sewage. A third sample of wastewater was collected from a retention basin, a storage facility for wastewater prior to discharge into the Labe. Significant levels (mg/l) of Cr, Zn and Cu were found in this sample, along with detectable levels of Mn, Pb, Ni and Co.

Regarding the sediment analysis, higher levels of Cr, Zn, Cu, Pb, and Cd are found in the sediment collected downstream, compared to that collected upstream. Thus the metals discharged from Synthesia are accumulating in the river water sediment. Discharge of Cr, Zn, Cu and Pb is regulated in Europe by specific EC Environmental Legislation. All are cited in List 2 of the Dangerous Substance Discharges Directive (76/464/EEC), selected on the basis of their toxicity, persistence and bioaccumulation. Such discharges are controlled using an Environmental Quality Objective (EQO) approach with quality standards being set nationally. However member states are required by the Directive to establish programmes to reduce pollution by these substances. The manufacture of compounds of these metals and/or their utilisation requires further investigation.

In conclusion, the rivers Labe and Bilina contain levels of heavy metals typical of a water system that receives industrial and domestic waste, from both past and present human activities. Although levels are not indicative of severe heavy metal pollution, experimental studies have shown that heavy metals, especially Cd, Cr, Cu, Hg and Zn are toxic to some aquatic and benthic species at environmentally realistic levels , such as those found here. The availability of these metals, and thus there ability to bioaccumulate in aquatic organisms will be dependant on many parameter not measured in this study, such as chemical speciation/form; physical parameters such as pH, conductivity, temperature; the organic content of the sediment, and the presence of more abundant metallic species such as Fe.

However, we can conclude that through human activity, the levels of heavy metals (notably Mn, Cr, Zn, Cu, Pb and Cd) found in the fresh water sediments of the Labe and Bilina are slowly increasing above background levels associated with uncontaminated environments. Thus levels of heavy metals discharged into the Labe river must be carefully and consistently monitored, to allow for an on-going assessment of their impact on this aquatic ecosystem.

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INORGANIC ANALYTICAL RESULTS

Czech Republic, 1997

Sample type: Quality Control 1
Lab. code: Instrument QC 1
Preparation Date: 10.03.97

Other information: 8.0 mg/l Quality Control

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/l)
Manganese	7.89
Chromium	8.01
Zinc	8.02
Copper	8.02
Lead	8.00
Nickel	7.97
Cobalt	7.97
Cadmium	7.96

INORGANIC ANALYTICAL RESULTS

Czech Republic, 1997

Sample type: Quality Control 2
Lab. code: Instrument QC 2
Preparation Date: 10.03.97

Other information: 8.0 mg/l Quality Control

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/l)
Manganese	8.12
Chromium	8.13
Zinc	8.24
Copper	8.00
Lead	8.08
Nickel	5.01
Cobalt	7.95
Cadmium	8.05

INORGANIC ANALYTICAL RESULTS

Czech Republic, 1997

Sample type: Certified Reference Material
Lab. code: PACS-1
Preparation Date: 10.03.97

Other information: Trace Elements in Marine Sediment. Certified by the National Research Council, Canada.

Analysis method: ICP-AES

METAL	% Recovery
Manganese	82.5
Chromium	75.9
Zinc	84.2
Copper	83.6
Lead	82.5
Nickel	81.4
Cobalt	88.3
Cadmium	89.2

INORGANIC ANALYTICAL RESULTS

Spolchemie Chemical Plant, Usti nad Labem, Czech Republic, 1997

Sample type: Industrial wastewater
Sample name: Spolchemie discharge water (no. 1)
Lab. code: MI7005
Sampling date: 26.02.97

Other information: Wastewater discharge from Spolchemie. Collected from the effluent canal prior to confluence with the Bilina river.

Analysis method: ICP-AES

METAL	CONCENTRATION (ug/l)
Manganese	190
Chromium	<10
Zinc	<10
Copper	30
Lead	<30
Nickel	<10
Cobalt	<10
Cadmium	<10

ORGANIC ANALYTICAL RESULTS

Spolchemie chemical plant,Usti nad Labem, Czech Republic, 1997

Sample type: Industrial waste water
Sample name: Spolchemie discharge water, (no.1)
Lab. code: MI7005
Sampling date: 26.02.97

Other information: Waste water discharge from Spolchemie. Collected from the effluent canal prior to cofluence with the Bilina river.

Analysis method: GC/MS screen

Number of compounds isolated: 54

Compounds identified to better than 90%:

Junipene
Benzene, (1-butyloctyl)-
Phenol, 2,4-bis(1,1-dimethylethyl)-
dl-Limonene
Naphthalene, 1-chloro-
Benzene, (1-pentylheptyl)-
Benzene, (1-methyldecyl)-
Docosane
Tricosane
Benzene, (1-ethynonyl)-
Benzene, (1-propyloctyl)-
Benzene, (1-methylundecyl)-
Tetratriacontane

Compounds tentatively identified :

Naphthalene
Decane, 2-methyl-
Hexadecane, 2,6,10,14-tetramethyl-
Benzene, (1-methylnonyl)-
Eicosane
Undecane, 4,6-dimethyl-

Octane, 6-ethyl-2-methyl-
Benzene, 1-pentylhexyl)-
Benzene, (1-ethyloctyl)-
Nonadecane
Dodecane, 1-iodo-
Hexadecane
Triacontane
(R,R)-3,8-Dimethyldecane
Tetradecane, 1-chloro-
Undecane, 4,7-dimethyl-
Decane
Dodecane
Nonahexacontanoic acid
Decane, 2-methyl-

INORGANIC ANALYTICAL RESULTS

Spolchemie Chemical Plant, Usti nad Labem, Czech Republic, 1997

Sample type: Sediment
Sample name: Bilina sediment downstream (no. 2)
Lab code: MI7006
Sampling date: 24.02.97

Other information: Sediment collected from the Bilina river (left bank, depth 50cm). Approximately 600 meters downstream from the Spolchemie plant discharge.

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/kg)
Manganese	936.5
Chromium	102.5
Zinc	397.0
Copper	103.0
Lead	69.5
Nickel	58.0
Cobalt	25.5
Cadmium	1.0

ORGANIC ANALYTICAL RESULTS

Spolchemie chemical plant,Usti nad Labem, Czech Republic, 1997

Sample type: Sediment
Sample name: Bilina sediment downstream, (no.2)
Lab. code: MI7006
Sampling date: 24.02.97

Other information: Sediment collected from the Bilina river (left bank, depth 50cm). Approximately 600m downstream from the Spolchemie plant discharge.

Analysis method: GC/MS screen

Number of compounds isolated: 42

Compounds identified to better than 90%:

Benzene, hexachloro-
Fluoranthene
dl-Limonene
Naphthalene
Pyrene
o,p'-DDT
Sulfur, mol. (S8)
5-Octadecene, [E]-
Cyclopropane, 1-methyl-2-0cty-, cis/trans
Benzene, (1-butyloctyl)-

Compounds tentatively identified :

p,p'-DDD
Heptadecane
Tetradecene
1-Nonadecene
Benzene, (1-propylnonyl)-
Benzene, (1-pentylheptyl)-
Benzene, (1-pentylhexyl)-

Benzene, (1-propyloctyl)-
Phenanthrene, 1-methyl-7-(1-methylethyl)-
Tricosane
Benzene, (1-pentyloctyl)-
Undecane, 3,9-dimethyl-
Phenanthrene
Tritetracontane
Benzene, (1-butylnonyl)-
Heptadecane, 2,6-dimethyl-
Benzene, (1-butylhexyl)-
Benzene, (1-ethylnonyl)-

INORGANIC ANALYTICAL RESULTS

Spolchemie Chemical Plant, Usti nad Labem, Czech Republic, 1997

Sample type: Sediment
Sample name: Bilina sediment upstream (no. 3)
Lab. code: MI7007
Sampling date: 24.02.97

Other information: Sediment collected from the Bilina river (left bank, depth 1.2m). Approximately 1000 meters upstream from the Spolchemie plant discharge.

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/kg)
Manganese	976.5
Chromium	81.5
Zinc	470.0
Copper	95.5
Lead	51.0
Nickel	69.5
Cobalt	30.5
Cadmium	1.0

ORGANIC ANALYTICAL RESULTS

Spolchemie chemical plant,Usti nad Labem, Czech Republic, 1997

Sample type: Sediment
Sample name: Bilina sediment upstream, (no.3)
Lab. code: MI7007
Sampling date: 24.02.97

Other information: Sediment collected from the Bilina river (left bank, depth 1.2m). Approximately 1000m upstream from the Spolchemie plant discharge.

Analysis method: GC/MS screen

Number of compounds isolated: 23

Compounds identified to better than 90%:

Cyclohexane,1-methyl-4-(1-methylethenyl)-, (R)-
Phenanthrene,2,4,5,7-tetramethyl-
Pentadecane, 2,6,10,14-tetramethyl-

Compounds tentatively identified :

5-Octadecene, [E]-
Tetradecane
Nonadecane
1-Dodecene
Octadecane
Pentacosane
Fluoranthene

INORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC Plant, Neratovice, Czech Republic, 1997

Sample type: Sediment
Sample name: Neratovice sediment upstream left (no. 4)
Lab. code: MI7008
Sampling date: 25.02.97

Other information: Sediment collected from the Labe river (left bank, depth 50cm). 300m above the weir at Neratovice, approximately 1.2km upstream from the Spolana Neratovice PVC plant.

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/kg)
Manganese	2614.6
Chromium	89.3
Zinc	359.2
Copper	95.2
Lead	68.0
Nickel	30.6
Cobalt	13.1
Cadmium	1.5

ORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC plant, Neratovice, Czech Republic, 1997

Sample type: Sediment
Sample name: Neratovice sediment upstream left, (no.4)
Lab. code: MI7008
Sampling date: 25.02.97

Other information: Sediment collected from the Labe river (left bank, depth 50cm). 300m above the weir at Neratovice, approximately 1.2km upstream from Spolana Neratovice PVC plant.

Analysis method: GC/MS screen

Number of compounds isolated: 26

Compounds identified to better than 90%:

1,3-Butadiene, 1,1,2,3,4,4-hexachloro-
Benzene, 1,2-dichloro-
1-Dodecene
Cyclohexene, 1-methyl-4-(methylethyl)-, (R)-
9-Octadecene, (E)-
Sulfur, mol.(S8)
Undecane
Hexacosane

Compounds tentatively identified :

Pyrene
Eicosane
5-Octadecene, (E)
Phenol, 2,4-bis(1,1-dimethylethyl)-
Heneicosane
Fluoranthene
Octachlexene-5-yne

INORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC Plant, Neratovice, Czech Republic, 1997

Sample type: Sediment
Sample name: Neratovice sediment downstream right (no. 5)
Lab. code: MI7009
Sampling date: 25.02.97

Other information: Sediment collected from the Labe river (right bank, depth 1.3m). Approximately 1.5km downstream from the Spolana Neratovice PVC plant.

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/kg)
Manganese	1983.5
Chromium	95.4
Zinc	393.1
Copper	95.4
Lead	65.1
Nickel	32.1
Cobalt	12.8
Cadmium	1.4

ORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC plant, Neratovice, Czech Republic, 1997

Sample type: Sediment
Sample name: Neratovice sediment downstream right, (no.5)
Lab. code: MI7009
Sampling date: 25.02.97

Other information: Sediment collected from the Labe river (right bank, depth 1.3m). Approximately 1.5km downstream from Spolana Neratovice PVC plant.

Analysis method: GC/MS screen

Number of compounds isolated: 25

Compounds identified to better than 90%:

Benzene, 1,2-dichloro-
1-Dodecene
Cyclohexene, 1-methyl-4-(1-methylethenyl), (R)-
Sulfur, mol. (S8)
5-Octadecene, (E)-
Pentacosane
Hexacosane
Heptadecane

Compounds tentatively identified :

1,1'-Biphenyl
Pyrene
Eicosane
Fluoranthrene
9-Octadecene, (E)-
Cyclopentane, propyl-

INORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC Plant, Neratovice, Czech Republic, 1997

Sample type: Sediment
Sample name: Neratovice sediment downstream left (no. 6)
Lab. code: MI6010
Sampling date: 25.02.97

Other information: Sediment collected from the Labe river (left bank, depth 0.8-1.0m). Approximately 2.7km downstream from the Spolana Neratovice PVC plant.

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/kg)
Manganese	1022.2
Chromium	87.7
Zinc	535.9
Copper	163.2
Lead	70.8
Nickel	28.3
Cobalt	10.9
Cadmium	n/d

ORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC plant, Neratovice, Czech Republic, 1997

Sample type: Sediment
Sample name: Neratovice sediment downstream left, (no.6)
Lab. code: MI7010
Sampling date: 25.02.97

Other information: Sediment collected from the Labe river (left bank, depth 0.8-1.0m). Approximately 2.7km downstream from the Spolana Neratovice PVC plant.

Analysis method: GC/MS screen

Number of compounds isolated: 37

Compounds identified to better than 90%:

1-Dodecene
Benzene, 1,4-dichloro-
Cyclohexene, 1-methyl-4-(1-methylethenyl)-, (R)-
9-Octadecene, (E)-
Sulfur, mol. (S8)
Docosane
Undecane, 3,9-dimethyl-

Compounds tentatively identified :

Fluoranthene
Heptadecane, 2,6,10,15-tetramethyl-
Hexadecane
Pyrene
Tetradecane
Undecane, 4,6-dimethyl-
Undecane, 4,7-dimethyl-
Phenol, 2,4-bis((1,1-dimethylethyl)-
Nonahexacontanoic acid
Undecane

INORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC Plant, Neratovice, Czech Republic, 1997

Sample type: Industrial wastewater
Sample name: Discharge water, right side (no. 7)
Lab. code: MI7011
Sampling date: 25.02.97

Other information: Wastewater discharge from Spolana. Collected from the effluent canal prior to confluence with the Labe river.

Analysis method: ICP-AES

METAL	CONCENTRATION (ug/l)
Manganese	170
Chromium	<10
Zinc	300
Copper	30
Lead	<30
Nickel	<10
Cobalt	<10
Cadmium	<10

ORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC plant, Neratovice, Czech Republic, 1997

Sample type: Industrial waste water
Sample name: Discharge water, right side, (no.7)
Lab. code: MI7011
Sampling date: 25.02.97

Other information: Waste water discharge from Spolana. Collected from canal proir to confluence with the Labe river.

Analysis method: GC/MS screen

Number of compounds isolated: 18

Compounds identified to better than 90%:

Benzene, 1,2,4-trichloro-
Sulfur, mol.(S8)
Docosane
Heptadecane

Compounds tentatively identified :

dl-Limonene
Heptadecane, 2,6,10,15-tetramethyl-
Undecane, 3,9-dimethyl-
Eicosane
Tetradecane
Dodecane

INORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC Plant, Neratovice, Czech Republic, 1997

Sample type: Sediment
Sample name: Neratovice upstream right (no. 8)
Lab. code: MI7012
Sampling date: 25.02.97

Other information: Sediment collected from the Labe river (right bank, depth 1.2m). 600m above a weir, approximately 1.5km upstream from the Spolana Neratovice PVC plant.

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/kg)
Manganese	3122.7
Chromium	95.5
Zinc	399.5
Copper	108.6
Lead	78.8
Nickel	33.8
Cobalt	14.7
Cadmium	1.01

ORGANIC ANALYTICAL RESULTS

Spolana Neratovice PVC plant, Neratovice, Czech Republic, 1997

Sample type: Sediment
Sample name: Neratovice upstream right, No.8
Lab. code: MI7012
Sampling date: 25.02.97

Other information: Sediment collected from the Labe river (right bank, depth 1.2m). 600m above the weir, approximately 1.5km upstream from the Spolana Neratovice PVC plant.

Analysis method: GC/MS screen

Number of compounds isolated: 19

Compounds identified to better than 90%:

dl-Limonene
Benzene, 1,4-dichloro-
Sulfur, mol. (S8)
Cyclopropane, 1-methyl-2-octyl-, cis/trans
9-Octadecene, (E)-
5-Octadecene, (E)-

Compounds tentatively identified :

Benzene, 1,1'-oxybis-
(E)-6-Chloro-4-methyl-2-heptene
Pyrene
Fluoranthene

INORGANIC ANALYTICAL RESULTS

Synthesia Chemical Plant, Pardubice, Czech Republic, 1997

Sample type: Industrial wastewater
Sample name: Discharge water 1 Synthesia (no. 9)
Lab. code: MI7013
Sampling date: 25.02.97

Other information: Untreated wastewater discharge from Synthesia. Collected from an effluent canal prior to confluence with the Labe river.

Analysis method: ICP-AES

METAL	CONCENTRATION (ug/l)
Manganese	380
Chromium	620
Zinc	1240
Copper	570
Lead	300
Nickel	20
Cobalt	<10
Cadmium	<10

ORGANIC ANALYTICAL RESULTS

Synthesia Chemical plant, Pardubice, Czech Republic, 1997

Sample type: Industrial waste water
Sample name: Discharge water 1 Synthesia, (no.9)
Lab. code: MI7013
Sampling date: 25.02.97

Other information: Untreated waste water discharge from Synthesia. Collected from an effluent canal prior to confluence with the Labe river.

Analysis method: GC/MS screen

Number of compounds isolated: 58

Compounds identified to better than 90%:

Benzene, 1,3,5-trichloro-
Benzene, 1,2-dichloro-
Benzene, 1,4-dichloro-
Benzene, 1,2,4-trichloro-
Benzene, 2,4-dichloro-1-methyl-
2,3-Dichlorotoluene
Benzene, nitro-
1,1'-Biphenyl
1H-Indene, 1-methylene-
Benzenemethanamine, N-phenyl-
Benzocycloheptatriene
Diazene, diphenyl-
Undecane
Diazene, diphenyl-
9H-Carbazole, 9-ethyl-
Octadecane
Triacontane

Compounds tentatively identified :

dl-Limonene
Benzeneamine, 4-ethyl-

Octadecane
Benzene, 1-chloro-4-(chloromethyl)-
Docosane
Tetratriacontane
Undecane, 4,7-dimethyl-
Undecane, 2,7-dimethyl-
Octane, 5-ethyl-2-methyl-
Benzenamine, N-sulfinyl-
Hexadecane, 2,6,10,14-tetramethyl-
Tetratriacontane
Tricosane
Docosane, 11-decyl-
Octadecane
1,3-Propanediamine, N,N'-bis(3-aminopropyl)-
Aniline, N-butyl-N-methyl-

INORGANIC ANALYTICAL RESULTS

Synthesia Chemical Plant, Pardubice, Czech Republic, 1997

Sample type: Sediment
Sample name: Synthesia sediment downstream (no. 10)
Lab. code: MI7014
Sampling date: 25.02.97

Other information: Sediment collected form the Labe river (right bank, depth 2.2m). Approximately 600m downstream from the Synthesia Chemical Plant discharge.

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/kg)
Manganese	271.2
Chromium	112.3
Zinc	370.3
Copper	98.1
Lead	68.4
Nickel	30.7
Cobalt	10.9
Cadmium	1.4

ORGANIC ANALYTICAL RESULTS

Synthesia Chemical plant, Pardubice, Czech Republic, 1997

Sample type: Sediment
Sample name: Synthesia sediment downstream, (no.10)
Lab. code: MI7014
Sampling date: 25.02.97

Other information: Sample collected from the Labe river (right bank, depth 2.2m). Approximately 600m downstream from Synthesia Chemical plant discharge.

Analysis method: GC/MS screen

Number of compounds isolated: 24

Compounds identified to better than 90%:

1,1'-Biphenyl
Sulfur, mol. (S8)
Benzene, 1,3-dichloro-
Naphthalene
1-Decanol
Docosane

Compounds tentatively identified :

1-Hexadecene
Hexatriacontane
5-Octadecene, (E)-
dl-Limonene
1,1'-Biphenyl, 2-methyl-
Pentadecane, 2,6,10-trimethyl-
Tritetracontane
Hexathiepane

INORGANIC ANALYTICAL RESULTS

Synthesia Chemical Plant, Pardubice, Czech Republic, 1997

Sample type: Industrial waste water
Sample name: Water from retention basin (no. 11)
Lab. code: MI7015
Sampling date: 25.02.97

Other information: Wastewater collected from a retention basin. Effluent from the basin is discharged into a waste canal which leads to the Labe river.

Analysis method: ICP-AES

METAL	CONCENTRATION (ug/l)
Manganese	590
Chromium	1120
Zinc	2130
Copper	980
Lead	520
Nickel	40
Cobalt	30
Cadmium	<10

ORGANIC ANALYTICAL RESULTS

Synthesia Chemical plant, Pardubice, Czech Republic, 1997

Sample type: Industrial waste water
Sample name: Water from retention basin, (no.11)
Lab. code: MI7015
Sampling date: 25.02.97

Other information: Waste water collected from a retention basin. Effluent from the basin is discharged into a waste canal which leads to the Labe river.

Analysis method: GC/MS screen

Number of compounds isolated: 43

Compounds identified to better than 90%:

Benzene, 1,2,3-trichloro-
Benzene, 1,2,4-trichloro-
Benzene, 1,2-dichloro-
Benzene, 1,3-dichloro-
Benzene, 2,4-dichloro-1-methyl-
2,3-Dichlorotoluene
1,1'-Biphenyl, 2-methyl-
Benzene, 1,1'-thiobis-
Benzenemethanamine, N-phenyl-
Diazene, diphenyl-
Benzenamine, N-(phenylmethylene)-
Sulfur, mol.(S8)
Naphthalene, 2-methyl-
Benzo[b]thiophe
Naphthalene
1,1'-Biphenyl
Diazene, diphenyl-
Benzene, 1-chloro-2-chloromethyl-

Compounds tentatively identified :

dl-Limonene
Benzeneamine, 4-ethyl-
Tricosane
Undecane, 5,7-dimethyl-
Benzene, nitro-
Acenaphthylene, 1,2-dihydro-
Tridecane
Benzenamine, N-sulfinyl-

INORGANIC ANALYTICAL RESULTS

Synthesia Chemical Plant, Pardubice, Czech Republic, 1997

Sample type: Industrial wastewater
Sample name: Discharge water 2 from Synthesia (no. 12)
Lab. code: MI7016
Sampling date: 25.02.97

Other information: Wastewater discharge from Synthesia. Collected from the effluent canal (no. 2) which receives waste effluent from the plant as well as treated sewage.

Analysis method: ICP-AES

METAL	CONCENTRATION (ug/l)
Manganese	320
Chromium	30
Zinc	220
Copper	90
Lead	<30
Nickel	<10
Cobalt	<10
Cadmium	<10

ORGANIC ANALYTICAL RESULTS

Synthesia Chemical plant, Pardubice, Czech Republic, 1997

Sample type: Industrial waste water
Sample name: Discharge water 2 from Synthesia, (no.12)
Lab. code: MI7016
Sampling date: 25.02.97

Other information: Waste water discharge from Synthesia. Collected from the effluent canal (no.2) which receives waste effluent from the plant as well as treated sewage.

Analysis method: GC/MS screen

Number of compounds isolated: 31

Compounds identified to better than 90%:

Benzene, 1,2,4-trichloro-
Benzene, 1,2-dichloro-
Benzene, 1,3-dichloro-
Benzene, 1,4-dichloro-
Benzofuran, 2-methyl-
Naphthalene
dl-Limonene

Compounds tentatively identified :

1,1'-Biphenyl
1H-Indene, 2,3-methyl-dihydro-1-methyl-

INORGANIC ANALYTICAL RESULTS

Synthesia Chemical Plant, Pardubice, Czech Republic, 1997

Sample type: Sediment
Sample name: Sediment Synthesia upstream (no 13)
Lab. code: MI7017
Sampling date: 25.02.97

Other information: Sediment collected from the Labe river (right bank, depth 1m). 500m above a weir, approximately 1.2km upstream from the Synthesia discharge canal (no. 2).

Analysis method: ICP-AES

METAL	CONCENTRATION (mg/kg)
Manganese	441.2
Chromium	72.7
Zinc	299.1
Copper	53.7
Lead	48.6
Nickel	29.6
Cobalt	11.6
Cadmium	n/d

ORGANIC ANALYTICAL RESULTS

Synthesia Chemical plant, Pardubice, Czech Republic, 1997

Sample type: Sediment
Sample name: Sediment Synthesia upstream, (no.13)
Lab. code: MI7017
Sampling date: 25.02.97

Other information: Sample collected from the Labe river (right bank, depth 1m). 500m above the weir, approximately 1.2km upstream from Synthesia discharge canal (no.2).

Analysis method: GC/MS screen

Number of compounds isolated: 31

Compounds identified to better than 90%:

1-Dodecene
dl-Limonene
5-Octadedene, (E)-
Docosane
Benzene, 1,3-dichloro-
Sulfur, mol. (S8)

Compounds tentatively identified :

Hexadecane
Heptadecene
Heneicosane
Tritetracontane
1-Tetradecene
Heneicosane
Decane, 2-methyl-
Pentadecane