Petkim: Organic Analytical Report 2: Analysis of wastes generated by integrated chemical production processes, including the manufacture of VCM and chlorinated organic solvents, at the Petkim chemical complex, Aliaga, Turkey.

Introduction

In addition to the samples of sediments and seawater taken adjacent to the two principal discharge points from the Petkim chemical complex (see "Petkim: Organic Analytical Report 1"), two samples were obtained of wastes arising from the manufacture of vinyl chloride monomer (VCM) and other organic chemicals:-

MI7019 - sludge from canal carrying waste from the manufacture of VCM, ACN and aromatic compounds to the combined treatment plant (prior to treatment, dewatering and incineration);

MI7020 - solid residue (ash) from incinerator, at point at which residues wash in to the sea.

Materials and Methods

Both samples were collected on 4th March 1997. The first sample, MI7019, was taken by an independent sampler and supplied to Greenpeace for analysis. Both samples were returned to the Greenpeace Research Laboratories, University of Exeter, UK, and analysed as outlined in the previous report ("Petkim: Organic Analytical Report 1"). Further details can be provided on request.

Results and Discussion

Results, in the form of lists of compounds identified in each sample, are included as Appendix 1. Both samples were found to contain a very wide range of organic contaminants. The organic screen technique employed is non-quantitative, but the degree of dilution required in order to be able to analyse the samples indicated that the content of organic matter was extremely high, particularly in sample MI7019 (sludge prior to incineration).

A total of 255 organic compounds were isolated from MI7019. A copy of the analytical chromatography trace is included in Appendix 2 to give an indication of the complexity of the waste. Of the 255 isolated, 64 compounds were identified with a reliability greater than 90%, the majority of which (39) were organohalogens (organic compounds of chlorine or bromine). Included among the 37 organochlorine compounds identified in the sample were chlorinated biphenyls (including the PCB 2,2',3,3',4,4',5,5',6-nonachloro-biphenyl), pentachloropropene, tetra-, penta- and hexachlorobutadiene (HCBD), hexachlorobenzene (HCB), hexachloroethane (HCE), chloromethyl diphenyl methane and a range of chlorinated benzene, toluene and phenol derivatives. The majority of these chemicals are highly toxic, persistent in the environment and have a tendency to accumulate in biological tissues because of their high fat solubility. This therefore represents an extremely hazardous mixture of chemicals, the complexity of which renders the assessment of overall toxicity practically impossible (Johnston *et al.* 1996). Almost all of the organochlorine chemicals identified are acutely toxic to animals and humans when present alone, and many can cause severe damage to the liver, kidney and other organs in animals exposed even to relatively low concentrations (Merck 1989). For example, HCBD is a potent kidney toxin in laboratory animals (Werner *et al.* 1995, Birner *et al.* 1995). HCB also damages the liver and kidney, as well as interfering with bone development, blood biochemistry, immune response and the nervous system (Thomas 1990, USPHS 1994). Both HCBD and HCB, along with 1,2-dichloro-2-butene, are confirmed carcinogens in animals (USPHS 1992, Newhook and Meek 1994, Sittig 1994). A number of the organochlorines identified are known to be potent developmental toxins and several, including HCB and PCBs, have been identified as having the potential to interfere with the endocrine system in animals (Colborn *et al.* 1993). Allsopp *et al.* (1995) gives a useful overview of the effects of organochlorines on human health.

For the majority of compounds identified, very little or no toxicological information exists. Note also that many of the compounds isolated from the sample simply could not be identified. Nevertheless, it is clear from the discussion above that the waste material is extremely hazardous. The wide range of chlorinated organic compounds which could be identified is typical of oxychlorination wastes arising from the PVC industry (Costner *et al.* 1994). In addition, the presence of HCB, HCBD and chlorinated biphenyls suggests that the waste may also contain significant quantities of dioxins (polychlorinated dibenzo-p-dioxins and furans), among the most toxic compounds known to man. This is investigated further below. The incineration of this waste would undoubtedly lead to the release to the atmosphere or accumulation in the ash of dioxins and other organic compounds, either carried through from the waste itself or generated by reactions occurring within the incinerator.

The second sample, residues from the incinerator (MI7020), was also found to contain a complex mixture of organic compounds. Of 157 compounds isolated, only 69 could be reliably identified (match quality greater than 90%), and these were dominated by polycyclic aromatic hydrocarbons (PAHs), including derivatives of anthracene, fluorene, naphthalene, indene and biphenyl. These toxic compounds are commonly found as products of incomplete combustion. No organochlorine compounds were identified in this sample. Nevertheless, this does not rule out the possible presence of dioxins and other persistent organochlorines at concentrations which, though significant, were below the limits of detection of the organic screening method employed. Unfortunately, it was not possible to forward this sample for dioxin analysis.

The precise origin of the ashes sampled, in terms of the batch of incinerated waste from which they arose, is not known. It is possible that the ashes sampled resulted from combustion of waste other than that arising from the oxychlorination process. Nevertheless, the positioning of ashes contaminated with such a range of organic compounds such that they can be washed in to the sea must be considered an unacceptable practice.

Supplementary Investigation: Analysis of VCM residue sludge from waste canal for dioxins and furans.

The manufacture of vinyl chloride monomer (VCM), the building block for PVC, involves a number of chemical reaction steps, including the synthesis of ethylene dichloride (EDC) in a reaction termed oxychlorination. The EDC is then converted to VCM. At the Petkim plant, as in many other PVC plants, the residues from the oxychlorination reaction, which contain a complex mixture of organochlorine compounds, are then used as feedstock for a second oxychlorination process, used in the manufacture of the chlorinated solvents trichloroethylene (TRI) and perchloroethylene (PER). This process also produces wastes which are heavily contaminated with chlorinated organic compounds (see sample MI7019 above). It is widely recognised that the residues of such oxychlorination reactions contain substantial concentrations of polychlorinated dioxins and furans (PCDD/Fs), commonly termed dioxins (Evers *et al.* 1989, EA 1997). These highly toxic substances are generated as unwanted by products of the processes used in the manufacture of VCM and chlorinated solvents. Indeed, ICI (1994) have stated that:-

"It is difficult to see how any of [the oxychlorination] conditions could be modified so as to prevent PCCD/PCDF formation without seriously impairing the reaction for which the process is designed".

As reported above, sample MI7019 (sludge from the canal carrying waste from manufacture of VCM, ACN and mixed aromatics to the waste treatment plant at Petkim) was found to be heavily contaminated with a wide range of chlorinated organic compounds, including several which are useful indicators of the presence of dioxins. In order to determine the presence and concentrations of dioxins in this sludge, a further sample (MI7047) was taken from the same location as MI7019 on 30th May 1997. This sample was forwarded to an accredited laboratory in the UK for analysis of dioxins and furans. Details of the method employed in the analysis, and relevant quality assurance and quality control data, can be provided on request.

The sample contained 56.94ug/kg of dioxins and furans, expressed as International Toxic Equivalents (ITEQ) and reported on a dry weight basis (full results in Appendix 3). In other words, the total concentration of all the dioxin and furan congeners present, although dominated by octachlorofuran (OCDF, see below), would have a toxicity equivalent to 56.94 ug/kg of tetrachlorodibenzodioxin (TCDD), the most toxic of the dioxin congeners. Concentrations reported by ICI (1994) for wastes of a similar nature (heavy ends from VCM product distillation) from the VCM plant at Runcorn (UK) were in the range 3.10-7.56 ug/kg ITEQ (average 5.60 ug/kg). Similarly, Stringer *et al.* (1995) reported 3.19 ug/kg ITEQ for a sample of VCM distillation residues from the US PVC industry. The dioxin concentration in the waste from Petkim, at 56.94 ug/kg ITEQ, is therefore approximately 10 times greater than published values for similar waste from other PVC plants.

The congener profile (i.e. a breakdown of the different forms, or congeners, of the dioxins and furans present - see Appendix 2) reveals that the Petkim waste contains a large proportion of highly chlorinated dibenzofurans; 18.1% heptachlorofurans and 75% octachlorofuran. This is typical of process wastes from oxychlorination reactions employed in the manufacture of VCM, perchloroethylene (PER) and trichloroethylene (TRI). For example, the profile compares well with those for samples analysed by ICI (1994), arising from the manufacture of EDC, VCM, TRI and PER.

Dioxins are among the most hazardous chemicals known to man. Tetrachlorodibenzodioxin (TCDD) is a confirmed animal carcinogen and a suspected carcinogen in humans; it is likely that other congeners are also carcinogens. Exposure to dioxins has been linked to a number of acute and chronic toxic effects in animals and humans, particularly where exposure occurs during sensitive stages of development (see Petersen *et al.* 1993 for review). For example, studies of children born to mothers exposed to high levels of dioxin at Times Beach, Missouri (US) have revealed increased incidences of neurological dysfunction, changes in brain development and persistent changes to the immune system (Cantor *et al.* 1993, Smoger *et al.* 1993). More recently, Egeland *et al.* (1994) reported a significant relationship between dioxin exposure and levels of reproductive hormones in men.

Summary

A sample of sludge waste arising from the manufacture of VCM and other organic compounds was found to contain more than 250 organic compounds, many of which were toxic, persistent and bioaccumulative organochlorines. The organochlorines identified were typical of wastes from oxychlorination processes used in the manufacture of VCM, PER and TRI and were indicative of the presence of substantial quantities of dioxins. This was confirmed through analysis of a second sample of the same waste, revealing that total dioxin levels (as toxic equivalents) in the Petkim waste were approximately 10 times higher than levels reported for similar wastes from the PVC industry in UK and the USA.

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ORGANIC ANALYTICAL RESULTS

Petkim Aliaga Incinerator, Turkey, 1997.

Sample type: Solid waste
Sample name: 2
Sampling date: 19.03.1997
Lab. code: MI7019

Other information: Sludge collected from VCM channel leading to Petkim Incinerator.

Analysis method: GC/MS screen

Number of compounds isolated: 255

Compounds identified to better than 90%:

(5-Methylcyclopent-1-enyl)methanol
1,1'-Biphenyl"
1,1'-Biphenyl, 2,2',3,3',4,4',5,5',6-nonachloro-"
1,1'-Biphenyl, 4-chloro-"
1,1,2,3,3-Pentachloro-1-propene"
1,3-Butadiene, 1,1,2,3,4,4-hexachloro-"
1,3-Butadiene, 1,1,3,4-tetrachloro-"
1,3-Butadiene, pentachloro-"
1,3-Cyclopentadiene, 1,2,3,4,5,5-hexachloro-"
1-(Ethyl-1-D)-2-pyridone
1-Butene, 1,4-dichloro-"
1-Butyne, 3-chloro-"
1-Cyclohexene-1-methanol
1-Methoxy-4-methylbenzotriazole
1-Propene, 3,3,3-trichloro-2-methyl-"
1-Propene, pentachloro-"
1H-Purine-2,6-dione, 3,7-dihydro-1,3-dimethyl-"
2-Butene, 1,4-dichloro-"
2-Butenedioic acid, 2,3-dibromo-, (Z)-"
2-Furaldehyde, O-methyloxime"
2-Hexenal
2-Hexenoic acid, 6-phenyl-, ethyl ester"
2H-1-Benzothiopyran, octahydro-, trans-"
3-Decen-2-one

3-Undecen-5-yne, (Z)-"
9H-Fluorene
Anthracene, 9-methyl-"
Azulene
Benzaldehyde
Benzene, (2-chloroethenyl)-"
Benzene, (2-chloroethyl)-"
Benzene, 1,2-dichloro-"
Benzene, 1,4-dichloro-"
Benzene, 1-chloro-2-(1-chloroethyl)-"
Benzene, 1-chloro-2-ethyl-"
Benzene, 1-chloro-2-methyl-"
Benzene, 1-chloro-3-ethyl-"
Benzene, 1-chloro-4-ethyl-"
Benzene, chloro(2-chloroethyl)-"
Benzene, hexachloro-"
Benzene, pentachloro(trichloroethenyl)-"
Benzene, pentachloro-"
Benzenemethanol, 4-chloro-3-nitro-"
Benzocycloheptatriene
Bicyclo(3.2.1)octan-2-one-exo-4-D
Butane, 1,2,3,4-tetrachloro-"
Chloromethyldiphenylmethane
Docosane
Ethane, hexachloro-"
Ethane, pentachloro-"
Heptadecane
Hexatriacontane
N-(3'-Chlorophenyl)-2-hydroxyimino acetamide
Naphthalene, 1,4-dibromo-"
Octachloropentafulvalence
Octadecane
Phenanthrene
Phenol, 4-amino-2,6-dichloro-"
Phenol, 4-chloro-5-methyl-2-nitro-"
Propane, 2-bromo-1-chloro-"
Propane, 3-chloro-1,1,1-trifluoro-"
Sulfone, chloro phenyl"
trans-1,3-Cyclohexandicarboxamide"
Tricosane

Compounds tentatively identified :

[1,1'-Biphenyl]-3-ol, dichloro-
1,1,1,2,3,3,3-Heptachloropropane
1,1,2,4-Tetrachlorobut-1-ene
1,1-Dichloro-1-nitropropane
1,3,5-Triazine-2,4-diamine, 6-chloro-N-ethyl-

1,3-Bromochlorocyclobutane
1,3-Butadiene, 2-chloro-, dimer
1,3-Cyclopentadiene, 5,5-dimethyl-
1,5-Cyclooctadiene, 1,6-dichloro-
1-Butene, 1,1,3,3,4,4,-hexachloro-
1-Butene, 3,4-dichloro-
1-Chloro-2-(2-chloropropyl)-benzene
1-Propene, 1,1,2,3,3,3-hexachloro-
1-Propene, 1,1,3-trichloro-2-methyl-
1-Propene, 1,2,3,3-tetrachloro-
1-Propene, 1,2,3-trichloro-
1-Propene, 1,3-dichloro-
1-Propene, 2,3-dichloro-
2,3,4,6-Tetrabromo-5-methylbenzoic acid
2,6-Dichloro-1-hexen-4-yne
2-Amino-5-chlorothiazole
2-Amino-8-methylimidazo[1,5-a]-1,3,5-triazin-4(3H)-one
2-Butene, 1,4-dichloro-, (E)-
2-Butene, 1-bromo-2-chloro-
2-Chloropyrazine
2-Hexen-4-yne
2-Pyrrolidinone, 1-methyl-
3,4,5-Tribromo-2,6-dichlorobenzoic acid
3,4-Dichloro-3-methylbutene
3,6-Dibromo-1,2,4-triazine 1-oxide
3-Bromo-1,2,4-triazine 2-oxide
3-Methyl-2-(1',1',5'-trimethyl-5'-hexenyl)-2-cyclopropenyl methyl ketone
4,4-Dimethyltricyclo(3,2,1.0)octan-6-one
4,5-Dichloropicolinonitrile
4-(2',2'-Dimethyl-6'-methylidenecyclohexyl)-2-methyl-3-buten-2-ol
4-Bromo-1-ethylpyrazole
4-Chloro-2-phenylaniline
6-Chloro-5H-benz0-cycloheptene
7,7-Dichlorotetracyclo[4.1.0.0(2,4.0(3,5)]heptane
8-Decenoic acid, 5-ethenyl-3,5,9-trimethyl-, methyl ester
9-Ethylfluorene
9H-Fluorene
A,A,A,A',A',A',-Hexachloro-m-xylene
Anthracece, 1-chloro-
Benzamine, 2-chloro-
Benzene, (1,2-dichloroethyl)-
Benzene, (chloromethyl)-
Benzene, (dibromomethyl)-
Benzene, (nitromethyl)-
Benzene, 1,1'-(1-methyl-1,2-ethanediyl)bis-
Benzene, 1,1'-(3-methyl-1-propene)-
Benzene, 1,1'-(3-methyl-1-propene-1,3-diyl)bis-
Benzene, 1,2,4,5-tetrachloro-
Benzene, 1,4-dichloro-2,5(trichloromethyl)-

Benzene, 1,4-dichloro-2-ethenyl-
Benzene, 1-(bromomethyl)-3-chloro-
Benzene, 1-chloro-2-propyl-
Benzene, 1-chloro-3-ethenyl-
Benzene, 4-chloro-1,2-dimethyl-
Benzene, chloro(chloromethyl)(1-methylethenyl)-
Benzene, chloro(chloromethyl)(1-methylethyl)-
bis-(3,3-Dichloropropyl) ester
Bornane-2,6-dione
Butane, 1,2,2,4-tetrachloro-
Carbonothioic dichloride
cis-2,3-Dichlorospiro(cyclopropane-1,9'-(9H)fluorene
Cyclobutane, 1-bromo-3-chloro-
Cyclopentane, 1,1,3-trimethyl-
Cyclopentene, 1-chloro-
Cyclopentene, octachloro-
Cyclopropane, 1,1-dichloro-2-ethenyl-
Cyclopropane, 1,1-dichloro-2-propenyl-, cis+trans
Cyclopropane, pentachloro-
Cyclotriacontane
Ethene, trichloro-
Fluorene, 9-chloro-
Heptachloronorbornene (tentative from field samples)
Hexa-2,4-diyne-1,6-diol
Hexachloro-m-xylene
iso-Epoxy-estafiatin
Naphthalene, 1-chloro-
Nonahexacontanoic acid
Phenethylamine, p-chloro-N-(p-chlorobenzyl)-, hydrochloride
Phenol, 2,4-dichloro-6-methyl-
Phenol, 4-amino-3,5-dichloro-
Propane, 1,1,1,3-tetrachloro-
Propane, 1,1,2,2-tetrachloro-
Propanoyl chloride, 2,2-dichloro-
Propene, pentachloro-
Pyrimidine, 2-chloro-
Tetratriacontane
trans-1,2-Cyclobutanedicarbonyl chloride
trans-1-Chloro-2-m-chlorophenylcyclopropane
Tritetracontane

ORGANIC ANALYTICAL RESULTS

Petkim Aliaga Incinerator, Turkey, 1997.

Sample type: Solid waste
Sample name: 3
Sampling date: 19.03.1997
Lab. code: MI7020

Other information: Solid waste resulting from incinerator process.

Analysis method: GC/MS screen

Number of compounds isolated: 157

Compounds identified to better than 90%:

(+-)-3,3-Dimethyltricyclo[5.4.0.0(2,9)]undecan-8-one
1,1'-Biphenyl
1,1'-Biphenyl, 2,2'-dimethyl-
1,1'-Biphenyl, 2-ethyl-
1,1'-Biphenyl, 4-methyl-
1,1':3',1''-Terphenyl
1,4-Ethenoanthracene, 1,4-dihydro-
1H-Cyclopropa[b]naphthalene, 1a,2,7,7a-tetrahydro-
1H-Indene
1H-Indene, 1,1-dimethyl-
1H-Indene, 1-methyl-
1H-Indene, 1-methylene-
1R-Methyl-2T-phenylcyclopropane
2,8-Dimethyldibenzothiophene
4-Hexenoic acid, 3-methyl-2,6-dioxo-
9H-Fluorene
9H-Fluorene, 1-methyl-
9H-Fluorene, 2,3-dimethyl-
9H-Fluorene, 2-methyl-
Acenaphthylene
Acenaphthylene, 1,2-dihydro-
Anthracene
Anthracene, 2-methyl-
Benzene, 1,1'-methylenebis-
Benzene, 1,2,3-trimethyl-

Benzene, 1-ethenyl-4-ethyl-
Benzene, 1-methyl-2-(2-propenyl)-
Benzene, 1-methyl-4-propyl-
Benzene, 2-ethenyl-1,3,5-trimethyl-
Benzene, 2-ethenyl-1,3-dimethyl-
Benzene, 2-ethenyl-1,4-dimethyl-
Benzene, 4-ethyl-1,2-dimethyl-
Benzene, cyclopropyl-
Benzene, ethenylmethyl-
Chrysene
Dibenzocycloheptadiene
Dibenzothiophene, 3-methyl-
Docosane
Dodecane, 2,6,10-trimethyl-
Eicosane
Fluoranthene
Hahnfett
Heneicosane
Hexacosane
Hexadecane, 2,6,10,14-tetramethyl-
Naphthalene, 1,2-dihydro-
Naphthalene, 1,2-dihydro-4-phenyl-
Naphthalene, 1,2-dihydro-6-methyl-
Naphthalene, 1,3-dimethyl-
Naphthalene, 1,6,7-trimethyl-
Naphthalene, 1-methyl-
Naphthalene, 2,3,6-trimethyl-
Naphthalene, 2,3-dimethyl-
Naphthalene, 2,7-dimethyl-
Naphthalene, 2-ethenyl-
Naphthalene, 2-ethyl-
Nonadecane
Octacosane
Octadecane
Pentacosane
Phenanthrene
Phenanthrene, 2,3,5-trimethyl-
Phenanthrene, 2,5-dimethyl-
Phenanthrene, 3-methyl-
Phenanthrene, 9,10-dihydro-1-methyl-
Pyrene
Tetracosane
Tetradecane
Tricosane
Triphenylene

Compounds tentatively identified :

(E)-4-Methyl-1-phenyl-1,3-pentadiene
[1,1'-Biphenyl]-2-ol, 5-(1,1-dimethylethyl)-
[8]paracyclophane-2,4-diene
1,1'-Biphenyl, 2-methyl-
1,1'-Biphenyl, 4,4'-dimethyl-
1,1':2',1"-Terphenyl
1,1,3-Trimethylindene
11H-Benzo[b]fluorene
11H-Cyclohepta[a]naphthalen-11-one, 9,10-dihydro-9,9-dimethyl-
1H-Indene, 1-ethylidene-
1H-Indene, 2,3-dihydro-
4H-Cyclopenta[def]phenanthrene
5-Ethylindan
7-exo-Phenyl-2,3-benzonorcaradiene
9,10-Dihydro-2-methyl-anthracene
Azulene, 7-ethyl-1,4-dimethyl-
Benzene, (2-methyl-1-butenyl)-
Benzene, [1-(2,4-cyclopentadien-1-ylidene)ethyl]-
Benzene, 1,1'-(1,2-ethenediyl)bis-
Benzene, 1,1'-[(methylthio)ethenylidene]bis-
Benzene, 1,2-propadienyl-
Benzene, 1,3-diethenyl-
Benzene, 1,5-cyclohexadien-1-yl-
Benzene, 1-methyl-2-(1-methylethyl)-
Benzene, 1-methyl-3-(1-methylethyl)-
Bi-2,4,6-cycloheptatrien-1-yl
Chrysene, 5-methyl-
Cyclohexane, 1-(1,5-dimethylhexyl)-4-(4-methylpentyl)-
Cyclotetradecane, 1,7,11-trimethyl-4-(1-methylethyl)-
Dibenzothiophene
Dodecane
Eicosane, 9-cyclohexyl-
Fluorene, 2,4a-dihydro-
Heptadecane
Heptadecane, 3-methyl-
Heptane, 2,2,4,6,6-pentamethyl-
Indene, 2-methyl-3-phenyl-
Indenoindene
Naphthalene, 1,4,5-trimethyl-
Naphthalene, 2,6-dimethyl-
Naphthalene, 2-phenyl-
Oleic acid, propyl ester
Pyrene, 1,3-dimethyl-
Pyrene, 1-methyl-
Tridecane
Tridecane, 3-methyl-
Trimethylgermylcyclopentadiene