

SIGNIFICANCE AND REMOVAL OF ORGANOCHLORINE
MICROPOLLUTANTS IN PRIMARY SEDIMENTATION

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by

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Significance and removal of organochlorine
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ABSTRACT

Literature pertaining to the sources, occurrence and analysis of polychlorinated biphenyls (PCB) and organochlorine insecticides (OCL) in waste waters and waste water sludges has been reviewed, with emphasis on the behaviour and removal of these compounds in waste water treatment processes.

Elementary analytical techniques have been developed and modified for the determination of PCB and OCL in waste water and waste water sludge samples and these procedures have been utilised in a survey of the concentrations of PCB, γ -HCH, aldrin, dieldrin and endrin in a large number of sewage sludges, sampled in the 'as disposed' form, from UK sewage treatment works. In addition, monthly samples of sludge were taken from three selected sewage treatment works over an eighteen month period in order to investigate temporal variations in the concentrations of these micropollutants in sewage sludges. PCB, γ -HCH and dieldrin were detected most frequently and at the highest concentrations, whilst aldrin and endrin occurred infrequently and displayed concentrations close to the analytical detection limit. The data have been compared with the results of similar studies in the UK and other countries and the implications of the presence of PCB and OCL in sludges which are disposed to the environment are discussed.

Advanced analytical techniques, comprising capillary column gas chromatography and capillary column gas chromatography-mass spectrometry have been established and have been compared with packed column gas chromatography in the analysis of a subsample of UK sewage sludges for PCB and OCL. Lower concentrations of determinands were detected by the two advanced analytical techniques than by the elementary packed column technique, owing to the superior separation and resolving power of the capillary column and the improved specificity of mass spectrometric detection. It is concluded that capillary column gas chromatography was the optimum analytical procedure.

In order to elucidate factors which may influence the behaviour of PCB and OCL in waste water, a series of jar tests have been carried out. Concentrations of solids, surfactants, grease and determinands in raw sewage were varied during these experiments in an attempt to investigate effects upon the partitioning of PCB and OCL between solid and liquid phases. It was observed that significant proportions of the determinands were associated with solids and that this association was independent of the presence of varying concentrations of solids, surfactants, grease and determinands.

The removal of PCB and OCL during primary sedimentation of raw sewage has been studied using a pilot plant facility. A matrix of sixteen experiments was performed, where the pilot plant was operated at four different hydraulic loadings, with four different influent suspended solids loadings. Significant removals of PCB, γ -HCH, aldrin, dieldrin and endrin into the primary sludge were observed, although no particular relationship with the removals of suspended solids was evident. It was concluded that a proportion of the PCB and OCL were both associated with non-settleable solids and were also present in the soluble form in raw sewage.

The significance of the behaviour of PCB and OCL in waste water treatment processes and the hydrological cycle have been discussed, in the particular context of water re-use operations.

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1. INTRODUCTION

During the last two decades, the occurrence in the environment of persistent organochlorine micropollutants, such as Polychlorinated Biphenyls (PCB) and Organochlorine Insecticides (OCL) has been widely reported (Peakall and Lincer, 1970; Nisbet and Sarofim, 1972; Edwards, 1973; Peakall, 1975; Moriarty, 1975). Residues of these chemical compounds have been detected in raw (Croll, 1969), potable (Croll, 1968) and waste waters (Schmidt et al., 1971) and in soils (Edwards, 1970), sediments (Hiller and Singh, 1976), aquatic and terrestrial organisms (Ernst et al., 1976; Zitko, 1976), ambient air (Singmaster and Crosby, 1976) and human fatty tissue (Abbott et al., 1972).

Although the production and usage of PCB and certain OCL has been severely limited in some developed countries, residues of the more persistent compounds remain in the environment as a legacy of their past use. Of the large number of insecticidal compounds produced, γ -HCH, Aldrin, Dieldrin, Endrin and components of the DDT group have been those most frequently detected in environmental samples (Babish et al., 1981), although residues of DDT and its analogues have declined in recent years as a result of legislative proscription of their use (United States Environmental Protection Agency, 1971). PCB formulations comprising mixtures of isomers which may contain from one to ten chlorine atoms in the molecule have been used in the past in such industrial applications as hydraulic, dielectric and thermal fluids (Kleinert, 1976), investment casting waxes (Hesse, 1976), carbonless copy paper (Fishbein, 1973) and capacitor manufacture (United States Environmental Protection Agency, 1975). Current usage of these compounds has been limited to 'closed-circuit' applications where loss to the environment will be negligible (Ballschmiter, 1979). Of the 209 possible PCB isomers, it is evident that the more highly chlorinated species exhibit the greater environmental stability and persistence and therefore PCB formulations containing these isomers have been most frequently detected in environmental samples (Zell et al., 1978).

Owing to the lipophilic nature of PCB and OCL compounds, bioconcentration (or biomagnification) of environmental residues through trophic levels in both aquatic and terrestrial food chains has been shown to occur (Gustafson, 1970), where organisms at the highest trophic level exhibit relatively high concentrations of persistent organochlorine residues in their fatty tissues. The risks to public health from the consumption of contaminated foodstuffs are relatively insignificant since few natural food chains are of importance to the human diet.

Although both PCB and OCL possess low water solubility, a major transport pathway in the environment is the hydrological cycle, where these hydrophobic compounds may be strongly adsorbed onto fine particles and sediments (Bedding et al., 1983). Elevated concentrations of PCB and OCL have been detected in bottom-feeding organisms at the lower trophic levels of aquatic food chains and, therefore, legislation has been introduced in several countries in order to limit the concentrations of both PCB and OCL in natural waters (Commission of the European Communities, 1979; United States Environmental Protection Agency, 1979; Commission of the European Communities, 1978) and aquatic organisms (United States Environmental Protection Agency, 1976). The presence of persistent organochlorine compounds in waste waters of both domestic and industrial origin is an additional area of concern since PCB and OCL are not easily degraded by conventional sewage treatment processes and will emerge from waste water treatment plants in either the final effluents or sewage sludges produced, with further potential for environmental contamination.

Water use by the consumer, whether domestic or industrial in nature, results in a deterioration of its quality, with the wastewater produced requiring appropriate treatment before discharge to the environment (Tebbutt, 1979). The aim of wastewater management is to protect the environment in a manner determined by economic, social and political pressures (Metcalf & Eddy, 1979). Justification of the cost of removal of a pollutant and the ultimate disposal of the removed pollutant constitute the fundamental considerations for wastewater treatment processes (Winkler, 1981). The relative distribution between removed pollutants in the form of sewage sludge and those considered permissible to pass out of the treatment process in the effluent has been summarised by the terse maxim that pollution can

never be eliminated, only biased towards a more acceptable context (Winkler, 1981). The methods of exploitation of water resources for as many uses as possible have been referred to as multi-purpose schemes. The main disadvantage is that more polluted sources are utilised as drinking water sources, with the necessity for more complex and expensive treatment methods. Furthermore, multi-purpose schemes can only attempt to resolve the often conflicting requirements of various water uses (Tebbutt, 1973).

Water treatment, its supply and use and the subsequent treatment of wastewater form a complex system, representing an integral part of the hydrological cycle. The interdependence of water, wastewater and pollution control has been demonstrated by the establishment of the Regional Water Authorities, responsible for the management of the whole water cycle (Tebbutt, 1977). Furthermore, the practice of recirculating reclaimed wastewater has produced a change in terminology from 'sewage works' to 'water reclamation works' in the Severn-Trent Water Authority, reflecting the change in status of wastewater (Brewin & Hellawell, 1980).

The hydrological cycle represents an important mode of transport for a variety of waste materials. The susceptibility of aquatic ecosystems to pollution is due to the complex structure of food webs (Forstner & Wittmann, 1979). Nutrients, such as nitrogen or phosphorus compounds, (Lee et al., 1978) and refractory chemical substances (Englande & Reimers, 1982) may cause serious disturbances in aquatic environments.

Wastewater treatment processes, both domestic and industrial, represent the most important source of pollutants in the hydrological cycle (Forstner & Wittmann, 1979). Organochlorine compounds have been regarded as important contaminants owing to their biological recalcitrance and ability to bioaccumulate in aquatic sediments, flora and fauna with further potential for biomagnification through terrestrial food chains (Edwards, 1973).

Such compounds give rise to concern in essentially three respects during wastewater treatment; if present at sufficiently high concentrations, their toxic properties may disturb biological treatment processes, accumulation in sewage sludges may present problems

during subsequent disposal operations or failure to remove them may result in the discharge to receiving waters of unacceptably contaminated effluents.

Emphasis on the control of specific pollutants, especially organic compounds, and their impact upon the quality of a receiving water is dependent on the use for which they are required. Different water quality criteria are defined for potable supplies, freshwater life, agricultural uses, marine life, industrial water supplies and recreational uses (Daniels & Conyers, 1976). Such criteria may specify limits for pathogenic microorganisms, plant nutrients and other chemical substances. The latter will include individual elements or chemical compounds up to complex synthetic organic substances. Some chemicals are harmful to health, others have organoleptic properties, imparting objectionable taste, colour or odour to the water. It is the persistent chemicals which cause most concern; these include nitrogen (as ammonia or nitrates), phenol, trace organics and specific toxic elements and organic compounds (Englande & Reimers, 1982).

Organic compounds in surface waters prompt four specific areas of concern; water reuse and effects on human health, effects on other uses of river waters; ecological effects in rivers, and contamination of the hydrosphere (Bedding et al., 1983).

Successful water quality management is achieved by adherence to water quality criteria. In the U.K., such criteria are implemented via the Public Health Acts, 1936 and 1961, the Water Act, 1973 and the Control of Pollution Act, 1974, as well as recognition of various EEC Directives (Goodman, 1980).

Terminology such as 'poisonous, noxious or polluting' recurs in legislation for water pollution control, although its definitions are open to debate (Chalmers, 1981). More specific water quality criteria attempt to clarify permissible degrees of pollution by being more explicit. Water quality criteria are specified by various authorities for drinking water (World Health Organisation, 1970; World Health Organisation, 1971; Government of the U.S.S.R., 1973; U.S. Environmental Protection Agency, 1976; European Economic Community, 1980) for surface water intended for abstraction of drinking water (European Economic Community, 1975), for freshwater supporting fish

life (European Economic Community, 1978), for surface water (Government of the U.S.S.R., 1970), for irrigation water (Federal Water Control Administration, 1968), and to control pollution of the aquatic environment caused by the discharge of toxic substances (European Economic Community, 1976).

Wastewater treatment processes are important in controlling the quality of surface waters; they determine the quantities of organic micropollutants in effluents which may be discharged to receiving waters and the quantities of organic micropollutants accumulated in sewage sludges which may indirectly contaminate water resources via land application.

Since the discharge of industrial effluents directly to watercourses is proscribed under the Control of Pollution Act, 1974, (Government of Great Britain, 1974), virtually all industrial wastes enter the sewerage system and may contribute significantly to the toxic organic input.

Due to the presence of organochlorine micropollutants in sewage influents, it may be advantageous under some conditions to preferentially accumulate them in the sewage sludges during sewage treatment, thus facilitating controlled disposal with the sludge rather than unregulated discharge to the receiving waters. Among the objectives of wastewater treatment are the removal of suspended solids and floating material, biodegradable organic constituents, pathogenic organisms, specific compounds such as those of nitrogen, phosphorus or organic substances and dissolved inorganic solids (Metcalf & Eddy, 1979). Methods of wastewater treatment combine physical, chemical and biological processes (Tebbutt, 1977; Metcalf & Eddy, 1979; Steel & McGhee, 1979). Figure 1 represents a typical treatment process incorporating preliminary treatment, primary sedimentation, the activated sludge process.

Influent wastewater or raw sewage undergoes preliminary treatment in order to remove coarse solids and grit which might otherwise adversely effect subsequent treatment processes or mechanical equipment. Storm tanks serve to damp excessive variations in flow rate to the wastewater treatment process. Hydraulic overloading may produce a deterioration in effluent quality (Steel & McGhee, 1979).

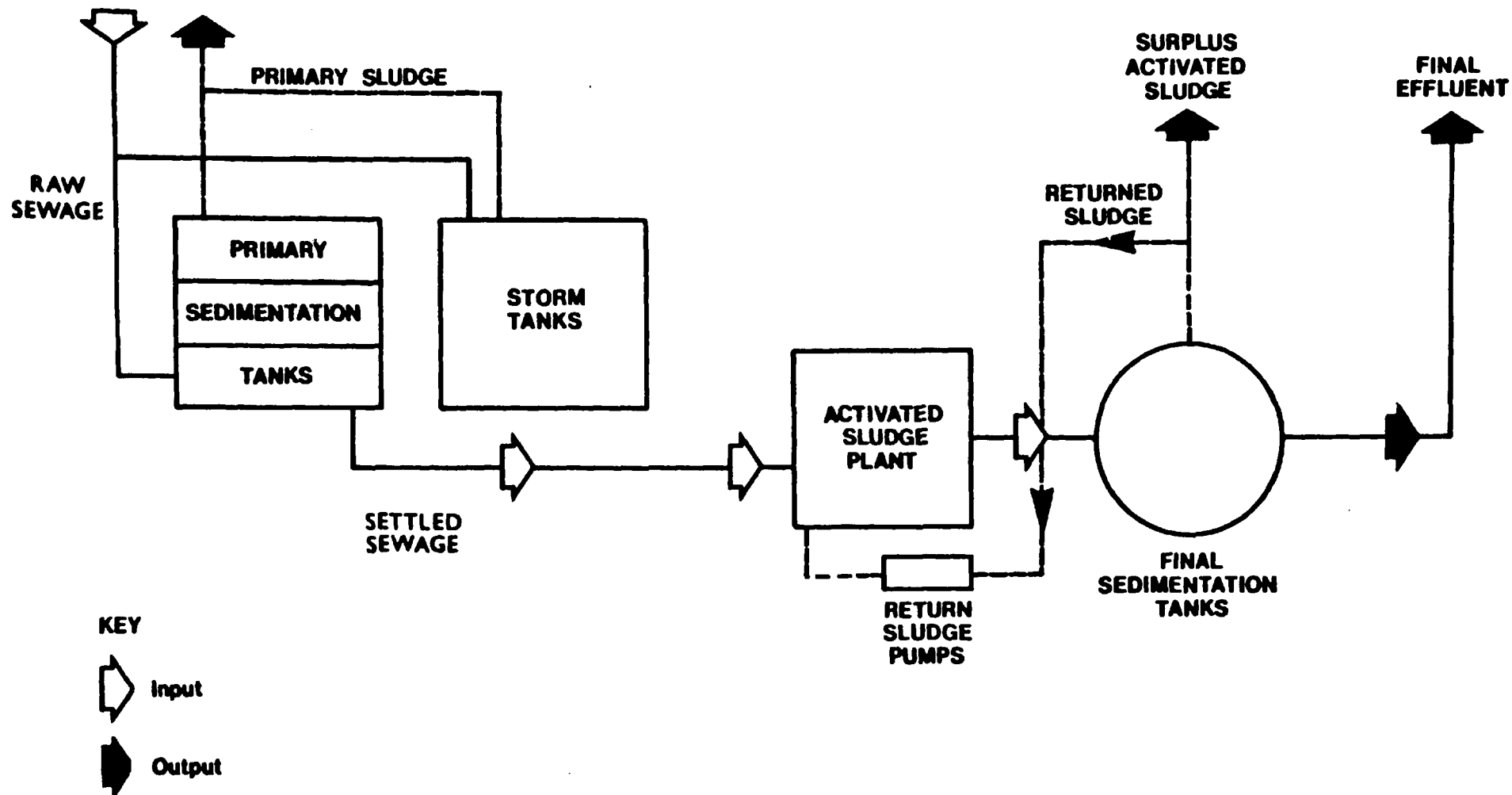


Figure 1. Schematic representation of typical sewage treatment processes

Primary treatment has traditionally comprised a sedimentation process to remove suspended solids by settling under near quiescent conditions. Sedimentation may not remove all solids, however, due to the minimal settling velocity attributable to either the size or specific gravity of some particles. Colloidal particles are considered to have virtually no settling velocity (Tebbutt, 1977; Degremont, 1979). Chemicals may be added to remove colloidal solids or for the precipitation of phosphorus compounds during primary settling (Steel & McGhee, 1979). Removal of settleable solids is the principal role of primary sedimentation, a subsidiary objective is the removal of floating material by skimming. Solids removal during primary sedimentation has been correlated with surface loading rate, overflow velocity, influent solids concentration and the ratio between sedimentation tank diameter and height (Annesini *et al.*, 1979). Influent suspended solids concentrations were shown to be more important in controlling solids removal than was the surface loading rate, due to the effect on influent density and degree of flocculation; thus, it was considered that adoption of shorter retention times would not cause a significant loss in removal efficiency (Tebbutt & Christoulas, 1975). Generally, retention times in primary sedimentation tanks are short, approximately one or two hours at peak flow with surface loading rates of between 1 and 2.5 m³ m⁻² h⁻¹, thus primary sedimentation tanks are typically between 2 and 5 m in depth (Steel & McGhee, 1979). Most treatment plants incorporate mechanically cleaned sedimentation tanks of circular or rectangular design (Metcalf & Eddy, 1979), with radial and horizontal flows respectively. Horizontal tanks require less land area than circular tanks, especially when multiple tanks are used. However, circular tanks have longer weir lengths and simpler scraping mechanisms than horizontal tanks (Tebbutt, 1977; Metcalf & Eddy, 1979). Sedimentation affects the removal of settleable solids and concentrates these solids in a smaller volume, as sludge, and produces an effluent suitable for secondary or biological treatment.

Aerobic biological oxidation is usually employed for secondary treatment; microorganisms remove soluble and colloidal organic matter and are then removed themselves. This principle is basic to all secondary biological treatment, although its application may vary as either attached (film) growth or suspended growth processes (Metcalf & Eddy, 1979; Steel & McGhee, 1979). Attached growth pro-

cesses include the trickling or percolating filter and rotating biological contactors. Suspended growth processes include the activated sludge process and oxidation ponds.

The percolating filter operates by trickling settled sewage through a bed of stone or clinker containing microorganisms as a slime or film. The activated sludge process is a common alternative to the percolating filter method, offering advantages of higher organic loadings and a requirement for less land area. The conventional activated sludge process consists of a rectangular aeration tank, a sedimentation tank and a solids-return line from the base of the sedimentation tank. The returned solids are mixed with the incoming wastewater to maintain a mixed liquor suspended solids (MLSS) concentration typically in the range 2000-8000 mg l⁻¹. The biomass is maintained in suspension as flocs in the aeration tank by mechanical agitation or by air injected at the base of the tank. Injection of air also maintains a dissolved oxygen (DO) concentration of 1-2 mg l⁻¹ in the mixed liquor suspension. Modifications of a basic plug flow process such as 'tapered' or 'step' aeration and 'completely mixed' systems have aimed to correlate oxygen supply with demand, to minimise the effect of peak organic or hydraulic loads and to avoid locally high concentrations of toxic compounds (Steel & McGhee, 1979). The mixed liquor achieves rapid biodegradation of the wastes and provides a good quality sludge with rapid settling characteristics (Tebbutt, 1977).

Settling of activated sludge flocs is dependent on the degree of flocculation, size and density of the flocs, and their structure and water binding capacity; the important factors determining settling characteristics of the sludge include the biochemical and morphological structure, surface charge, hydrophilic-hydrophobic balance and solvation of particle surfaces. Bioflocculation and sedimentation are primarily influenced by the composition of the wastewater, microbial flora and operational parameters. Of secondary importance, resulting from the metabolic activities determined by the primary factors, are the surface properties of cells and their release of polymeric material (Eriksson & Axberg, 1981).

Stoke's Law, derived from a consideration of the physical factors affecting settlement of a particle, is not strictly applicable

to wastewater sedimentation (Tebbutt, 1979); agglomeration of irregularly shaped floc particles causes an increased settling velocity due to the formation of larger and heavier particles (Tebbutt, 1977). Both density and viscosity may vary with temperature; the particle density may vary from almost 1 g cm^{-3} for biological growth up to 4 g cm^{-3} for sand (Faisst, 1980).

Four types of settling arise due to the interaction and concentration of particles (Tebbutt, 1977; Metcalf & Eddy, 1979; Steel and McGhee, 1979; Anderson, 1981). Class or type 1 settling refers to the settlement of discrete particles with little interaction between particles at a low solids concentration as occurs in grit removal or primary sedimentation. Class or type 2 settling refers to the sedimentation of flocculent particles, with resultant increased mass and settling velocities; this type may occur in secondary settling after the activated sludge process, or in the removal of chemical flocs in settling tanks. The third type of sedimentation is called 'hindered' or 'zone' settling; at intermediate concentrations in the region of 2000 mg l^{-1} and above, the particles are sufficiently close together to form interparticulate forces which hinder the settling of neighbouring particles. As the particles are fixed relative to one another, the suspension settles as a unit with a solids-liquid interface at the top of the settling mass. Such settling occurs also during secondary settling following a biological treatment stage, and in the lower layers of primary sedimentation tanks. The fourth type of settling is referred to as 'compressive' settling; at high concentrations the particles are in contact, so forming a cohesive structure. Compression occurs due to the weight of particles, which increasingly accumulates by sedimentation from the suspension above. This fourth type usually occurs during sludge thickening or in the lower layers of a deep sludge mass as at the base of secondary settling tanks. During any sedimentation process, more than one type of settling would occur; it may be possible for all four types to be exhibited at various depths in a single sedimentation tank (Metcalf & Eddy, 1979). Thus, sedimentation is of paramount importance for efficient wastewater treatment overall and is applied during most stages. Final sedimentation aims to produce an effluent of a quality suitable for discharge to receiving waters. The normal standard attained is an effluent with a suspended solids (SS) concentration of 30 mg l^{-1} and a biochemical oxygen demand (BOD) of 20 mg l^{-1} or better (Tebbutt,

1977). Where more stringent or specific criteria are defined for limiting effluent discharge, tertiary treatment may be necessary. A combination of physical, chemical and biological processes may be utilised in order to remove organic compounds, toxic elements or dissolved inorganic solids for higher quality effluents (Metcalf & Eddy, 1979).

Treatment and disposal of sludges may account for up to half the total cost of wastewater treatment (Lester et al., 1983). Methods of sludge treatment prior to disposal include dewatering, drying, composting, thermal regulation and stabilization (Metcalf & Eddy, 1979). A common practice is the anaerobic digestion of sewage sludge, producing methane and a more stable sludge of reduced mass, so facilitating ultimate disposal.

In the U.K. approximately 1.24×10^6 tonnes of dry sewage solids are disposed of per annum; approximately 74% is disposed of to land, 23% is dumped at marine sites and the remaining 3% is incinerated (Government of Great Britain, 1978). Of the proportion disposed to land, 56% is applied to agricultural land and therefore a total of approximately 64.4% (some 0.8×10^6 tonnes) is disposed to farm land and the oceans. Disposal of sewage sludge to agricultural land is considered to be beneficial in terms of the return of organic matter and inorganic nutrients to the soil (Government of Great Britain, 1970; Government of Great Britain, 1977). The dumping of sewage sludge at sea is also an important disposal option in economic terms where it exists as the only viable alternative for large sewage treatment works situated near the coast, when the costs of transporting by road large volumes of liquid sludge are considered.

It would appear that potentially the most important route for contamination of food chains by organic compounds is the disposal of sewage sludge to pasture land (Lindsay, 1982), since between 6 and 14% of the dry matter food intake of foraging livestock may be soil (Healy, 1968). In the case of lactating cows, lipophilic persistent organochlorine compounds consumed in this manner may be released into the milk; this has been demonstrated to occur in a limited number of isolated cases (Bergh and Peoples, 1977; De Haan, 1977; Lindsay, 1979).

Although plant intake of organochlorine compounds has been reported, from both insecticide-treated soils (Lichtenstein, 1959; Nash, 1968) and soils amended with sewage sludge contaminated by PCB (Lawrence and Tosine, 1977), it appears that the greater proportion of persistent organochlorine residues present in soils or sludge-amended soils will be tightly bound to humic materials (Beall and Nash, 1971; Jacobs *et al.*, 1976) and therefore be largely unavailable to plants (Lindsay, 1982).

The disposal of sewage sludge contaminated with PCB and dieldrin in the Firth of Clyde has been demonstrated to result in elevated concentrations of these residues in bottom sediments and fauna (Halcrow *et al.*, 1974) and in certain fish species (Waddington *et al.*, 1973). The discharge of PCB and dieldrin to sewage treatment works in the area has subsequently been reduced and the residue concentrations have decreased (Lindsay, 1982). If marine disposal of sewage sludge is carried out in areas where dispersion and dilution are rapid, few or no significant deleterious effects may be incurred.

There exist at present within the European Economic Community no legislation or guidelines for the disposal of sewage sludge contaminated by persistent organic micropollutants. In the USA, an interim regulatory section of the Criteria for Classification of Solid Waste Disposal Facilities and Practices proposes a limit of 10 mg kg^{-1} for PCB in sewage sludges which are disposed of to agricultural land used for growing 'food-chain crops' (crops produced for animal feed) (United States Environmental Protection Agency, 1979).

Aqueous effluents from industries concerned with the manufacture and usage of PCB generally contain relatively high concentrations of PCB, as a result of spillage, production losses and personal/clothes washing. The waste production and treatment practices of the sole PCB manufacturer in the U.S.A. have been fully evaluated and documented. Concentrations in the trade effluent varied between 150 and $750 \text{ } \mu\text{g l}^{-1}$; these were subsequently reduced by a factor of approximately 100, by dilution in the receiving municipal sewers (United States Environmental Protection Agency, 1976a). Similar studies of the waste waters discharged from transformer and capacitor manufacturing plants revealed PCB concentrations of between 0.3 and $120 \text{ } \mu\text{g l}^{-1}$ and 3.2 and $13,300 \text{ } \mu\text{g l}^{-1}$ respectively (United

States Environmental Protection Agency, 1976b). Little information is available on waste waters from investment casting plants, where highly-chlorinated PCB are used in the mould-forming waxes, but concentrations in raw sewage sampled from sewers adjacent to manufacturing plants were found to average $11.1 \mu\text{g l}^{-1}$ (Stratton and Sosebee, 1976). Although the use of Arochlor 1242 in carbonless copy paper has been discontinued (Hutzinger *et al.*, 1973) waste-paper recycling plants may continue to receive such items and it is evident that waste waters from recycling plants can contain up to $25 \mu\text{g l}^{-1}$ of PCB (United States Environmental Protection Agency, 1976c). Although occurrence of PCB in municipal raw sewage may be due predominantly to contributions from trade effluents, it is apparent that a low-level background contribution from domestic sources exists. Analysis of raw sewages sampled from 6 sewage treatment works in Canada revealed PCB concentrations of between 0.5 and $27.1 \mu\text{g l}^{-1}$. The highest concentrations were encountered in samples obtained from those plants receiving varying proportions of industrial effluent in the raw sewage, although raw sewage from one plant treating strictly domestic waste water contained PCB concentrations up to $1.6 \mu\text{g l}^{-1}$ (Lawrence and Tosine, 1977). A further study of 22 sewage treatment plants in Canada reported average PCB concentrations in raw sewage of between 0.1 and $2.8 \mu\text{g l}^{-1}$ (Shannon *et al.*, 1976). In the U.K. concentrations of PCB in raw sewage entering a conventional sewage treatment works over a five day period varied between 0.049 and $0.082 \mu\text{g l}^{-1}$. The plant received a negligible amount of trade effluent (McIntyre *et al.*, 1981a).

Two investigations of PCB in waste waters entering treatment plants in the U.S.A. report concentrations in the raw sewage of 39.0 to $470.0 \mu\text{g l}^{-1}$ (Bergh and Peoples, 1977) and 0.54 to $3.1 \mu\text{g l}^{-1}$ (Dube *et al.*, 1974) respectively. In the former case, trade effluent from PCB manufacture was known to be discharged to the plant.

OCL in waste waters have been shown to arise from both domestic and industrial sources. In the U.S.A, investigation of trade effluents from pesticide manufacturing concerns revealed typical OCL concentrations of approximately $9,270 \mu\text{g l}^{-1}$ (United States Environmental Protection Agency, 1976d). In the U.K., γ -HCH and dieldrin have been used in moderate quantities for timber preservation and moth-proofing of woollen products such as carpets (Government of

Great Britain, 1974). Analysis of final effluents from Scottish sewage works which received trade effluents from the carpet industries revealed dieldrin concentrations of up to $10 \mu\text{g l}^{-1}$ (Lowden *et al.*, 1969). Although alternative moth-proofing chemicals to dieldrin are now available and in use (Government of Great Britain, 1979), dieldrin in carpet factory effluents may still arise from washing of imported wool (Anon, 1979). Studies in the U.S.A of trade effluents from textile factories report dieldrin concentrations of between 12,000 and 65,000 $\mu\text{g l}^{-1}$ (Wilroy, 1963) and 1,200 $\mu\text{g l}^{-1}$ (Bernholz *et al.*, 1971) respectively.

The use of certain OCL (including γ -HCH and dieldrin) in the home may also give rise to residues in domestic sewage (Government of Great Britain, 1974) and it is further postulated that concentrations of OCL up to $0.15 \mu\text{g l}^{-1}$ in raw sewage may be derived from human excretion following the consumption of food containing residues of these compounds (Brooks, 1972). However, studies of γ -HCH concentrations in wholly domestic sewage at a treatment works in Northern Ireland have suggested that peak loadings of this compound in sewage were unrelated to human excretion (as measured by BOD₅ or urea/creatinine loadings) but were related to the discharge of washing water (Harper *et al.*, 1977). Concentrations of γ -HCH in the raw sewage varied between 0.034 and $0.339 \mu\text{g l}^{-1}$ over the period of study. Similar concentrations of γ -HCH of up to $0.125 \mu\text{g l}^{-1}$ have been detected in raw sewage in Israel (Kahanovitch and Lahav, 1974). Analyses of OCL in raw sewage over a five day-period at a U.K. sewage treatment works, which received negligible volumes of trade effluent, revealed concentrations of DDE between 0.020 and $0.31 \mu\text{g l}^{-1}$ and dieldrin between 0.024 and $0.039 \mu\text{g l}^{-1}$ (McIntyre *et al.*, 1981a). A summary of reported PCB and OCL concentrations in waste waters is included in Table 1.

Owing to the apolar nature of PCB and OCL, both of these groups of chemical compounds will be only sparingly soluble in waste water and will be principally associated with inorganic and organic sewage solids. It therefore appears likely that simple mechanical separation processes, such as sedimentation, will achieve significant removal of PCB and OCL into primary and secondary sludges. However, the settling process may be affected by such factors as hydraulic and suspended solids loadings and by the presence in raw sewage of com-

Table 1. Concentrations of polychlorinated biphenyls and organo-chlorine insecticides occurring in waste waters.

Compound	Waste Water Type	Concentration $\mu\text{g l}^{-1}$	Source
PCB	Raw sewage containing industrial effluent	4.1-8.7	Stratton & Sosebee (1976)
	Industrial effluent from investment casting plants	11.5-355.0 2.5	Kleinert (1976) Hesse (1976)
	Transformer plant effluent	up to 5,200	Hesse (1976)
	Raw sewage with industrial effluent	30-470	Bergh & Peoples (1977)
		0.5-17.5	Lawrence & Tosine (1977)
		0.1-2.8	Shannon <u>et al.</u> (1976)
		0.049-0.082	McIntyre <u>et al.</u> (1981a)
	γ -HCH	0.147	Harper <u>et al.</u> (1977)
		0.125	Kahanovitch & Lahav (1974)
Dieldrin	Domestic raw sewage	0.024-0.039	McIntyre <u>et al.</u> (1981a)
		76.0	Bernholz <u>et al.</u> (1971)
DDE	Domestic raw sewage	0.02-0.03	McIntyre <u>et al.</u> (1981a)

ponents such as fats, oils, greases and surfactants, which may influence the partitioning of apolar compounds between solid and liquid phases in raw sewage.

A detailed study of a sewage treatment works in the U.S.A. has shown that two stage biological treatment can achieve an overall removal of approximately 86% of PCB concentrations in the raw sewage of between 39 and 470 $\mu\text{g l}^{-1}$ (Bergh and Peoples, 1977). It was also demonstrated that PCB accumulated in primary and secondary sludges and in the trickling filter film. A comprehensive survey of 22 sewage treatment works in Canada revealed that an average of 50% removal was achieved during primary sedimentation, with approximately 44% of the initial PCB load discharged in the final effluent and 56% retained in primary and secondary sludges (Shannon *et al.*, 1976). An investigation of PCB removal during primary sedimentation at a U.K. sewage treatment works reported removal efficiencies over a five day period of between 33 and 75% with a mean removal of 51% (McIntyre *et al.*, 1981a). The removal efficiency of suspended solids by the primary sedimentation tanks at this sewage treatment works averaged 59% over the five day period of study and varied between 52% and 68%, which constituted a broadly similar range of removal efficiencies to those of PCB. Concentrations of PCB detected in the incoming raw sewage varied between 47 ng l^{-1} and 82 ng l^{-1} . Similar investigations during pilot-scale experiments using an integrated waste water treatment/water reclamation plant revealed PCB removals of approximately 50% in chemically aided sedimentation (Van Rensburg *et al.*, 1980).

A general paucity of information exists on the behaviour of organochlorine insecticides in waste water treatment processes. The removal of dieldrin by a conventional sewage treatment works has been investigated (Wilroy, 1963), where concentrations of 46.0 $\mu\text{g l}^{-1}$ in raw sewage were reduced to 2.0 $\mu\text{g l}^{-1}$ in the final effluent, reflecting average removals of 75% in primary sedimentation and 95% overall removal with two-stage and chlorine contact treatment. Dieldrin removals of 99% from wool-processing waste waters have been reported, where high concentrations ($>500 \mu\text{g l}^{-1}$) were treated by dilution with raw domestic sewage and stabilisation in a lagoon with long retention times (Bernholz *et al.*, 1971). The removal of DDE and dieldrin during primary sedimentation has been reported, where average

removals of 37% and 48% for DDE and dieldrin respectively were achieved (McIntyre et al., 1981a). The removal of γ -HCH has been studied in several domestic sewage treatment works in Northern Ireland, where concentrations in the final effluent were reduced by 44% over those present in the raw sewage (Harper et al., 1977). Similar analyses have been conducted at a number of sewage treatment works in the United States, where concentrations of γ -HCH, dieldrin, heptachlor and heptachlor epoxide were measured. Removal of these four compounds was found to be extremely variable, between 0% and 87%, for the treatment plants studied (United States Environmental Protection Agency, 1980). The behaviour of certain organochlorine insecticides in a pilot-scale activated sludge plant treating domestic primary effluents has been investigated (Saleh et al., 1980). Removals of aldrin, dieldrin and various DDT analogues were found to vary in the range 0% to 62%, depending to a large extent upon the influent concentrations of particular compounds. The reported removals of PCB and OCL during waste water treatment processes are summarised in Table 2.

Although only a limited volume of information is available on the removal of persistent organochlorine compounds during waste water treatment, it is apparent from the studies performed that significant removal of these substances into primary and secondary sludges will occur, with subsequent environmental implications for the mechanisms of sewage sludge disposal.

Analyses of sewage sludges from 22 treatment works in Canada revealed PCB concentrations in the range $0.6 \mu\text{g g}^{-1}$ to $11.8 \mu\text{g g}^{-1}$, (Shannon et al., 1976) whilst a more limited survey of 4 sewage treatment plants in Southern Ontario reported concentrations to vary between $92 \mu\text{g l}^{-1}$ and $192 \mu\text{g l}^{-1}$ (a dry weight concentration of approximately 1.84 to $3.84 \mu\text{g g}^{-1}$) (Lawrence and Tosine, 1977). OCL have also been detected in Canadian sludges, with concentrations of individual compounds lying in the range $0.7 \mu\text{g l}^{-1}$ to $20.77 \mu\text{g l}^{-1}$. The compounds measured included DDT analogues, chlordane, heptachlor, heptachlor epoxide, γ -HCH, aldrin and dieldrin (Liu et al., 1975). The highest concentrations of OCL were detected in sludges sampled from those sewage treatment works which received significant volumes of trade effluents. In the U.S.A., there have been several studies of both PCB and OCL concentrations in sewage sludges. Furr et al. (1976)

Table 2. Removal of polychlorinated biphenyls and organochlorine insecticides during waste water treatment

Compound	Type of treatment	Removal %	Source
PCB	Primary sedimentation/ percolating filters	86	Dube <u>et al.</u> , (1974)
	Primary sedimentation/ activated sludge	71	Bergh & Peoples (1977)
	Primary sedimentation/ various secondary treatment processes	66	Shannon <u>et al.</u> (1976)
	Primary sedimentation	51	McIntyre <u>et al.</u> (1981a)
Dieldrin	Primary sedimentation/ percolating filter	<95	Bernholz <u>et al.</u> (1971)
	Primary sedimentation	51	McIntyre <u>et al.</u> (1981a)
	Primary sedimentation/ equalisation lagoon	99	Wilroy (1963)
	Activated sludge pilot plant	0-63	Saleh <u>et al.</u> (1980)
γ -HCH	Primary sedimentation/ percolating filters	44	Harper <u>et al.</u> (1977)
DDE	Primary sedimentation	37	McIntyre <u>et al.</u> (1981a)
Aldrin	Activated sludge pilot plant	0-79	Saleh <u>et al.</u> (1980)

found concentrations of PCB and dieldrin to vary between $0.01 \mu\text{g g}^{-1}$ and $23.1 \mu\text{g g}^{-1}$ and between 0.03 and $2.2 \mu\text{g g}^{-1}$ respectively, in sewage sludges from 18 treatment plants, whilst other studies have reported PCB concentrations of between $10.8 \mu\text{g g}^{-1}$ and $765 \mu\text{g g}^{-1}$ (Erickson and Pellizzari, 1979; De Carlo, 1974; Dacre, 1978). The insecticides DDT, DDD, aldrin, dieldrin and chlordane have also been detected at concentrations in the range $0.25 \mu\text{g g}^{-1}$ to $16.2 \mu\text{g g}^{-1}$ (Farell and Salotto, 1973; Kowal and Pahreu, 1979). One study conducted in the Netherlands detected dieldrin at a mean concentration of $7.5 \mu\text{g g}^{-1}$ in sewage sludges (De Haan, 1977). In the U.K., analysis of sewage sludges from Glasgow, London and Manchester revealed PCB concentrations of between 0.1 and $185 \mu\text{g g}^{-1}$ (Holden, 1970) and two consecutive annual surveys of 11 sewage treatment works reported PCB concentrations in the range 0.004 to $2.68 \mu\text{g g}^{-1}$ (Water Research Centre, 1973; Water Research Centre, 1974). A survey of 7 sewage treatment plants in Scotland detected PCB at concentrations of between 0.07 and $6.16 \mu\text{g g}^{-1}$ (Cunningham *et al.*, 1973). Concentrations of PCB and certain OCL in 40 U.K. sewage sludges have been reported recently. PCB concentrations varied between 0.02 and $0.46 \mu\text{g g}^{-1}$, DDE concentrations between 0.01 and $0.49 \mu\text{g g}^{-1}$, γ -HCH between <0.01 and $0.93 \mu\text{g g}^{-1}$ and dieldrin between <0.01 and $1.26 \mu\text{g g}^{-1}$ (McIntyre and Lester, 1982).

The significance of PCB and OCL in sewage sludges is unclear at present. Where sewage sludges are disposed of to agricultural land, there exists the possibility of translocation of persistent organic residues into crop or forage plants and the ingestion of sewage sludge-amended soil by grazing animals (Lindsay, 1982). Studies on the contamination of different soil types by certain OCL have established the uptake of these compounds by crop plants (Lichtenstein, 1959; Nash, 1968), whilst a study in Canada (Lawrence and Tosine, 1977) has indicated elevated PCB concentrations in crops grown on land treated with PCB-contaminated sewage sludges. Analysis of milk samples taken from cows pastured on grazing land which had been fertilised with sewage sludge containing significant concentrations of PCB indicated that the milk was unsuitable for human consumption (Bergh and Peoples, 1977). Where sewage sludges are disposed of to sea, the presence of PCB and OCL may result in the contamination of sediments and marine fauna, as has occurred at the sludge dumping areas in the Firth of Clyde (Cunningham *et al.*, 1973).

Techniques available for the determination of PCB and OCL in waste waters and waste water sludges are essentially similar to those applied to surface and drinking waters, with the necessary addition of more stringent clean-up procedures for the reduction of gross interferences extracted from these complex sample matrices. Analytical methods for OCL initially were subject to unknown interferences which were then subsequently identified as PCB residues (Reynolds, 1969). Consequently, any analytical scheme designed for either group of compounds must be capable of separating the other group in order to facilitate analyses.

Procedures which have been used for the isolation of PCB and OCL from aqueous media include solvent extraction, adsorption onto porous polymers or resins and steam distillation. The isolation of PCB and OCL from drinking and natural waters by adsorption onto Tenax GC plugs has been described (Versino *et al.*, 1976), where quantitative recoveries of both groups of compounds were obtained. The use of Amberlite XAD resins for the adsorption of PCB and OCL from potable waters has also been reported (Musty and Nickless, 1974). A total of ten OCL and a PCB (Arochlor 1260) were recovered by this procedure, although aldrin, DDE and PCB were not quantitatively recovered. Subsequent work by the same authors investigated the effectiveness of a reversed phase liquid-liquid adsorbent system for the extraction of PCB and OCL from potable and river waters, consisting of Chromosorb W coated with n-undecane and Carbowax 4000 monostearate. Quantitative recoveries of organochlorine compounds from spiked water samples were experienced with the exception of β -HCH, aldrin, DDE and Arochlor 1260. Variation of pH between 5 and 9 and chloride concentrations of 10 to 25,000 mg l⁻¹ in water samples were found to have insignificant effects on the efficiency of the adsorption system. A comparison of the reversed phase system, Amberlite XAD-4 resin and porous polyurethane foam indicated, that with river water samples, similar results were obtained for all three methods. However, it was also apparent that Arochlor 1260 was not effectively recovered by the preferred method and that for environmental water samples containing suspended solids, solvent extraction would be the optimum procedure (Musty and Nickless, 1976). Polyurethane foam plugs coated with DC-200 liquid phase have been employed for the extraction of OCL from rain water samples in the U.K. (Wells and Johnstone, 1978), where few suspended solids were present. This system of concentration was found

to operate successfully in the ng l^{-1} concentration range for the compounds γ -HCH, DDE, dieldrin and endrin. It appears, therefore, that solid adsorbent systems for the concentration of organochlorine residues can only be applied where water samples contain low or negligible suspended solids concentrations and low concentrations of gross contaminants. Consequently, solvent extraction procedures are preferred for other types of samples, i.e. waste waters.

The most common solvent extraction technique for waters and waste waters involves manually shaking the sample with a suitable solvent (n-hexane) in a separating funnel (Wells and Johnstone, 1978). More efficient recovery of residues may be obtained by utilising mechanical stirring (Croll, 1972), ultrasonic mixing (Albone et al., 1972) and blending (McIntyre et al., 1979) or by successive extractions with fresh solvent (Holden, 1970). Soxhlet extractions have been found to be efficient for dried samples of sewage sludge and soil samples, using solvent mixture of acetone and hexane (Bergh and Peoples, 1977). The extraction of OCL from waste waters and waste water sludges has been reported, with the use of 15% methylene chloride in hexane and centrifugation to recover the solvent, a procedure which has been recommended by the United States Environmental Protection Agency (1981). In a study of PCB concentrations in raw sewages, samples were blended with acetonitrile and the aqueous mixture was subsequently extracted with hexane (Lawrence and Tosine, 1977). The extraction of PCB and OCL from sewage sludges has been achieved using small volumes (5 ml) of sludge and hexane solvent (20 ml) in conjunction with a high-speed laboratory disperser (McIntyre et al., 1980).

The extraction procedures available for PCB and OCL in waters and particularly waste waters are not specific and will extract other organic materials, thereby necessitating the use of clean-up procedures to purify the extract prior to analysis. These procedures possess a two-fold function, in that not only can gross contaminants be removed which may interfere with the analysis, but also the basis of the separation upon which clean-up is conducted may serve to act as a confirmatory analytical step. Solvent partition methods which have been used for the clean-up of OCL extracts include those employing dimethyl-formamide/hexane (deFaubert Maunder et al., 1964), acetonitrile (Hills, 1959), dimethylformamide (Burchfield and Storrs, 1953)

and dimethylsulphoxide (Wood, 1969). Quantitative recoveries are normally obtained, depending upon the particular determinand. Thin layer chromatography on silica gel and alumina adsorbents has been used for the clean-up of PCB and OCL extracts where negligible amounts of fat and lipid material have been present (Ruzicka and Abbott, 1973; Bäumler and Rippstein, 1961; Abbott and Thomson, 1964). The extract is normally applied as a streak onto relatively thick (1-5 mm) layers of adsorbent and after development with a typically non-polar solvent, the relevant areas of adsorbent are removed from the plate and the determinands eluted with a suitable solvent. Column chromatography has been extensively used for the clean-up of extracts for PCB and OCL analysis and also for the separation of PCB from the more polar OCL. The majority of workers in the U.S.A. have traditionally preferred the use of 'Florisil' (calcium magnesium silicate) column chromatography as a combined clean-up and separation procedure for PCB and OCL, the former being eluted from the column with hexane and the latter with a 15% diethyl ether/hexane mixture (American Public Health Association/American Water Works Association/Water Pollution Control Federation, 1976). Difficulties have been experienced with variations in the activity of the Florisil from batch to batch, but this has been largely overcome by the development of a standardisation test using lauric acid in order to determine accurate elution volumes for the various determinands (Mills, 1968). In the U.K., the favoured method for clean-up and class separation has been alumina column chromatography (for the removal of fat and lipid material) followed by silica gel adsorption chromatography for the separation of PCB from OCL. This procedure, originally developed by Holden and Marsden (1969) uses hexane eluent for the alumina column, followed by a first fraction eluted by hexane from the silica gel column (containing PCB, aldrin and p,p'DDE) and a second fraction eluted with 10% diethyl ether/hexane (containing the remaining OCL). The performance characteristics of this method have been thoroughly evaluated, including the activation and subsequent controlled deactivation of the adsorbents, the volumes of elution solvents required and the size of the columns. This technique has been developed further by other workers (Wells and Johnstone, 1977), who derived a clean-up scheme for a total of 17 organochlorine compounds, without the use of the mixed diethyl ether/hexane solvent, which was found to extract interferences from silica gel even after extensive heat treatment of the adsorbent. Although this method is claimed to be superior to the original proce-

cedure (Holden and Marsden, 1969), the longer time periods involved in clean-up and analysis can only be justified if a wide variety of PCB and OCL residues are present, which is often not the case with environmental samples. Where raw and waste water samples are to be analysed, the clean-up procedure may be further modified by the substitution of an alumina/alumina-silver nitrate column (Holmes and Wood, 1972) for the alumina column, in order to remove elemental sulphur, sulphur compounds and carotenes. This method has proved successful in the analysis of PCB and OCL in raw sewages (McIntyre et al., 1981a) and sewage sludges (McIntyre et al., 1979; McIntyre et al., 1980). Other techniques which have been employed for the removal of elemental sulphur and sulphur compounds from water and waste water extracts include shaking with elemental mercury (Chau and Cochrane, 1968) or elemental copper (Hrudey, 1978), refluxing with copper-aluminium alloy (Schultman et al., 1971), reaction with tetrabutylammonium sulphite (Jensen et al., 1974) and reaction with cyanide in acetone solution (Bartlett and Skoog, 1954). However, the above procedure (Holmes and Wood, 1972) is considered to be desirable since it combines clean-up, lipid removal and sulphur removal into one convenient experimental step. Simpler clean-up methods which have been reported include oxidation of paper mill effluent extracts with chromium dioxide solution, prior to the analysis of PCB (Easty and Wabers, 1977), treatment with concentrated sulphuric acid (Kadoun, 1969) or chromic acid (Easty and Wabers, 1978). In these cases, some OCL may be partially degraded during treatment and the methods are only suitable for determination of PCB, which are stable under extremes of pH (Hutzinger et al., 1972). Gel permeation chromatography with Sephadex LH-20 in ethanol has been used for the clean-up of grain extracts prior to OCL analysis (Horler, 1968) whilst a chromatographic system utilising 'Bio-Beads SX-2' and cyclohexane eluent has been investigated by Stalling et al. (1972) for the removal of lipid from fish extract for PCB and OCL analysis and was found to be successful.

Although a number of analytical methods have been used for the determination and quantitation of PCB and OCL, by far the most frequently used technique has been gas-liquid chromatography with electron capture detection, which has detection limits in the lower picogram range for many chlorinated organic compounds. Many types of gas chromatographic columns have been used for the separation of

PCB and OCL compounds, with both non-polar and polar liquid phases. The most successful and widely-used column packing is 1.5% OV-17 + 1.95% QF-1 coated onto a high performance support, which achieves acceptable separation of 13 common OCL and which has been recommended by USEPA as part of a standard method (United States Environmental Protection Agency, 1981).

There have recently been reports on the use of glass capillary column gas-liquid chromatography for the analysis of both PCB and OCL in environmental samples (Tressl and Wessely, 1977; Mattsson and Nygren, 1976) and also the use of combined capillary gas chromatography/mass spectrometry (Eichelberger et al., 1974; Tindall and Wininger, 1980). Although these two analytical procedures offer obvious advantages with respect to sensitivity, specificity and confirmation of compound identity, particularly in the case of complex sample matrices, their suitability for routine analysis of large numbers of samples has yet to be assessed.

Thin-layer chromatography has been applied extensively in the separation and detection of OCL. Silica gel and alumina layers have been used most frequently, although considerable success has been obtained using Florisil (Elbert and Stanley, 1965). The analysis of 9 common OCL in water samples has been reported (Smith and Eichelberger, 1965) together with an evaluation of adsorbents and solvent developing systems. It was concluded that silica gel plates in conjunction with carbon tetrachloride solvent produced superior chromatograms, detection limits of approximately 10 µg of each pesticide being reported. A total of 14 chlorinated hydrocarbons and OCL were separated in water extracts by Kawahara et al. (1967) using alumina layers and a 20% carbon tetrachloride in hexane solvent mixture. The developed chromatograms were visualised by spraying with a Rhodamine B solution and duplicate chromatograms were then redissolved in solvent prior to gas chromatographic analysis. The efficiency of removal from the plate and overall extraction efficiency were also investigated. One- or two dimensional TLC systems may be utilised (Eliakis et al., 1968) or one-dimensional systems with multiple solvent developments (Szokolay and Madaric, 1969). A total of ninety common pesticides, including OCL, have been separated to varying degrees using Florisil adsorbent and five successive solvent systems (Hamilton and Simpson, 1969).

Paper chromatography has also been applied to the analysis of OCL in environmental samples, but its use has declined somewhat due to the advent of more specific and precise instrumental techniques. Several comprehensive reviews of the subject are available (Getz, 1963; Coffin, 1966; Aly and Faust, 1975).

Although the analysis of OCL has progressed towards the accurate, sensitive methods of gas chromatography/mass spectrometry, the determination of PCB still presents problems in that chromatograms obtained from environmental sample extracts are rarely identical to those of the standard Arochlor formulations. Differences in recovery of individual components and the possible degradation of particular isomers may be responsible. Even with the use of capillary column techniques in conjunction with mass spectrometry, it is not possible to characterise an Arochlor formulation completely, so that the reporting of PCB concentrations will remain as a group parameter. The use of more sophisticated techniques such as single or multiple ion detection and negative chemical ionisation mass spectrometry have been used to advantage in the analysis of PCB and OCL in complex sample matrices (Dougherty *et al.*, 1972). Although adequate analyses may be achieved with packed column GC in the determination of PCB and OCL in relatively clean water samples, the analysis of these compounds in waste water and other complex samples may be complicated by varying concentrations of interfering compounds which may be co-chromatographed, resulting in the co-elution of determinand and interferent peaks and miscalculation of determinand concentration. Since these drawbacks are due to the limited resolving power of packed columns, the use of capillary columns of higher resolution and with greater numbers of theoretical plates should bring about improvements in the analytical protocol. An additional limitation with the use of packed column EC-GC has been the inability to employ temperature programming during analysis. Owing to the great sensitivity of EC detectors, the increase in column bleed with oven temperature, even for the most carefully prepared and inert packings, will result in severe and unacceptable baseline drift. Therefore, in all but low-sensitivity analyses, isothermal temperature regimes are applied. Whilst displaying good stability and reproducibility, these analyses are not optimised where mixtures of compounds are to be determined, since sensitivity will be biased towards early-eluting peaks in the chromatogram. This disadvantage may be overcome when capillary

columns are employed, since the low carrier gas flow rates used and the inertness of the column have negligible effects upon the stability of the detector when temperature programming is utilised. It is therefore possible to optimise the elution temperature, separation and resolution of individual compounds and consequently to maximise sensitivity to a range of determinands.

The production of glass capillary columns of an inert nature with stable liquid phase coatings (Grob *et al.*, 1978) and in particular the recent introduction of highly inert and stable fused silica capillary columns have improved both qualitative and quantitative aspects of analysis. In conjunction with novel sample introduction procedures (Grob and Grob, 1976; Grob and Grob, 1978a; Grob and Grob, 1978b; Grob, 1978) these potentially provide a powerful and quantitative analytical tool. Few reports are available of the analysis of PCB and OCL by capillary GC, the pioneering work having been conducted by Schulte and Acker (1974). PCB and certain OCL have been analysed in sewage sludge using a glass capillary column coated with SF-96 liquid phase, where 'splitting' injection procedures were used (Mattsson and Nygren, 1976), which have been shown to result in discrimination against high-boiling point sample components (Grob and Grob, 1976). The majority of packed columns resolve approximately 14 to 35 PCB peaks, whilst capillary columns may separate up to 70 (Mattsson and Nygren, 1976). In the former case, it is not feasible to identify individual isomers, since many PCB peaks will co-elute. However in the latter case, some individual isomers may be identifiable. This is particularly important where GC-MS is employed, since co-elution of compounds into the mass spectrometer will preclude the acquisition of pure individual spectra. It appears, therefore, that capillary column GC, both alone and in conjunction with mass spectrometry, will constitute a definitive method of analysis.

The development and availability of quantitative analytical techniques, is a prerequisite for the accurate determination of persistent organochlorine compounds in waste water samples in order to gain an appreciation of the behaviour and fate of such micropollutants in waste water treatment processes and during sludge disposal operations. This will permit an appreciation of the possible environmental and public health implications of the presence of persistent toxic micropollutants in raw and treated waste waters and in sewage sludges which are disposed to the environment to be obtained.

2. OBJECTIVES

There exists at present a grave paucity of information concerning the analysis, behaviour and removal of toxic organic micropollutants in waste water treatment processes and the fate and effects of such substances when discharged to the environment. Hydrophobic recalcitrant organic compounds such as polychlorinated biphenyls and organochlorine insecticides are among those substances of most concern in the hydrological cycle (Commission of the European Communities, 1976) owing to their resistance to degradation in waste water treatment systems and their accumulation in sewage sludges. Similarly, little information is available on the concentrations of these organic micropollutants in sewage sludges and the fate of residues when disposed to land or sea. In addition, problems may be encountered in achieving accurate quantitative analyses of PCB and OCL in waste waters and waste water sludges owing to the extreme complexity of the sample matrices. In order to address these problems, the work undertaken here has been performed in two major parts, involving the following objectives.

1. Study of the factors influencing the behaviour and removal of PCB and OCL during the primary sedimentation process, including:

(i) the development and evaluation of elementary analytical methods for the quantitative determination of PCB and γ -HCH, aldrin, dieldrin and endrin in waste waters and waste water sludges;

(ii) application of these methods to studies of the behaviour of PCB and OCL in waste water sedimentation using initially laboratory-scale experiments and subsequently a pilot-scale primary sedimentation plant.

2. Investigation of the occurrence, distribution and concentrations of PCB and OCL in sewage sludges, encompassing a range of sludge types and development and evaluation of advanced analytical techniques for the determination of these micropollutants in waste water samples, including:

(i) a survey of PCB and OCL concentrations in a large number of sewage sludges from UK sewage sludges using the elementary techniques developed under 1 (i) above.

(ii) development of advanced analytical methods, involving a comparison of the elementary packed column gas chromatographic method with capillary column gas chromatography and combined capillary column gas chromatography/mass spectrometry for the quantitation of PCB and OCL in a number of sewage sludge extracts.

3. MATERIALS AND METHODS

3.1. Primary sedimentation pilot plant

3.1.1. Location

The primary sedimentation pilot plant was located at Hogsmill Valley Sewage Treatment Works (Thames Water Authority), near Kingston-upon-Thames, Surrey.

The dry weather flow (DWF) design flow for the works was $33 \times 10^3 \text{ m}^3 \text{ d}^{-1}$; however, a DWF of $56.7 \times 10^3 \text{ m}^3 \text{ d}^{-1}$ and a total flow of $59.1 \times 10^3 \text{ m}^3 \text{ d}^{-1}$ have been reported, an upward trend being apparent in sewage flow, up to approximately 700 l s^{-1} (Eves, 1980).

3.1.2. Plant Description

3.1.2.1. Plant assembly

The primary sedimentation pilot plant used was essentially that reported previously (Rossin *et al.*, 1982), which consisted of nine separate systems. Raw sewage was drawn from the works by the sewage intake system and delivered to the constant head loading and distribution system. Solids amendment and persistent organics addition were made prior to division of the sewage flow into the sedimentation tanks via the mixing chambers. The effluent overflows were used to operate the flow measuring system. The sludge discharge system was used at the end of each experiment. Samples of influent, effluent and sludges were collected from the appropriate sampling points. A diagram of the pilot plant is presented in Fig. 2.

3.1.2.2. Sewage intake

The sewage intake system drew raw sewage from the distribution chamber of the primary sedimentation tanks of the works, after having been subjected to preliminary treatment for grit removal and comminution. A submersible pump was supported inside a stainless steel drum, 0.9 m in height and 0.6 m in diameter, with circular holes of 20 mm diameter around the base to allow the entry of sewage, while preventing the entry of large solids. The pump, Turo type T1-32 UM2 (Moffat and Bell Ltd., Surrey) delivered sewage at a constant rate. Both the pump and the drum were supported by steel chain, also used to lift them clear from the distribution chamber.

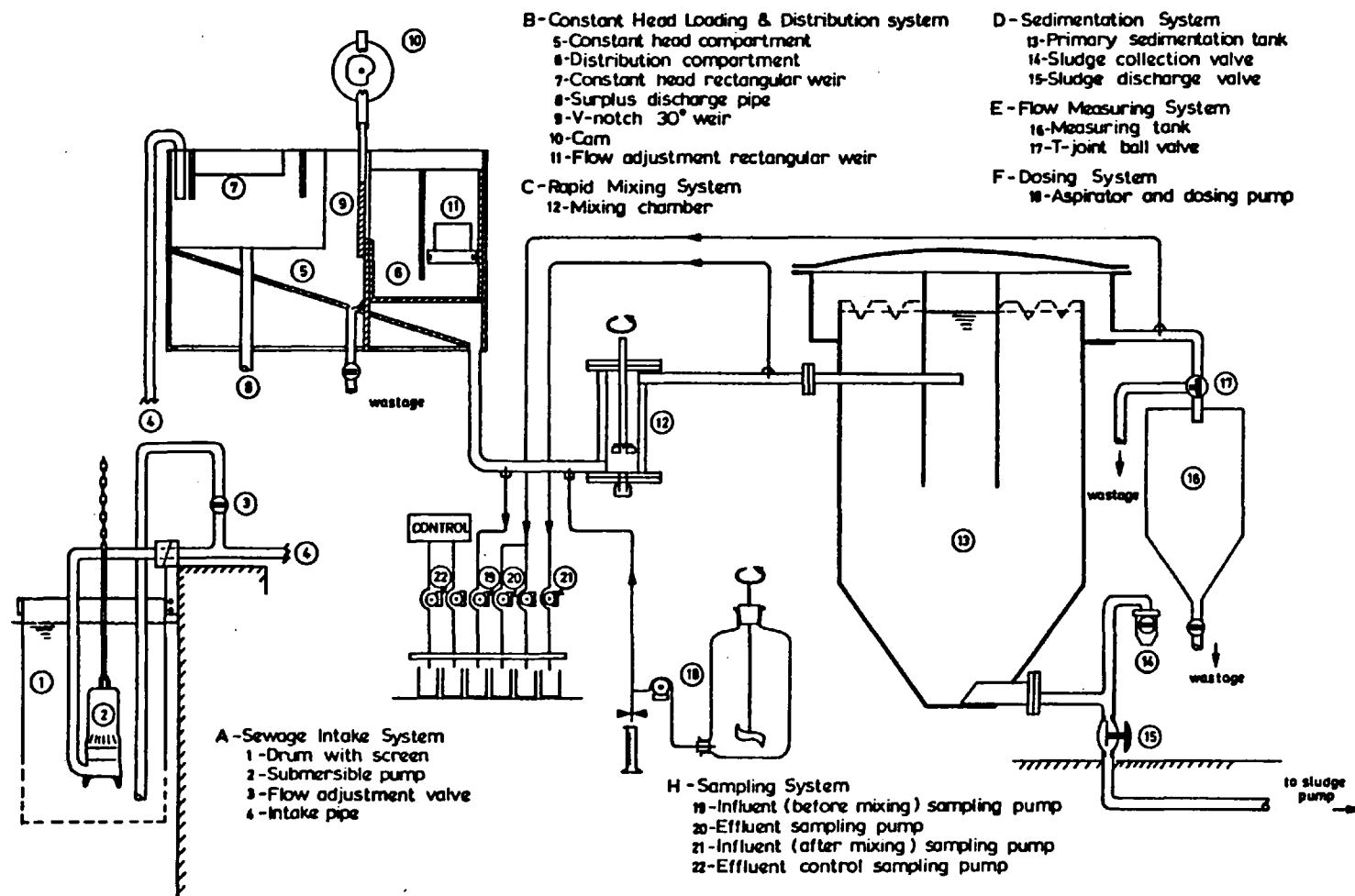


Figure 2. Diagram of the primary sedimentation pilot plant

3.1.2.3. Constant head loading and distribution systems

The constant head loading and distribution system was constructed of perspex due to it being transparent and easily machined. The system consisted of two compartments. Raw sewage was delivered to the first compartment which provided a constant level by diverting the necessary flow over two lateral rectangular weirs to waste. The second compartment distributed the sewage flow equally to both sedimentation tanks using two further adjustable rectangular weirs. The two compartments were separated by a perspex wall in which was mounted an adjustable 30° V-notch weir, which controlled the flow rate from the delivered sewage in the first compartment to the second distribution compartment. This angle was chosen as it provided sensitive control of flow and was unlikely to become blocked. The V-notch weir was manually adjusted, and a graduated scale was used to set the desired flow rate. All the weirs were protected by baffles to avoid turbulence at the surface of the liquid.

3.1.2.4. Organochlorine micropollutant addition

Concentrated solutions of PCB, aldrin and endrin were made up in acetone (100 ml), combined and diluted with distilled water in an aspirator (12 l) to give individual concentrations of approximately 250 $\mu\text{g l}^{-1}$. This solution was then pumped into the second compartment of the constant head loading and distribution system at the point where sewage entered from the V-notch weir, thus facilitating mixing prior to the distribution of dosed sewage to both sedimentation tanks. A peristaltic pump, 'multiperspex 2115' (LKB Ltd., Croydon, Surrey) delivered the solution through silicone rubber tubing at flow rates of between 2 and 20 ml min^{-1} , depending upon the raw sewage flow rate. In this way, concentrations of PCB, aldrin and endrin were increased by approximately 50 ng l^{-1} above the background present in the raw sewage.

3.1.2.5. Solids amendment

Primary sludge was collected from the primary sedimentation tanks of the works following desludging and stored in 60 l aspirators. The sludge was diluted with primary effluent to produce a sludge slurry which was thoroughly mixed in a storage tank used to amend the solids concentration of sewage prior to sedimentation. Reconstitution of sewage to achieve varying properties has been reported elsewhere (White & Allos, 1976). The slurry was added via a model HRSV 214 flow

inducer (Watson-Marlow Ltd., Falmouth, Cornwall) through silicone rubber tubing to the second compartment of the constant head loading and distribution system just before distribution to the sedimentation tanks, but after it had flowed over the two adjustable rectangular weirs, so that either one or both sedimentation tanks received sewage with amended solids concentrations.

The speed of pumping and the sludge dilution were determined by the solids amendment required at a specific flowrate. Nominal increases in solids concentration of 150, 300 and 500 mg l⁻¹ above the raw sewage solids concentration were made, such that the amended raw sewage solids concentrations to be studied ranged approximately from 400 to 1000 mg l⁻¹.

3.1.2.6. Mixing chambers

The influent sewage to each sedimentation tank passed through a mixing chamber. Each mixing chamber consisted of a 0.155 m diameter acrylonitrile-butadiene-styrene (ABS) pipe 0.4 m in length with four perspex baffles mounted internally. Sewage entry was at the bottom and exit at the top. Complete mixing of sewage with added organochlorines and additional solids was therefore achieved.

3.1.2.7. Sedimentation tanks

Two identical humus tanks (KTS Construction Co. Ltd., Tamworth, Staffordshire) were used, constructed of glass-fibre. The framework supporting the tanks was adjustable in order to level the tanks in situ. The upper part of the sedimentation tanks was cylindrical, with a height of 1.1 m and a diameter of 1.0 m, and a conical 60° hopper at the base. The total height of each sedimentation tank was 1.9 m and the capacity 0.915 m³. Influent sewage was introduced into the centre of an internal tube of 0.8 m height and 0.3 m diameter mounted vertically in the centre of each sedimentation tank. The effluent passed over twenty one 45° V-notch weirs into a circular trough surrounding the top of the tank and exited through a peripherally situated outlet pipe. The sedimentation tanks were covered with domed glass-fibre lids to prevent disturbance of the liquid surface.

3.1.2.8. Flow adjustment and measurement

After adjusting the V-notch weir, the total flow through the pilot plant was determined. Each effluent outlet pipe was fitted with a tee joint ball valve, enabling the effluent to be directed either to waste or into a 50 l measuring tank. Both effluents were collected in the measuring tank and the total flow rate was calculated from the time taken to reach the 50 l mark. Fine adjustment of the V-notch weir achieved the desired flow rate for a specific experiment. The effluents from each sedimentation tank were collected alternately to measure their flowrates. Any difference in flowrates between the sedimentation tanks was eliminated by balancing the flows by adjusting the two rectangular weirs in the distribution system as described in section 3.1.2.3.

3.1.2.9 Sludge discharge

On completion of an experiment, the entire contents of each sedimentation tank were emptied in turn as extraction of sludge alone was not possible. A diaphragm valve in the sludge line from the base of the sedimentation tank was opened to allow the sludge and sewage supernatant to be pumped by a model D30 pump (Mono Pumps Ltd., London) into two storage tanks. Approximately 10 l of the remaining liquid was used to ensure that all the sludge was transferred into the storage tanks by rinsing out the hopper base.

3.1.2.10 Sampling

The influent sewage sample points were located equidistant between the mixing chamber exit and the sedimentation tank entry. A 90° reducing tee joint in the influent pipe allowed a tight fitting bung to be secured, through which a glass tube was positioned. The glass tube had been bent to an angle of 90° in a smooth curve, such that its orifice was directed into the sewage flow. At the other end was secured a length of silicone rubber tubing and a screw clamp. Sampling was performed by unscrewing the clamp, allowing the first 100 ml to escape to waste and then collecting the next 500 ml in a 600 ml polypropylene beaker. A 1 l polypropylene measuring cylinder was used to measure 400 ml, after having been rinsed with the additional 100 ml.

Effluent samples were collected directly by positioning the collection beaker below the effluent outlet pipe. Again, a 500 ml

sample was collected for rinsing (100 ml) and measurement (400 ml) into a 1 l measuring cylinder. Separate sample collection beakers were used for influent and effluents for both sedimentation tanks, to minimise the risk of sample contamination error.

Sludge samples were taken following sludge discharge. The sludge and sewage supernatant were thoroughly mixed using the paddle stirrers in one of the two storage tanks and by pumping between the tanks, so as to produce a homogeneous sludge suspension. Several litres of this suspension were removed and used to fill a wide-mouthed 2 l bottle.

3.1.2.11. Drainage and pipework

The sewage intake line was constructed of vulcathene tubing. All interconnecting pipework including unions, elbows, valves and tee joints were supplied by Durapipe Ltd. (Cannock, Staffordshire), of size 50 mm diameter and ABS material. The main drainage line taking waste flows from the effluent outlets, constant head loading system drain and lateral weir overflows was 100 mm ABS pipe. The associated tubing for the Mono pump was flexible PVC pipe.

3.1.3. Experimental design and programme

The primary sedimentation tanks had a fixed surface area and volume, thus the relationship between flow and surface loading was also fixed. The flows and surface loading rates used during the work reported here covered the range of those reported in the literature. In the U.K., primary sedimentation tanks usually have surface loading rates of $30 \text{ m}^3 \text{ m}^{-2} \text{ d}^{-1}$ with retention times of approximately 2 h for flows of 3 DWF, giving 50 to 60% removal of suspended solids (Tebbutt, 1979). Primary sedimentation tanks are normally designed to provide 1.5 to 2.5 h retention times, although some provide 0.5 to 1.0 h when used prior to biological treatment; ranges of surface loading rates have been quoted at 32-48 and 80-120 $\text{m}^3 \text{ m}^{-2} \text{ d}^{-1}$ for average and peak flows, respectively (Metcalf & Eddy, 1979).

Ranges reported by Anderson (1981) for retention times were from 2.2 to 6.7 h and for surface loading rates of between 0.45 and $1.09 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$, equivalent to 10.8 and $26.2 \text{ m}^3 \text{ m}^{-2} \text{ d}^{-1}$, respectively. Conditions used during the preliminary experiments are included in

Table 3. Operational parameters for the preliminary pilot plant experiments

Conditions	Flow ($l\ s^{-1}$)	Surface loading rate ($m\ h^{-1}$)	Retention time (h)
1 DWF	0.111	0.6	2.3
3 DWF	0.333	1.7	0.8
Variable Flow	0.075-0.168	0.38-0.85	-

Table 3. Those used in the subsequent work are summarised in Table 4, derived from the flow rates and primary sedimentation tank capacity as reported previously (Rossin *et al.*, 1982b). Dry weather flow has been defined as the average daily rate of flow recorded on days when not more than 3 mm of rain had fallen in the preceding 24 h (Escritt, 1972).

The experiments were designed to examine the effects of increased SS concentrations on organic contaminant removal at each flow rate. Experiments using raw sewage unamended with solids were designated SS₀, while treatment corresponding to progressively increasing SS concentrations were designated SS₁, SS₂ and SS₃. For the initial experiment using 1 DWF discrete samples were analysed for SS concentrations and organic contaminant concentrations, together with the composite samples proposed for future work, as described in section 2.1.5. The experimental programme is set out in Table 5.

3.1.4 Plant Operation

The sedimentation tanks were operated in parallel, receiving the same flow rate but with differing solids concentrations. As can be seen from the experimental programme (section 3.2.3), solids amendment SS₃ was paired with SS₀, and SS₁ with SS₂, so that approximately equal quantities of primary sludge (200 l) were collected for every experimental run.

The time schedule adopted for each experiment was dependent on flow rate. The sedimentation tanks were filled with raw sewage and

Table 4. Values of primary sedimentation pilot plant parameters used

DWF	Flow rate ($l\ s^{-1}$)	Retention time (h)	Surface loading rate ($m^3\ m^{-2}\ d^{-1}$)
0.5	0.055	4.6	6.1
1.0	0.111	2.3	12.2
3.0	0.333	0.8	36.6
5.0	0.555	0.5	61.0

Table 5. Experimentation programme for the primary sedimentation pilot plant

Experiment	Sampling	Flow	Solids amendments
I	Composite & discrete	1 DWF	SS ₀ , SS ₃
II	Composite	1 DWF	SS ₁ , SS ₂
III	Composite	5 DWF	SS ₀ , SS ₃
IV	Composite	5 DWF	SS ₁ , SS ₂
V	Composite	3 DWF	SS ₀ , SS ₃
VI	Composite	3 DWF	SS ₁ , SS ₂
VII	Composite	0.5 DWF	SS ₀ , SS ₃
VIII	Composite	0.5 DWF	SS ₁ , SS ₂

the appropriate solids amendments commenced. One retention time was allowed to elapse before beginning organic contaminant additions. After a further retention time had passed, sampling commenced and lasted for a period equivalent to ten retention times. Therefore, at 1 DWF, samples were collected for a full 24 h period.

During each experiment, as well as manual sampling, several operations were performed to maintain steady functioning. The sludge slurry used for solids amendments was stirred at regular intervals using paddle stirrers and the delivery line was continuously monitored to clear blockages, so maintaining a constant amendment. For the SS₃

amendment one pump was used at a high addition rate; for SS₁ and SS₂, the same sludge slurry was added at two different rates, one to each sedimentation tank; sludge slurry additions ranged from 400 to 1000 ml min⁻¹. For experiments using SS₁ and SS₂ amendments, raw sewage was also monitored for suspended solids, COD and organic contaminant concentrations. The flow rates to each sedimentation tank were checked throughout the experimental period, and the weirs were kept free from solids and floating matter which would otherwise interfere with the flow rates.

3.1.5. Sample composites

The sampling frequency was determined by the flow rate for a specific experiment, so that flow proportional composites were obtained. The influent and effluent samples collected (400 ml) were transferred via a measuring cylinder into 2.5 l amber glass sample bottles. For the initial experiment using 1 DWF, samples were collected at intervals of 30 min, six samples being composited over 1 3 h period approximating to the hydraulic retention time. Discrete samples were also collected at 30 min intervals for the first experiment, in order to compare the mean values of discrete samples with the composite values, with respect to suspended solids and organic contaminant concentration. Composite sample values have been reported to be statistically different from discrete sample means (Walker, 1982). At flow rates of 0.5, 3 and 5 DWF corresponding sample composite periods were adopted, approximating to their respective retention times. Thus, six samples were composited at frequencies of 60, 10 and 6 min for 0.5, 3 and 5 DWF, respectively. Although each flow rate experiment lasted for a different period, all experiments at all flow rates involved the same total volume of sewage and consequently a constant total organic contaminant addition and equivalent sludge additions at each increased solids amendment.

Each experiment generated 10 influent, 10 effluent and one sludge sample for each sedimentation tank. Each sample was analysed for PCB and OCL, total and filtered COD and suspended solids concentrations, including volatile and non-volatile suspended solids.

3.2. Jar Tests

3.2.1. Experimental Programme

A large sample of raw sewage (20 l) was subdivided into aliquots in 1000 ml beakers and these were transferred to a 'Sedimentation Jar Tester' (Aztec Environmental Control Ltd., Croydon, UK) in batches of four. The raw sewage contained in the beakers was then fortified with incremental concentrations of solids, surfactants, grease and PCB and OCL. After stirring for 2 h at 100 rev min^{-1} , two subsamples were withdrawn for PCB and OCL analysis. The first aliquot was analysed without treatment whilst the second was centrifuged for 10 min at 1200 xg and the supernatant was withdrawn for analysis. The remaining raw sewage was taken for solids, surfactant and grease analysis. A summary of the experimental programme is presented in Table 6.

3.2.2. Addition of solids, surfactants, grease, PCB and OCL

Primary sludge of known solids concentration was used to fortify the raw sewage contained in the beakers to the required concentrations. Grease fortification of the raw sewage was achieved using a solution of corn oil in acetone (100 g l^{-1}). Surfactant addition to the raw sewage was achieved using an aqueous solution of domestic washing-up liquid (10 g l^{-1}). PCB and OCL were spiked into the raw sewage by microsyringe in acetone solution (100 mg l^{-1}).

3.3. Routine analyses

3.3.1. Solids.

Total and suspended solids concentrations were determined using the standard procedure (Government of Great Britain, 1972). For the primary sedimentation plant 50 ml of influent and effluent samples were filtered; sludge samples were diluted tenfold and then a 50 ml portion of the dilution was filtered. Filtration was performed using GF/C filters (Whatman Ltd., Maidstone, Kent), which were dried in an oven at 105°C before and after filtration to determine the suspended solids concentration. The dried filters were then ashed in a furnace at 450°C and reweighed in order to determine the non-volatile suspended solids (NVSS) and the volatile suspended solids (VSS) concentrations.

3.3.2. Chemical oxygen demand.

A modification of the standard method (Government of Great Britain, 1972) was used to determine the chemical oxygen demand (COD).

Table 6. Summary of Jar-Test Experiments

SOLIDS	Experimental Variable			DETERMINANDS
	MBAS	GREASE		
3 l sewage	4 l sewage	4 l sewage		4 l sewage
1.10 l distilled water	0.03 l distilled water	0.03 l distilled water		PCB 50 ng l ⁻¹
				OCL (background)
4 l sewage	4 l sewage	4 l sewage		4 l sewage
0.10 l distilled water	0.02 l distilled water	0.02 l distilled water		PCB 100 ng l ⁻¹
	0.01 l MBAS solution	0.01 l grease solution		OCL 100 ng l ⁻¹
4 l sewage	4 l sewage	4 l sewage		4 l sewage
0.05 l distilled water	0.01 l distilled water	0.01 l distilled water		PCB 200 ng l ⁻¹
0.05 l primary sludge	0.02 l MBAS solution	0.02 l grease solution		OCL 200 ng l ⁻¹
4 l sewage	4 l sewage	4 l sewage		4 l sewage
0.10 l primary sludge	0.03 l MBAS solution	0.04 l grease solution		PCB 400 ng l ⁻¹
				OCL 400 ng l ⁻¹

Total COD and filtered COD values were determined; the filtered COD samples being taken from the filtrate following SS determinations, both being preserved by acidification to 1% v/v nitric acid.

The method used a premixed oxidising reagent containing silver sulphate (7.89 g l^{-1}) and potassium dichromate (1.613 g l^{-1}) in 79% v/v sulphuric acid. Sample volumes of between 1 and 5 ml were heated with 9.5 ml of the oxidising reagent and 0.5 ml of mercuric sulphate solution (200 g l^{-1}) in 250 ml reaction flasks on an isomantle for 2 h. After cooling and rinsing down the condenser with distilled water, the residual dichromate was titrated against a 0.0625 N ferrous ammonium sulphate solution using two drops of ferrous phenanthroline indicator.

3.3.3. Surfactants

Surfactants were determined as methylene blue active substances (MBAS) using a standard method (Government of Great Britain, 1972) which involved chloroform extraction of a methylene blue-surfactant complex and colorimetric determination at 650 nm against standard solutions of Manoxol OT.

3.3.4. Grease

An aliquot (100 ml) of raw sewage was extracted with petroleum ether (25 ml) in a 250 ml separating funnel by manual shaking for 3 min. After separation of the organic phase and filtration (Whatman GF/C (U.C)) to remove solid material, the extract was evaporated to dryness in a pre-weighed round-bottomed flask (100 ml). After drying overnight in a desiccator, quantities of petroleum ether extractable grease were determined gravimetrically.

3.3.5. Polychlorinated biphenyls and organochlorine insecticides

Aliquots of raw and settled sewages were taken for analysis by the method of McIntyre *et al.* (1981a). Sewage sludge samples were analysed using the methods originally developed by McIntyre *et al.* (1980) and subsequently modified during these studies. Sewage sludges in dewatered cake form were analysed using the method of McIntyre *et al.* (1981b). Both methods involved extraction with hexane, clean-up of the sample extract by alumina/silver nitrate column chromatography and separation of PCB from COI by silica gel adsorption column chromatography.

Where sewage sludge samples were prepared for analysis by gas chromatography/mass spectrometry, aliquots (50 ml) of sludge were extracted twice with hexane (2 x 150 ml). After centrifugation at 1350 x g for 10 min to achieve phase separation, clean-up and separation procedures described by McIntyre *et al.* (1980) were employed, but with the use of larger clean-up columns and correspondingly greater quantities of adsorbents and eluting solvents.

3.3.5.1. Packed column gas chromatography

Packed column GC analyses were performed on a Hewlett-Packard 5710A gas chromatograph (Hewlett-Packard, Winnersh, UK) equipped with an electron capture detector. The glass column (2 m x 3 mm i.d.) was packed with 1.5% OV-17 + 1.95% QF-1 on 100/120 mesh Supelcoport (Phase Separations Ltd., Queensferry, UK) and operated at 200°C with a carrier gas (95%/5% Argon/methane) flow rate of 50 ml min⁻¹. Injector and detector temperatures were 250°C and 300°C respectively. On-column injections of 1-5 µl of standards and sample solutions were made using a 10 µl Hamilton microsyringe (Phase Separations Ltd.).

3.3.5.2. Capillary column gas chromatography

Capillary column analyses were performed on a Carlo Erba Fractovap 4200 gas chromatograph (Erba Science Ltd., Swindon, UK) equipped with a 'Grob-type' split-splitless injector and an electron capture detector. A fused silica capillary column, 25 m x 0.24 mm i.d., wall coated with CP Sil 5 liquid phase (similar to OV-101) was used with the following temperature programme: 40°C for 2 min, 50°C min⁻¹ to 150°C, then 8°C min⁻¹ to 275°C, hold for 5 min. Injector and detector temperatures were 250°C and 300°C respectively. The 'splitless hot-needle' injection technique was used for all analyses, with sample volumes of 2 µl. The septum purge flow from the injector was set at 5 ml min⁻¹ during analyses and the bottom split valve (50 ml min⁻¹) was opened 40 s after injection. Scavenger gas (95%/5% argon/methane) was supplied to the detector at 40 ml min⁻¹.

3.5.5.3. Gas chromatography-mass spectrometry

The Carlo Erba gas chromatograph was subsequently interfaced to a JEOL JMS-D300 double focussing mass spectrometer with a JMA-2000H data processing system (JEOL Ltd., Tokyo, Japan). Chromatographic conditions were identical to those described above, with the exception

that the fused silica capillary column was connected directly to the ion source of the mass spectrometer in order to optimise resolution and sensitivity. The mass spectrometer was operated under the following conditions.

Accelerating voltage	3.0 KV
Ionising voltage	70 eV
Ionising current	300 μ A
Ion source temperature	250°C
GC-MS transfer line	250°C
Electron multiplier	1.5 KV
Resolution	1000.

The multiple ion detection (MID) system was used to monitor specific characteristic ions in the mass spectra of PCB and OCL compounds. Thus, the ions recorded were M/Z 181 (γ -HCH), M/Z 237 (aldrin, dieldrin, endrin) and M/Z values 328, 364, 398, 432 and 468 (PCB). Calibration of the system and quantitation of samples were carried out automatically by the data system after injection of the requisite standard and sample solutions.

3.3.5.4. Standard calibration solutions

Standard solutions of PCB (Arochlor 1260, Monsanto Ltd., Newport, UK) were prepared in hexane (Fisons 'Distol' reagent, Loughborough, UK) at concentrations varying between 50 μ g l⁻¹ and 500 μ g l⁻¹, for calibration of the gas chromatographs. The organochlorine insecticides γ -HCH, aldrin, dieldrin and endrin (obtained from Alltech Associates, Carnforth, UK) were also made up in hexane, at concentrations of between 10 μ g l⁻¹ and 200 μ g l⁻¹. Identical solutions were used for calibration of the gas chromatograph/mass spectrometer.

4. RESULTS

4.1. Removal of PCB during primary sedimentation at different flow rates

No information on the concentrations of PCB (or any other OCL) in the raw sewage at the Hogsmill Valley sewage treatment works was available when this programme commenced. Indeed information on the behaviour of these types of chlorinated materials during primary sedimentation was almost non-existent. Moreover, there was little evidence to indicate what might control the behaviour of these materials during primary sedimentation. A preliminary experiment was therefore undertaken to elucidate practical problems and attempt to identify the effect of flow rate (surface loading) to pilot plant on PCB removal, since variations in flow are a regular occurrence at any sewage treatment works and particularly those receiving combined sewage containing storm water. The flows selected were 1 DWF (the normal flow to the works), 3 DWF (the maximum storm flow to the works) and a variable flow (which corresponded to normal diurnal variation in flow). Operation, control and sampling of the pilot plant were as described in Section 3.1.2.

Since raw sewage entering the primary sedimentation process is effectively separated into primary sludge and settled sewage, it is possible to calculate a mass balance of PCB at this stage. The PCB concentrations measured in the raw sewages, settled sewages and primary sludges for each of the three experiments are included in Table 7.

Table 7. Polychlorinated biphenyl concentrations in samples taken from the primary sedimentation pilot plant.

Flow	Average concentrations ng l ⁻¹		
	Raw sewage (Range)	Settled sewage (Range)	Primary sludge
1 DWF	11.6 (3.7-32.0)	3.6 (1.8-5.7)	151.9
3 DWF	10.3 (2.0-31.5)	5.4 (2.0-14.0)	304.2
Variable flow	20.0 (9.7-32.7)	8.4 (4.3-13.8)	110.1

The range of PCB concentrations was within one order of magnitude, varying between 3.8 ng l^{-1} and 32.0 ng l^{-1} at dry weather flow (1 DWF) between 2.0 ng l^{-1} and 31.5 ng l^{-1} at three dry weather flow (3 DWF) and between 9.7 ng l^{-1} and 32.7 ng l^{-1} for the variable flow experiment. Settled sewage PCB concentrations varied between 1.8 and 5.7 ng l^{-1} at dry weather flow, between 2.0 and 14.0 ng l^{-1} at three dry weather flow and between 4.3 and 13.8 ng l^{-1} under variable flow conditions. PCB in the primary sludges displayed an approximate 10-fold increase over raw sewage concentrations, with levels of 151.9 ng l^{-1} , 304.2 ng l^{-1} and 110.1 ng l^{-1} for 1 DWF, 3 DWF and variable flow respectively. Overall, PCB concentrations in the samples were low, which may be expected from the mainly domestic waste water which is treated at the Hogsmill Valley Works.

Additional information about the variation of PCB concentrations in the raw sewage influent to the pilot plant may be gained from examination of Fig. 3, where raw sewage PCB concentrations measured during the three experiments are plotted, together with the variation in raw sewage flow to the main sewage treatment plant. During the 1 DWF experiment, it is evident that there are three main peaks in PCB concentration, which correspond reasonably well with the peaks occurring in the flow, the first two, at 2200 h and 1200-1300 h on days 2 and 3 respectively, probably being caused by the discharge of domestic waste water to the works. During this experiment, a particularly heavy storm occurred in the area and the resultant storm water flow exceeded the hydraulic capacity of the sewage treatment works, thus bringing about a prolonged period (12 h) of constant, maximum flow (850 l s^{-1}). Associated with this event was the third peak in PCB concentration which may be explained by the resuspension of solids deposited in the sewers during times of low flow.

During the 3 DWF experiment, a similar pattern is apparent, with two well-defined peaks in PCB concentration at 2100 h and 1500 h respectively, corresponding with flow maxima. No discernible pattern was evident during the variable flow experiment, which may be due, with the exception of one high value, to the limited range of the PCB concentrations in the raw sewage.

When the average PCB concentrations for the three types of samples are multiplied by the necessary volumetric data, the mass

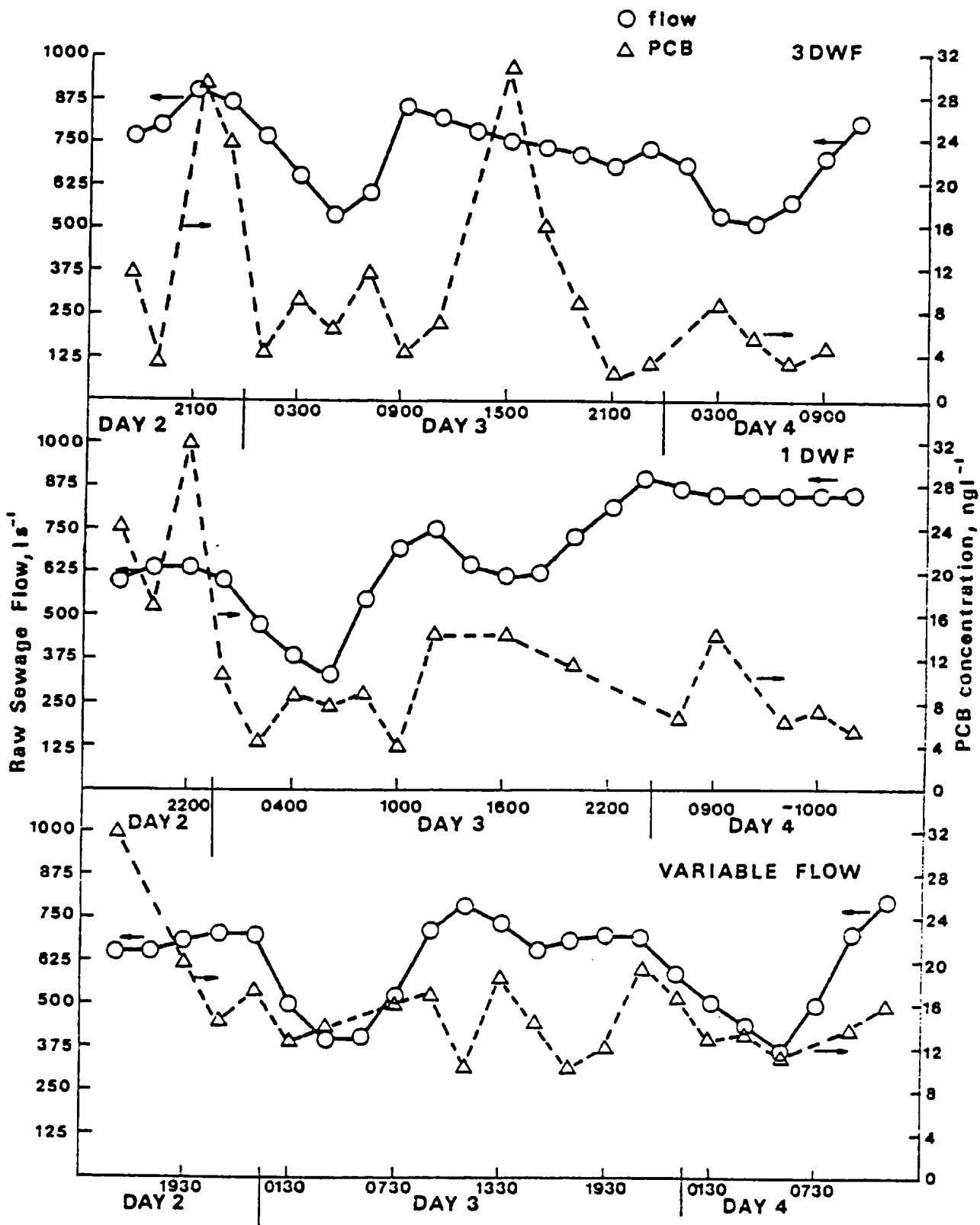


Figure 3. Variation in Raw Sewage PCB Concentration with Volumetric Flow to the Sewage Treatment Works.

Table 8. Mass balances of PCB through the primary sedimentation pilot plant.

Flow	Raw sewage load (μg)	Settled sewage load (μg)	Primary sludge load (μg)	Removal efficiency (%)	Balance error (%)
1 DWF	203.25	62.01	145.41	69.0	2
3 DWF	516.08	269.87	291.12	48.0	9
Variable flow	-	-	-	58.0	-

balances, containing in Table 8, are obtained. The mass balances for suspended solids are included in Table 9, for comparative purposes.

Table 9. Mass balances of suspended solids through the primary sedimentation pilot plant.

Flow	Raw sewage load (g)	Settled sewage load (g)	Primary sludge load (g)	Removal efficiency (%)	Balance error (%)
1 DWF	4397.9	1839.8	3240.4	58.1	15.5
3 DWF	19590.9	8217.2	19783.5	58.0	2.5
Variable flow	-	-	-	38.6	-

No mass balances were calculated for the variable flow experiment, since the data obtained were not amenable to treatment in this manner. In order to calculate percentage removals of suspended solids for the variable flow experiments, the average influent and effluent suspended solids concentrations were calculated at 2 h intervals. The flow rates, which were determined at hourly intervals, were averaged over the two-hourly periods and thus average removal efficiencies of suspended solids were determined, so as to calculate a

mean removal efficiency for the experiment. Average PCB removal was calculated in a similar manner.

The balance calculated for the one dry weather flow (1 DWF) and three dry weather flow (3 DWF) experiments agree particularly well, with errors of 2 and 9% respectively in the masses of PCB recovered.

Removals of PCB, 69% and 48%, for 1 DWF and 3 DWF respectively, calculated from raw sewage to settled sewage, are broadly similar to the suspended solids removals of 58% for 1 DWF and 3 DWF. The mass balances obtained for suspended solids exhibit a similar pattern to those of PCB, with good balances for both constant flow experiments.

4.2 Development of a method for the analysis of aldrin and endrin in sewage and sewage sludge

The analytical method used in the previous study had been further refined by McIntyre et al. (1980) to permit the analysis of the OCL insecticides dieldrin and γ -HCH. When this method was applied to samples from the Hogsmill Valley sewage treatment works additional peaks were regularly observed on the chromatograms and it was considered that these could be OCL insecticides. Two OCL insecticides which have been extensively used are aldrin and endrin and the suitability of the method for the analysis of these compounds was therefore evaluated. Five replicate subsamples of each of two sewage sludges and five replicate subsamples of raw sewage were analysed for these two compounds using the method of McIntyre et al. (1980). Further sets of five subsamples of each were then spiked with known concentrations of aldrin and endrin ($10 \mu\text{g l}^{-1}$) and were then analysed in a similar manner in order to determine extraction efficiency data for the two compounds.

Results of these analyses, including quantities of aldrin and endrin recovered from the samples and the percentage recoveries, are presented in Tables 10 and 11 for sewage sludge and raw sewage respectively.

Table 10. Recoveries of aldrin and endrin from mixed primary and digested mixed primary sludges.

Compound	Mean ¹ weight recovered		RSD		Recovery	
	from subsample (ng)		%		%	
	unspiked	spiked	unspiked	spiked		
Mixed Primary Sludge						
Aldrin	1.0	28.8	7.3	6.1	85.5	5.5
Endrin	2.8	41.6	6.8	5.3	88.2	5.6
Digested Mixed Primary Sludge						
Aldrin	ND	27.6	6.9	5.7	84.9	6.1
Endrin	3.3	40.7	7.9	5.1	85.1	4.8

1 of 5 replicates

Table 11. Recoveries of aldrin and endrin from raw sewage

Compound	Mean ¹ weight recovered		RSD		Recovery	
	from subsample (ng)		%		%	
	unspiked	spiked	unspiked	spiked		
Aldrin	2.0	39.4	9.2	4.7	74.8	6.1
Endrin	5.3	43.9	7.8	5.2	77.2	5.6

1 of 5 replicates

It is evident that the analytical method was suitable for the determination of aldrin and endrin in raw sewage and different types of sewage sludge. The extent and precision of the recoveries from sewage sludge were particularly good and the extraction efficiency of the two compounds from raw sewage (74.8% and 77.2% for aldrin and endrin respectively) were acceptable. Some difficulty was experienced initially with the determination of aldrin. Since this compound is eluted from the silica gel adsorption column with the PCB fraction (in the first 6.0 ml of hexane elutate), accurate control of the silica gel deactivation (3.0% w/w H₂O) was found to be critical in avoiding division of the aldrin between this fraction and the second, more polar insecticide elutate.

It was evident during the course of these experiments that aldrin and endrin were present in the samples at only low concentrations and that their detection in the presence of relatively high concentrations of interferences may be difficult. Aldrin elutes from the majority of non-polar packed gas chromatographic columns amongst a number of early PCB peaks and, in the case of high PCB concentrations, the aldrin peak may be partially or wholly obscured. Similar problems may be encountered to a lesser extent with endrin, which is subject to interference at low concentrations by unknown compounds.

4.3 Investigation of factors influencing the behaviour of polychlorinated biphenyls and organochlorine insecticides in waste water

Further to the preliminary pilot plant experiments described in Section 4.1, a series of jar tests was conducted in order to investigate possible factors which may affect and determine the behaviour of PCB and OCL during the primary sedimentation of raw sewage. Since the removal of PCB during the earlier pilot plant study could not be explained completely by the behaviour of solids, two additional parameters were selected for study; surfactants (methylene blue active substances-MBAS) and fats, oils and greases (FOG). As PCB and OCL are lipophilic in nature, the presence of FOG in raw sewage may affect their partitioning between solid and aqueous phases in waste waters. Similarly, concentrations of MBAS may enhance the aqueous solubility of essentially non-polar organics and thus influence the distribution of such compounds in waste water, thereby affecting their behaviour during primary sedimentation. A summary of the experimental regime adopted has been presented previously in Table 6. The results of these series of experiments to determine the degree of solid-association of PCB and OCL in raw sewage at varying concentrations of solids, MBAS, FOG and determinands are presented in Figure 4.

It is evident that these organic micropollutants are associated with suspended solids to a significant degree, since concentrations of PCB and OCL measured in the centrifuged samples were lower than those observed in the uncentrifuged samples. Little variation in the concentrations of PCB and OCL measured in the solids experiments was observed, with the exception perhaps of γ -HCH. Similar results were obtained during the MBAS experiment, where no

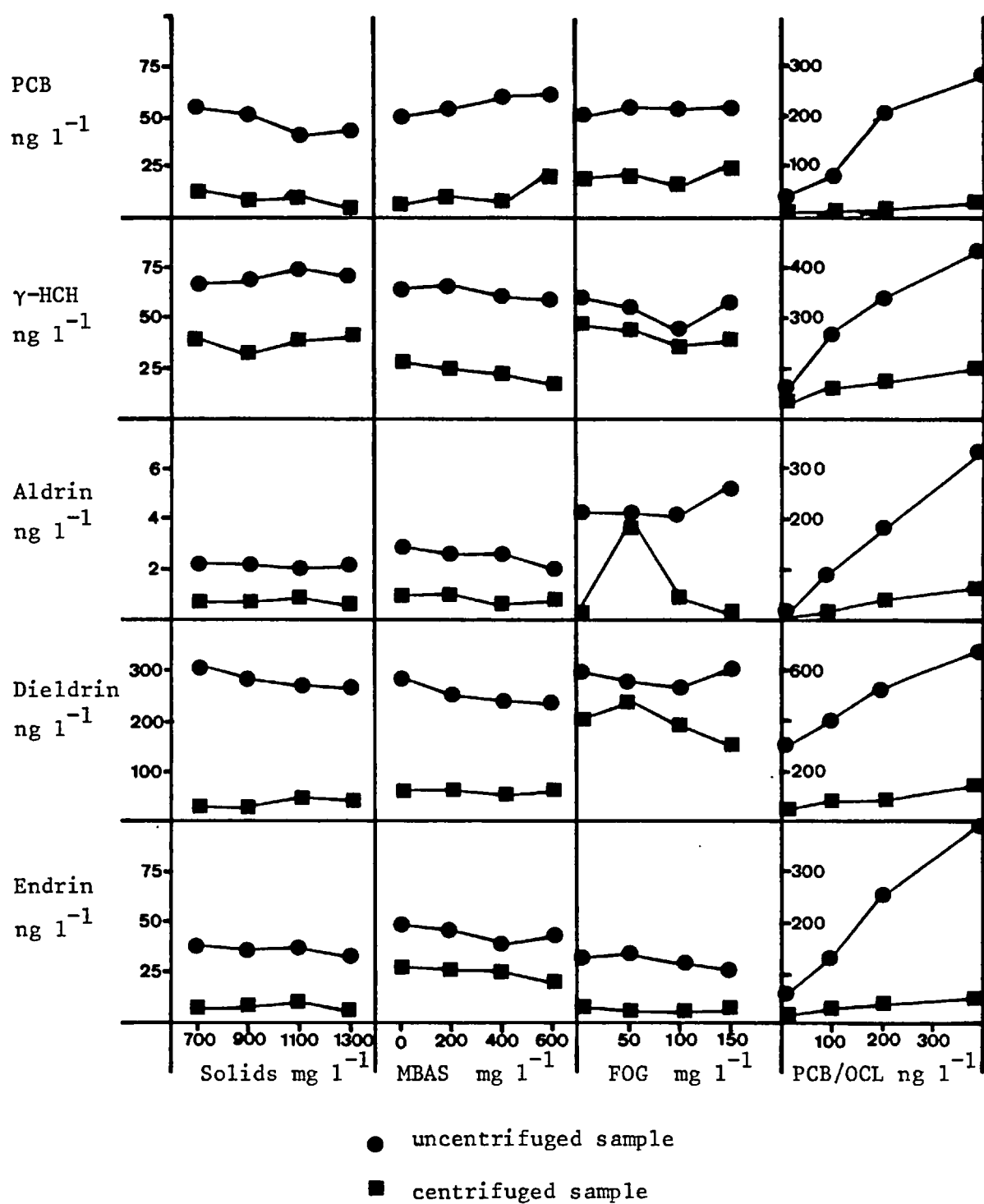


Figure 4. Effect of varying concentrations of solids, MBAS, FOG and PCB/OCL on the distribution of PCB and OCL in raw sewage

effects on the partition of PCB and OCL between solid and liquid phases in the raw sewage were encountered, even at the higher MBAS concentrations. In the series of FOG experiments, again, no observable effects were evident for any of the 5 determinands. During the experiment with increasing PCB and OCL concentrations, γ -HCH, dieldrin and endrin concentrations detected in the centrifuged samples were observed to increase with the quantities of determinands added to the raw sewage; PCB and aldrin exhibited a similar trend although to a lesser extent and it is considered that this is a function of the higher aqueous solubility of γ -HCH, dieldrin and endrin.

In general, the presence of varying concentrations of solids, MBAS and FOG in raw sewage samples had no observable effect upon the partitioning of PCB and OCL between aqueous and solid phases in raw sewage. When determinand concentrations in the raw sewage were increased above indigenous background levels, correspondingly higher concentrations of determinands were detected in the aqueous phase after centrifugation, although the greater quantities were associated with solids.

4.4. Removal of PCB and OCL during primary sedimentation

4.4.1. The removal of SS, NVSS, COD and filtered COD during primary sedimentation pilot plant experiments.

Throughout the pilot plant experiments, SS, NVSS, COD and filtered COD were monitored. The analytical data for influent, effluent and sludge samples are presented in Table 12. It was considered necessary to conduct these analyses in order that the performance of the pilot plant could be compared with that of full-scale primary sedimentation tanks. In addition it was intended to attempt correlation of organochlorine insecticides and PCB removals with those of SS, NVSS, COD and filtered COD for the purpose of more fully understanding the behaviour of these micropollutants.

Removals and mass balances were calculated using equations (1) and (2) respectively and are presented in Table 13.

$$\text{Removal (\%)} = 100 \frac{[\text{influent}] - [\text{effluent}]}{[\text{influent}]}, \quad (1)$$

$$\text{Mass balance (\%)} = 100 \frac{[\text{effluent}] + ([\text{sludge}] \times \text{tank volume} / \text{total flow volume})}{[\text{influent}]} \quad (2)$$

Table 12. Concentrations of SS, NVSS, COD and filtered COD in pilot plant samples.

		0.5 DWF				DWF				3 DWF				5 DWF			
		SS ₀	SS ₁	SS ₂	SS ₃	SS ₀	SS ₁	SS ₂	SS ₃	SS ₀	SS ₁	SS ₂	SS ₃	SS ₀	SS ₁	SS ₂	SS ₃
SS	Influent	438	565	713	981	331	390	667	858	408	792	822	994	418	442	651	767
mg l ⁻¹	Effluent	130	146	166	227	125	143	242	304	217	245	267	322	208	196	263	258
	Sludge	2600	3720	5220	9040	1905	4465	4800	8900	3940	9980	9340	6660	5280	4550	7450	7240
NVSS	Influent	88	120	159	194	84	97	180	229	67	157	153	178	104	129	167	149
mg l ⁻¹	Effluent	51	57	59	72	59	63	127	160	57	61	74	70	66	94	119	67
	Sludge	272	680	940	1560	385	750	1200	4800	800	1940	2120	1000	940	1300	1800	1160
COD	Influent	690	749	957	1496	434	571	881	1138	611	892	922	1317	724	935	834	1096
mg l ⁻¹	Effluent	330	325	348	497	246	272	349	475	449	552	530	702	434	394	518	579
	Sludge	3500	5950	3850	12850	2190	5200	7700	15800	5900	9300	17700	9200	5400	5800	9550	12300
filtered	Influent	144	140	147	191	101	97	141	190	111	162	170	193	163	126	151	197
COD	Effluent	145	134	136	205	85	106	96	124	122	139	151	152	136	95	94	158
mg l ⁻¹	Sludge	400	820	320	960	108	272	1150	1800	780	600	940	580	309	430	370	290

SS₀ denotes raw sewage SS₁, SS₂ and SS₃ denote increasing SS fortifications of raw sewage.

Table 13. Percentage removals and mass balances for SS, NVSS, COD and filtered COD during primary sedimentation pilot plant experiments.

		0.5 DWF				DWF				3 DWF				5 DWF			
		SS ₀	SS ₁	SS ₂	SS ₃	SS ₀	SS ₁	SS ₂	SS ₃	SS ₀	SS ₁	SS ₂	SS ₃	SS ₀	SS ₁	SS ₂	SS ₃
SS	Removal	70**	74**	77**	77**	62**	63**	64**	65**	47**	69**	68**	68**	50**	56**	50*	66**
	Mass balance	67	74	85	81	75	109	81	117	108	104	97	72	134	114	130	99
NVSS	Removal	42**	53**	63**	63**	30**	35**	29**	30**	15**	61**	51**	61**	37**	27**	29**	55**
	Mass balance	78	89	87	88	100	114	112	234	153	111	127	73	124	141	156	99
COD	Removal	52**	57**	64**	67**	43**	52**	60**	58**	27**	38**	43**	47**	40**	58**	38**	47**
	Mass balance	80	102	70	87	89	105	94	150	128	123	115	95	110	84	152	131
filtered	Removal	-1	4	8	-7	16*	-9	32**	35	-10	14*	11*	21*	17*	25**	38**	20**
COD	Mass balance	118	139	111	139	91	127	119	139	150	107	120	97	96	99	81	90

All values are expressed as percentages.

* Indicates that removals are significant at the 5% level

** Indicates that removals are significant at the 1% level.

Mass balances were calculated as a check on percentage removals. Generally, the mass balances of SS, NVSS, COD and filtered COD were acceptable.

The ranges of percentage removals for SS, NVSS, and COD were as follows: SS 47-77 (average 65), NVSS 15-63 (average 43) and COD 27-67 (average 52). The removals of these determinants were statistically significant in all experiments. Generally the percentage removals of SS, NVSS and COD were higher at elevated influent suspended solids concentrations but decreased with increasing hydraulic load. The removal of filtered COD was up to 38% although at low flow rates it tended to be insignificant. The percentage removal of filtered COD seemed independent of influent suspended solids concentration but was enhanced by greater flow rates. The effect of increasing hydraulic load on the removal of SS at various influent suspended solids concentrations has been summarised in Table 14. The average percentage

Table 14. Percentage removals of SS during primary sedimentation pilot plant experiments under varying SS and hydraulic loadings.

	0.5 DWF	DWF	3 DWF	5 DWF
SS ₀	70.3	62.2	46.8	50.2
SS ₁	74.2	63.3	69.4	55.7
SS ₂	76.7	63.7	67.5	59.6
SS ₃	76.9	64.6	67.6	66.4
Average	74.5	63.5	62.8	58.0

removals of SS at the four flow rates have been calculated and are graphically represented in Figure 5. The range of average removal of SS is 58-75% and between overflow rates corresponding to 1 DWF- 5 DWF the average percentage removal for the SS varied by only 6%.

The behaviour of SS during the series of pilot plant experiments has also been assessed by relating influent suspended solids concentrations and the suspended solids concentrations removed at each hydraulic load. A summary of linear regression analyses performed on the data is presented in Table 15. There is a strong correlation bet-

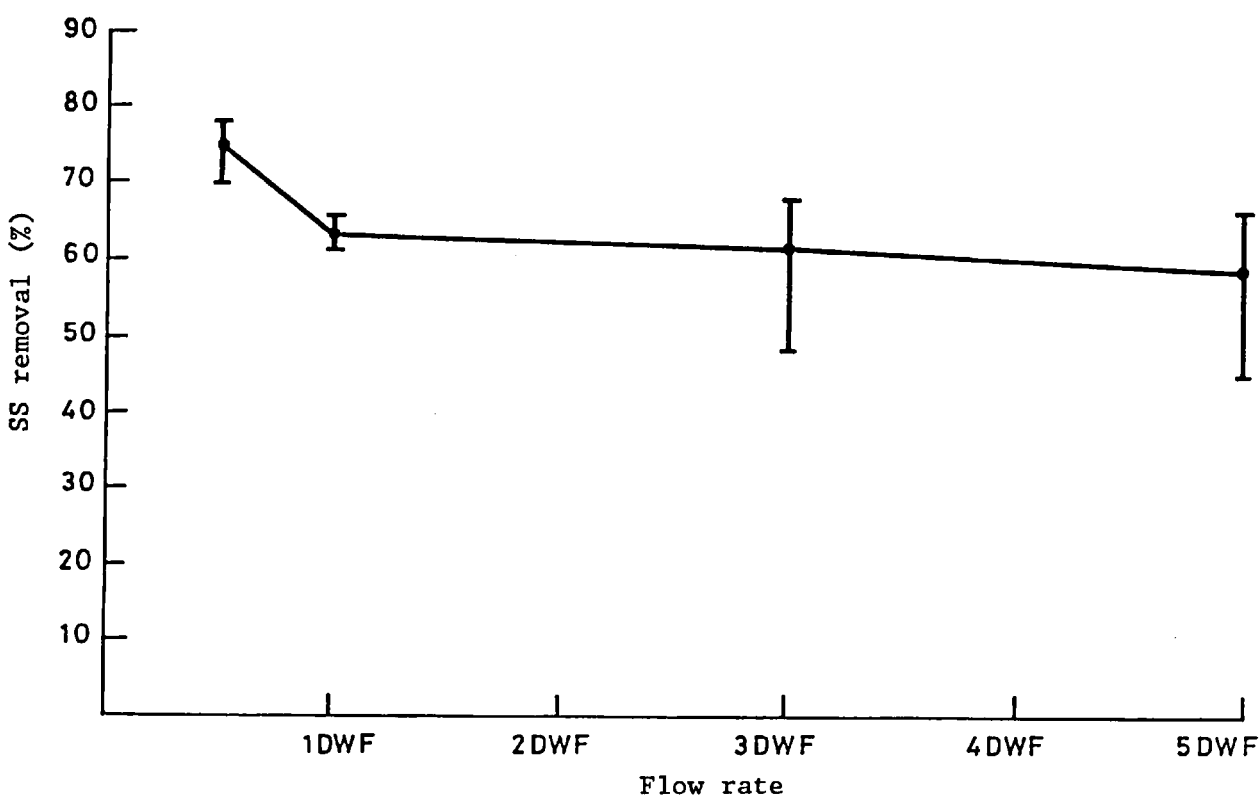


Figure 5. Average SS removal during primary sedimentation pilot plant experiments at various hydraulic loadings

Table 15. Linear regression analyses of the concentrations of suspended solids removed in mg l^{-1} , versus influent concentrations of suspended solids, in mg l^{-1} .

Flow rate	Constants		Regression coefficient	Intercept of x-axis, mg l^{-1}
	a	b		
0.5 DWF	-45.89	0.82	0.999	56
DWF	-10.76	0.66	1.000	16
3 DWF	-149.78	0.84	0.996	168
5 DWF	-124.62	0.81	0.995	154

ween the concentration of suspended solids removed and the influent concentration of suspended solids, demonstrated by regression coefficients of 0.995-1.000. The gradients of the lines are similar, with no tendency to steepen with decreasing flow. The intercept of the x-axis, which can be considered to represent the concentration of non-settleable solids, is similar between 3 DWF and 5 DWF. However at 0.5 DWF and 1 DWF the intercepts of the lines of best fit yield much lower values of estimated concentration of non-settleable solids.

4.4.2. Removal of PCB and OCL during primary sedimentation pilot plant experiments at varying suspended solids concentration and surface loading

In order to investigate the factors which may influence the removal of PCB and OCL in the primary sedimentation process, the pilot plant was operated under four different influent suspended solids loadings at four different hydraulic loadings, as described in Section 3.1.4. Concentrations of PCB and OCL in the samples, percentage removals and mass balances calculated for each of the experiments are included in Tables 16 to 31. Closer examination of the percentage removals of PCB, γ -HCH, aldrin, dieldrin and endrin is facilitated by a summary of these data in Table 32, together with suspended solids removals for comparative purposes. Over the range of experiments the removal of γ -HCH varied between 14.3% at SS, solids loading under 0.5 DWF flow conditions and 49.4% at SS, solids loading under the 3 DWF flow regime.

Table 16. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 438 mg l⁻¹ suspended solids (SS_O) at 0.5 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	137.0	108.0	98.7	118.0	587.0
Effluent sample composite 1	106.0	85.0	34.3	104.8	307.8
Influent sample composite 2	67.3	73.1	69.9	58.0	427.7
Effluent sample composite 2	43.0	72.9	53.7	50.0	316.0
Influent sample composite 3	71.1	87.2	75.0	96.0	226.4
Effluent sample composite 3	45.3	81.8	72.0	72.0	222.4
Influent sample composite 4	59.2	67.7	109.0	82.0	489.5
Effluent sample composite 4	42.7	55.3	46.5	54.5	237.0
Influent sample composite 5	67.0	70.9	62.1	77.1	395.0
Effluent sample composite 5	41.3	62.3	50.1	63.8	300.1
Influent arithmetic mean	80.4	81.4	82.9	86.2	425.1
Effluent arithmetic mean	55.7	71.5	51.3	69.0	276.6
Percentage Efficiency Removal (%)	30.7	12.2	38.1	20.0	34.9
Concentration in sludge (ng l ⁻¹)	237.6	168.4	463.6	494.8	1,614.0
Mass balance (%)	120.4	105.2	108.4	90.8	118.7

Table 17. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 565 mg l⁻¹ suspended solids (SS₁) at 0.5 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	49.1	8.8	159.1	75.2	391.0
Effluent sample composite 1	39.4	5.3	89.0	78.6	268.4
Influent sample composite 2	92.0	6.9	53.9	98.1	544.6
Effluent sample composite 2	79.6	4.7	49.2	72.0	321.0
Influent sample composite 3	55.7	9.8	49.2	79.2	196.7
Effluent sample composite 3	66.6	4.1	29.3	62.8	161.1
Influent sample composite 4	90.4	2.6	31.4	75.2	297.1
Effluent sample composite 4	75.0	1.5	14.9	30.0	208.8
Influent sample composite 5	91.4	5.3	70.9	50.2	527.0
Effluent sample composite 5	64.0	4.8	62.4	46.2	231.2
Influent arithmetic mean	75.7	6.7	72.9	75.6	391.3
Effluent arithmetic mean	64.9	4.1	49.0	57.9	238.1
Percentage Efficiency Removal (%)	14.3	38.8	32.8	23.4	39.2
Concentration in sludge (ng l ⁻¹)	143.6	35.4	649.0	599.6	2,002.6
Mass balance (%)	107.6	107.0	80.6	79.3	108.7

Table 18. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 713 mg l⁻¹ suspended solids (SS₂) at 0.5 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	109.0	4.2	52.4	102.3	234.0
Effluent sample composite 1	78.1	3.2	37.9	67.9	101.1
Influent sample composite 2	77.6	6.2	59.0	87.2	329.1
Effluent sample composite 2	38.2	3.3	44.0	62.8	125.2
Influent sample composite 3	112.2	6.2	70.1	97.3	240.4
Effluent sample composite 3	92.0	2.2	28.9	67.0	141.8
Influent sample composite 4	108.8	4.8	97.0	85.3	173.1
Effluent sample composite 4	95.2	3.2	85.2	69.7	142.0
Influent sample composite 5	74.7	8.6	95.1	116.0	303.5
Effluent sample composite 5	41.2	3.5	70.4	100.8	281.0
Influent arithmetic mean	96.5	6.0	74.7	97.4	255.9
Effluent arithmetic mean	68.9	3.1	53.2	73.6	158.2
Percentage Efficiency Removal (%)	28.6	48.3	28.8	24.4	38.2
Concentration in sludge (ng l ⁻¹)	226.4	60.0	379.2	352.5	2,252.2
Mass balance (%)	121.3	86.1	99.1	105.2	85.1

Table 19. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 981 mg l⁻¹ suspended solids (SS₃) at 0.5 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	105.4	99.8	121.6	109.3	469.2
Effluent sample composite 1	83.7	96.1	110.0	99.5	435.2
Influent sample composite 2	161.0	70.5	139.2	100.0	505.8
Effluent sample composite 2	69.9	40.0	41.4	98.6	238.2
Influent sample composite 3	154.0	58.7	128.8	99.0	324.1
Effluent sample composite 3	72.4	37.6	80.9	70.4	231.4
Influent sample composite 4	181.0	77.0	101.0	103.5	306.8
Effluent sample composite 4	96.4	78.8	64.6	93.1	264.4
Influent sample composite 5	89.4	89.0	113.5	63.5	315.8
Effluent sample composite 5	52.2	92.4	84.9	45.6	217.2
Influent arithmetic mean	138.1	79.0	120.8	95.1	384.3
Effluent arithmetic mean	74.9	69.0	76.4	81.4	277.3
Percentage Efficiency Removal (%)	45.8	12.7	36.8	14.4	27.8
Concentration in sludge (ng l ⁻¹)	860.0	210.4	1,294.0	312.7	1,430.0
Mass balance (%)	112.5	101.8	80.2	99.6	110.6

Table 20. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 331 mg l⁻¹ suspended solids (SS_O) at 1 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	57.5	2.0	57.9	N.D.	31.5
Effluent sample composite 1	32.5	0.5	26.7	N.D.	30.4
Influent sample composite 2	36.4	1.2	60.0	N.D.	19.7
Effluent sample composite 2	25.2	N.D.	11.0	N.D.	20.0
Influent sample composite 3	31.6	1.6	38.3	N.D.	26.1
Effluent sample composite 3	18.2	N.D.	8.3	N.D.	28.7
Influent sample composite 4	52.2	1.7	34.5	N.D.	57.2
Effluent sample composite 4	39.2	0.3	19.3	N.D.	59.0
Influent sample composite 5	60.0	1.0	21.0	N.D.	39.7
Effluent sample composite 5	44.0	0.3	14.7	N.D.	20.0
Influent arithmetic mean	47.7	1.5	42.3	N.D.	34.8
Effluent arithmetic mean	31.8	0.3	16.0	N.D.	31.6
Percentage Efficiency Removal (%)	33.3	80.0	62.2	-	9.2
Concentration in sludge (ng l ⁻¹)	266.0	16.0	315.0	-	N.D.
Mass balance (%)	101.1	112.3	118.8	-	-

Table 21. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 390 mg l⁻¹ suspended solids (SS₁) at 1 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	48.0	1.1	48.3	N. D.	57.9
Effluent sample composite 1	31.4	0.5	24.5	N. D.	52.2
Influent sample composite 2	37.5	1.8	44.0	N. D.	35.5
Effluent sample composite 2	29.7	0.5	25.9	N. D.	34.8
Influent sample composite 3	33.1	2.2	53.8	N. D.	34.4
Effluent sample composite 3	20.0	0.3	11.0	N. D.	20.0
Influent sample composite 4	63.4	2.8	59.2	N. D.	106.8
Effluent sample composite 4	43.2	0.5	27.0	N. D.	67.0
Influent sample composite 5	65.5	2.5	69.0	N. D.	50.0
Effluent sample composite 5	51.3	0.7	23.0	N. D.	42.3
Influent arithmetic mean	49.5	2.1	54.9	N. D.	56.9
Effluent arithmetic mean	35.1	0.5	22.3	N. D.	43.3
Percentage Efficiency Removal (%)	29.1	76.2	59.4	-	23.9
Concentration in sludge (ng l ⁻¹)	460.0	19.7	414.0	-	N. D.
Mass balance (%)	81.2	115.1	111.3	-	-

Table 22. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 667 mg l⁻¹ suspended solids (SS₂) at 1 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	50.4	2.4	58.4	22.0	75.2
Effluent sample composite 1	25.2	2.0	23.6	15.2	51.2
Influent sample composite 2	66.4	2.8	65.4	22.0	75.2
Effluent sample composite 2	38.0	1.6	31.6	10.8	35.6
Influent sample composite 3	43.6	3.2	37.6	26.8	64.0
Effluent sample composite 3	39.2	1.6	30.0	8.8	60.4
Influent sample composite 4	35.6	3.6	39.2	13.2	80.4
Effluent sample composite 4	30.8	2.0	20.4	6.4	70.8
Influent sample composite 5	44.0	3.6	39.2	17.6	62.0
Effluent sample composite 5	31.2	1.6	34.4	10.8	22.0
Influent arithmetic mean	48.0	3.1	48.0	20.3	71.4
Effluent arithmetic mean	32.9	1.8	28.0	10.4	48.0
Percentage Efficiency Removal (%)	31.5	41.9	41.7	48.8	32.8
Concentration in sludge (ng l ⁻¹)	550.0	35.0	412.0	136.0	960.0
Mass balance (%)	75.6	82.4	94.7	113.9	116.6

Table 23. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 858 mg l⁻¹ suspended solids (SS₃) at 1 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	67.3	3.6	59.8	26.8	70.4
Effluent sample composite 1	51.6	1.8	40.4	18.0	45.4
Influent sample composite 2	63.2	3.2	49.6	22.0	64.4
Effluent sample composite 2	39.2	1.6	30.0	4.0	48.8
Influent sample composite 3	56.8	2.0	48.8	13.2	35.6
Effluent sample composite 3	40.4	1.6	36.0	13.2	33.6
Influent sample composite 4	32.4	2.8	40.8	14.4	64.4
Effluent sample composite 4	30.4	2.4	26.8	4.4	44.8
Influent sample composite 5	40.4	4.4	49.2	16.4	95.2
Effluent sample composite 5	29.2	2.8	23.6	8.8	85.6
Influent arithmetic mean	52.0	3.2	49.6	25.9	66.0
Effluent arithmetic mean	38.2	2.0	31.4	9.7	51.6
Percentage Efficiency Removal (%)	26.5	37.5	36.7	62.5	21.8
Concentration in sludge (ng l ⁻¹)	495.0	21.0	370.0	206.0	495.0
Mass balance (%)	74.5	96.5	90.4	111.2	80.5

Table 24. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 408 mg l⁻¹ suspended solids (SS_O) at 3 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	30.4	11.5	26.6	9.2	84.8
Effluent sample composite 1	14.6	6.7	26.5	8.0	50.4
Influent sample composite 2	43.0	10.8	39.6	16.2	110.0
Effluent sample composite 2	23.2	8.7	20.5	7.5	67.2
Influent sample composite 3	26.6	13.3	29.3	15.3	78.4
Effluent sample composite 3	30.5	12.7	30.8	11.2	69.7
Influent sample composite 4	71.3	7.3	30.6	8.1	100.8
Effluent sample composite 4	22.2	5.8	14.9	2.8	86.1
Influent sample composite 5	78.4	6.3	32.0	6.2	85.0
Effluent sample composite 5	57.9	5.6	18.4	3.7	82.1
Influent arithmetic mean	49.9	9.8	31.6	11.0	91.8
Effluent arithmetic mean	29.7	7.9	22.2	6.6	71.1
Percentage Efficiency Removal (%)	40.5	19.4	29.7	40.0	22.5
Concentration in sludge (ng l ⁻¹)	235.5	15.5	164.5	51.0	398.0
Mass balance (%)	119.3	117.4	103.3	119.3	101.6

Table 25. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 792 mg l⁻¹ suspended solids (SS₁) at 3 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	56.1	8.3	57.4	28.2	161.3
Effluent sample composite 1	24.3	7.5	23.2	18.1	93.7
Influent sample composite 2	71.1	5.2	46.3	27.5	107.5
Effluent sample composite 2	27.3	4.8	27.9	11.2	50.4
Influent sample composite 3	67.4	7.5	69.2	12.7	123.2
Effluent sample composite 3	30.3	5.3	26.7	8.1	100.6
Influent sample composite 4	35.0	10.0	35.0	8.1	64.7
Effluent sample composite 4	22.0	6.8	29.1	7.6	63.3
Influent sample composite 5	39.9	3.3	42.4	12.7	93.6
Effluent sample composite 5	32.6	3.1	28.5	11.5	81.1
Influent arithmetic mean	54.0	6.9	50.1	17.8	110.1
Effluent arithmetic mean	27.3	5.5	27.1	11.3	77.8
Percentage Efficiency Removal (%)	49.4	20.3	45.9	36.5	29.3
Concentration in sludge (ng l ⁻¹)	315.6	28.8	428.0	138.8	908.0
Mass balance (%)	125.4	101.9	102.1	97.4	89.3

Table 26. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 822 mg l⁻¹ suspended solids (SS₂) at 3 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	47.8	4.2	52.2	29.5	82.0
Effluent sample composite 1	23.7	2.3	21.4	9.6	46.4
Influent sample composite 2	62.7	4.9	67.0	25.6	96.0
Effluent sample composite 2	56.9	2.9	33.8	14.2	74.5
Influent sample composite 3	57.7	6.7	69.5	35.3	99.5
Effluent sample composite 3	50.3	2.8	56.0	17.8	80.0
Influent sample composite 4	43.2	2.9	48.6	28.2	76.2
Effluent sample composite 4	38.9	2.7	31.4	19.9	62.0
Influent sample composite 5	41.5	3.0	44.4	21.2	96.8
Effluent sample composite 5	27.0	2.9	34.6	18.1	64.5
Influent arithmetic mean	50.6	4.3	56.3	28.0	90.1
Effluent arithmetic mean	39.4	2.7	45.9	15.9	65.5
Percentage Efficiency Removal (%)	22.1	37.2	18.5	43.2	27.3
Concentration in sludge (ng l ⁻¹)	283.6	23.0	392.0	188.0	550.0
Mass balance (%)	96.4	113.5	87.4	111.5	98.5

Table 27. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 994 mg l⁻¹ suspended solids (SS₃) at 3 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	55.8	6.9	64.6	16.6	95.5
Effluent sample composite 1	34.4	8.3	75.0	16.2	88.0
Influent sample composite 2	70.8	8.1	106.6	31.8	90.0
Effluent sample composite 2	40.9	6.7	99.2	16.2	87.1
Influent sample composite 3	57.6	9.4	70.2	13.0	110.4
Effluent sample composite 3	35.4	9.5	41.0	6.9	102.5
Influent sample composite 4	100.6	4.6	100.8	20.2	135.1
Effluent sample composite 4	47.4	3.0	59.4	13.0	117.3
Influent sample composite 5	90.6	3.2	61.8	20.2	89.5
Effluent sample composite 5	64.4	3.2	34.4	15.0	68.8
Influent arithmetic mean	75.1	6.4	80.8	20.4	104.1
Effluent arithmetic mean	44.5	6.1	61.8	13.5	92.7
Percentage Efficiency Removal (%)	40.7	4.7	23.5	33.8	11.0
Concentration in sludge (ng l ⁻¹)	336.0	19.8	310.8	62.4	675.0
Mass balance (%)	121.4	92.6	105.5	124.3	81.8

Table 28. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 418 mg l⁻¹ suspended solids (SS_O) at 5 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	50.7	9.9	95.5	42.6	68.0
Effluent sample composite 1	38.4	8.0	59.0	24.3	61.5
Influent sample composite 2	60.5	5.8	55.4	19.7	102.2
Effluent sample composite 2	15.0	4.6	30.2	19.7	60.8
Influent sample composite 3	54.6	7.8	88.2	18.8	118.4
Effluent sample composite 3	17.3	7.0	53.1	11.7	84.0
Influent sample composite 4	72.4	7.4	105.3	33.2	78.0
Effluent sample composite 4	53.4	6.6	66.1	35.2	71.6
Influent sample composite 5	44.2	13.4	93.4	39.0	89.6
Effluent sample composite 5	14.4	12.3	61.3	22.1	80.0
Influent arithmetic mean	56.5	8.9	87.6	30.7	91.2
Effluent arithmetic mean	31.0	7.7	53.9	22.6	71.6
Percentage Efficiency Removal (%)	45.1	13.5	38.5	26.4	21.5
Concentration in sludge (ng l ⁻¹)	599.0	27.2	1,014.0	253.0	558.0
Mass balance (%)	79.0	97.9	71.6	78.7	85.8

Table 29. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 442 mg l⁻¹ suspended solids (SS₁) at 5 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	71.6	20.1	73.8	93.6	729.1
Effluent sample composite 1	41.8	16.4	40.4	7.4	163.4
Influent sample composite 2	88.2	21.7	85.6	21.8	574.4
Effluent sample composite 2	56.8	10.2	48.2	31.6	129.3
Effluent sample composite 3	83.2	29.8	49.4	42.0	654.0
Influent sample composite 3	56.0	15.1	36.6	21.8	524.7
Influent sample composite 4	47.4	31.4	95.0	24.4	592.5
Effluent sample composite 4	57.8	17.6	36.0	9.3	184.4
Influent sample composite 5	55.2	13.2	47.0	15.0	508.5
Effluent sample composite 5	53.6	11.3	37.2	21.8	501.3
Influent arithmetic mean	69.1	23.2	70.2	39.4	582.0
Effluent arithmetic mean	53.2	14.1	39.7	18.4	343.0
Percentage Efficiency Removal (%)	23.0	39.3	43.4	53.3	41.0
Concentration in sludge (ng l ⁻¹)	451.0	53.3	576.0	410.0	1,787.8
Mass balance (%)	97.7	136.8	88.5	83.3	129.3

Table 30. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 651 mg l⁻¹ suspended solids (SS₂) at 5 DWF.

Parameter	γ -HCH (ng l ⁻¹)	aldrin (ng l ⁻¹)	dieldrin (ng l ⁻¹)	endrin (ng l ⁻¹)	PCB (ng l ⁻¹)
Influent sample composite 1	71.4	18.8	92.0	47.6	525.6
Effluent sample composite 1	52.6	14.4	45.8	21.8	393.0
Influent sample composite 2	83.8	14.9	89.6	53.5	649.0
Effluent sample composite 2	65.8	11.3	50.0	27.6	553.2
Influent sample composite 3	90.2	27.1	65.8	16.0	790.0
Effluent sample composite 3	67.6	17.5	57.8	47.4	570.3
Influent sample composite 4	85.0	21.3	79.8	56.2	724.0
Effluent sample composite 4	73.2	26.7	47.6	44.6	181.7
Influent sample composite 5	73.2	14.2	72.8	30.4	877.7
Effluent sample composite 5	61.6	12.6	45.8	13.2	459.0
Influent arithmetic mean	80.7	19.3	80.0	40.7	694.1
Effluent arithmetic mean	64.2	16.5	49.4	30.9	431.4
Percentage Efficiency Removal (%)	20.4	14.5	38.3	24.1	37.8
Concentration in sludge (ng l ⁻¹)	348.0	53.3	387.0	209.0	1,429.0
Mass balance (%)	93.5	99.6	105.6	90.9	136.3

Table 31. Influent and effluent concentrations, percentage removal and mass balance for γ -HCH, aldrin, dieldrin, endrin and PCB during primary sedimentation of raw sewage containing 767 mg l^{-1} suspended solids (SS_3) at 5 DWF.

Parameter	γ -HCH	aldrin	dieldrin	endrin	PCB
Influent sample composite 1	60.2	6.1	52.8	30.4	90.0
Effluent sample composite 1	37.6	5.3	14.6	17.4	56.4
Influent sample composite 2	63.4	4.7	54.0	24.6	121.4
Effluent sample composite 2	52.3	5.1	15.3	27.9	97.8
Influent sample composite 3	100.6	4.3	119.4	18.8	172.5
Effluent sample composite 3	57.8	4.2	44.2	24.3	71.0
Influent sample composite 4	117.6	2.2	98.7	60.4	91.6
Effluent sample composite 4	78.9	1.8	63.3	63.5	97.5
Influent sample composite 5	87.7	4.0	95.2	39.3	93.5
Effluent sample composite 5	66.8	3.9	68.0	24.6	74.7
Influent arithmetic mean	85.9	4.3	84.0	34.7	113.8
Effluent arithmetic mean	58.7	4.1	41.1	31.5	79.5
Percentage Efficiency Removal (%)	31.7	4.7	51.1	9.2	30.1
Concentration in sludge (ng l^{-1})	986.0	41.0	1,159.2	286.0	648.0
Mass balance (%)	69.8	70.7	71.0	70.1	95.8

Table 32. Removal efficiencies of SS, PCB, γ -HCH, aldrin, dieldrin and endrin during the pilot plant experiments.

		Removal efficiency, %					
Flow		SS	γ -HCH	aldrin	dieldrin	endrin	PCB
0.5 DWF	SS ₀	70	30.7	12.2	38.1	20.0	34.9
	SS ₁	74	14.3	38.8	32.8	23.4	39.2
	SS ₂	77	28.6	48.3	28.8	24.4	38.2
	SS ₃	77	45.8	12.7	36.8	14.4	27.8
1 DWF	SS ₀	62	33.3	80.0	62.2	—	9.2
	SS ₁	63	29.1	76.2	59.4	—	23.9
	SS ₂	64	31.5	41.9	41.7	48.8	32.8
	SS ₃	65	26.5	37.5	36.7	62.5	21.8
3 DWF	SS ₀	47	40.5	19.4	29.7	40.0	22.5
	SS ₁	69	49.4	20.3	45.9	36.5	29.3
	SS ₂	68	22.1	37.2	18.5	43.2	27.3
	SS ₃	68	40.7	4.7	23.5	33.8	11.0
5 DWF	SS ₀	50	45.1	13.5	38.5	26.4	21.5
	SS ₁	56	23.0	39.3	43.4	53.3	41.0
	SS ₂	50	20.4	14.5	38.3	24.1	37.8
	SS ₃	66	31.7	4.7	51.1	9.2	30.1

Overall, the percentage removals did not follow a similar pattern to those of suspended solids although there is a suggestion that generally higher removals were obtained at the SS_0 solids loadings under the four different flow regimes. The absence of any apparent relationship between the removal of γ -HCH and suspended solids removal may be explained by the relatively high aqueous solubility of this compound ($7,800 \mu\text{g l}^{-1}$) and the generally low average concentrations ($40\text{--}130 \text{ ng l}^{-1}$) detected in the influent raw sewage during the course of these studies, such that a substantial proportion of the γ -HCH present in the raw sewage may have been in solution and therefore not amenable to removal by simple mechanical separation by settling. An additional possible explanation is the association of a proportion of γ -HCH with non-settleable solids; those of low density and small size which cannot be removed under the normal operational conditions of conventional primary sedimentation.

The removal of aldrin varied between 4.7% on two occasions SS_3 solids loading at 3 DWF and 5 DWF and 80.0% at SS_0 solids loading under 1 DWF flow conditions. In general, removals tended to be higher at the two lower hydraulic loadings (0.5 DWF and 1 DWF) and low on average, at 3 DWF and 5 DWF. This is in broad agreement with the lower suspended solids removals observed at the higher flow rates, although it is also evident that there is little agreement with suspended solids removals at different solids loading rates, with the exception of the 1 DWF experiment, where the removal efficiency of aldrin decreased with increments in solids loading. Overall, the poorest removal efficiencies were observed at the highest solids loadings (SS_3). Dieldrin removal was in the range 18.5% at SS_2 solids loading with 3 DWF hydraulic loading to 62.2% at SS_0 solids loading and 1 DWF hydraulic loading. The highest removals were observed throughout the 1 DWF experiment and, as in the case of aldrin, removal increased with incremental solids loading. With the exception of the 5 DWF experiment, where removals of dieldrin followed the general pattern of solids removal at the differing solids loadings, no particular relationship between dieldrin and suspended solids removals was observed. The aqueous solubility of dieldrin is approximately $200 \mu\text{g l}^{-1}$, whilst concentrations detected in the influent raw sewage to the primary sedimentation pilot plant during the course of these experiments were in the range $20\text{--}150 \text{ ng l}^{-1}$. It is perhaps possible, therefore to draw the facile conclusion that all the dieldrin present

could have been in the soluble form and not amenable to removal by sedimentation, although in practice, an equilibrium will be attained between aqueous and solid phases, resulting in removal of a proportion of the dieldrin attached to solids.

The percentage removals of endrin during the pilot plant experiments varied between 9.2% at SS₂ solids loading under 5 DWF flow conditions and 62.5% at SS₃ solids loading under 1 DWF hydraulic loading. The highest removals were observed to occur during the 1 DWF and 3 DWF experiments, whilst the lowest occurred at 0.5 DWF hydraulic loading. Little similarity was apparent between the removals of endrin and those of suspended solids with the exception of the 0.5 DWF experiment, where the general pattern of solids removals at different solids loadings was reflected in the variation of endrin removals. Endrin possesses a similar aqueous solubility to that of dieldrin and it is apparent that a significant portion of this compound present in the influent raw sewage to the sedimentation tank occurred in a form which was not amenable to removal under the prevailing operational conditions.

PCB removals during the pilot plant experiments varied between 9.2% at SS₀ solids loading under 1 DWF flow conditions and 41.0% at SS₁ solids loading at 5 DWF hydraulic loading. The highest average removals were observed to occur during the 5 DWF experiments and the lowest under 5 DWF flow conditions. Removals of PCB were broadly independent of suspended solids removals and no relationship with hydraulic loading was observed. The PCB formulation detected in the samples obtained from the pilot plant (Arochlor 1260) consists of many individual chlorobiphenyl isomers containing from 4 to 9 chlorine atoms, such that quantification of individual compounds in the mixture is not possible. Therefore, the removal efficiencies calculated here were derived on the basis of an average for all the isomers. Since the aqueous solubilities of the compounds will vary inversely with chlorine content, it is evident that varying proportions of the different isomers will be present in the raw sewage in differing forms and may be removed to varying degrees under changing conditions of solids and hydraulic loadings, thus influencing the calculated removal efficiencies.

A further summary of the results of the primary sedimentation pilot plant experiments is presented in Table 33, where the removal efficiencies of suspended solids, PCB, γ -HCH, aldrin, dieldrin and endrin have been averaged over the range of suspended solids loadings for each hydraulic loading. Average removals of γ -HCH over the range of hydraulic loadings did not vary excessively and fell between 29.9% and 38.2% with an overall mean removal of 32.1%. Of the five determinands analysed during this study, γ -HCH displayed the least variation in removal efficiency over the range of pilot plant operational conditions, which perhaps reinforces the hypothesis that a significant proportion of γ -HCH in raw sewage will be in the dissolved form and/or associated with non-settleable solids. Aldrin, dieldrin and endrin all exhibited maximum average removals during the 1 DWF experiments, the reasons for which are not evident. The overall average removal of dieldrin (39.1%) was the highest for all five determinands during the experimental programme. Average aldrin removals displayed the greatest variability (18.0% to 58.9%) over the range of hydraulic loadings, with those of dieldrin (29.4% to 50.0%) and endrin (20.6% to 51.9%) exhibiting a somewhat lesser degree of fluctuation. Average removals of PCB varied between 21.9% and 35.0% over the range of flow conditions employed and, with an overall average removal of 28.0% was the determinand most inefficiently removed during the primary sedimentation pilot plant experiments.

No correlations were observed between the removals of any of the five determinands and the removals of non-volatile and volatile suspended solids, total and soluble COD and it is also evident that hydraulic and solids loadings did not display any relationship with the removal of PCB and OCL. It appears that, at the ng l⁻¹ concentrations detected in raw sewage during the course of this study, PCB and OCL were associated with a predominantly non-settleable solids fraction or were in solution in the raw sewage.

4.4.3. Variation in influent γ -HCH and dieldrin concentrations and their effects upon removal

γ -HCH and dieldrin were not dosed into the influent to the primary sedimentation pilot plant during the course of these experiments, as their concentrations in raw sewage entering the Hogsmill Valley Water Pollution Control Works were sufficient to permit their

Table 33. Average removal efficiencies of suspended solids, PCB and OCL at different hydraulic loadings to the primary sedimentation pilot plant.

Hydraulic loading	Suspended Solids	γ-HCH	Aldrin	Dieldrin	Endrin	PCB
0.5 DWF	75.0	29.9	28.0	34.1	20.6	35.0
1 DWF	63.6	30.1	58.9	50.0	51.9	21.9
3 DWF	63.7	38.2	20.4	29.4	38.4	22.5
5 DWF	59.1	30.1	18.0	42.8	28.3	32.6
Overall mean removal effi- ciencies %	65.4	32.1	31.3	39.1	34.8	28.0

detection in both influent to and effluent from the pilot plant. It was considered that examination of the variation in the background indigenous concentrations of these two determinands without solids dosing (SS_0), in raw and settled sewage might allow an understanding in the variation of their removals during the primary sedimentation experiments. These results are included in Figures 6 and 7, for γ -HCH and dieldrin respectively, for each hydraulic loading regime employed.

During the 0.5 DWF and 1 DWF experiments, concentrations of γ -HCH in the influent and effluent followed a very similar pattern, with a virtually constant mass of this determinand being removed into the primary sludge. A greater degree of variation was evident during the 3 DWF and 5 DWF experiments and the highest removals were achieved at these hydraulic loadings. The concentrations in the raw sewage varied between approximately 20 ng l^{-1} and 140 ng l^{-1} over the four experiments and the removal efficiencies increased from 30.7% at 0.5 DWF through to 45.1% at 5 DWF. Concentrations of dieldrin measured in the influent to and effluent from the pilot plant did not follow similar trends, with the exception of the 3 DWF hydraulic loading, when low concentrations $< 40 \text{ ng l}^{-1}$ were detected in both influent and effluent throughout the experiment. Similar percentage removals for dieldrin of 38.1% and 38.5% were recorded during the 0.5 DWF and 5 DWF experiments respectively, when the influent concentrations exhibited a similar magnitude and degree of variation. The highest removal of dieldrin (62.2%) was attained during the 1 DWF experiment, where concentrations in the raw sewage influent to the pilot plant varied between approximately 20 ng l^{-1} and 60 ng l^{-1} and decreased gradually over the period of the experiment. The poorest dieldrin removal efficiency was recorded for the 3 DWF experiment (29.7%), when low concentrations were detected in both influent and effluent and when simultaneously the removal of suspended solids also reached a minimum level of 47%.

In general, there appeared to be little relationship between the concentrations of γ -HCH and dieldrin in the raw sewage influent to the pilot plant and the effective removal of these two compounds during primary sedimentation, with perhaps the exception in one instance where the lowest removal efficiency of dieldrin coincided with particularly low influent concentrations and a poor removal of suspended solids. A confounding variable for this type of analyses,

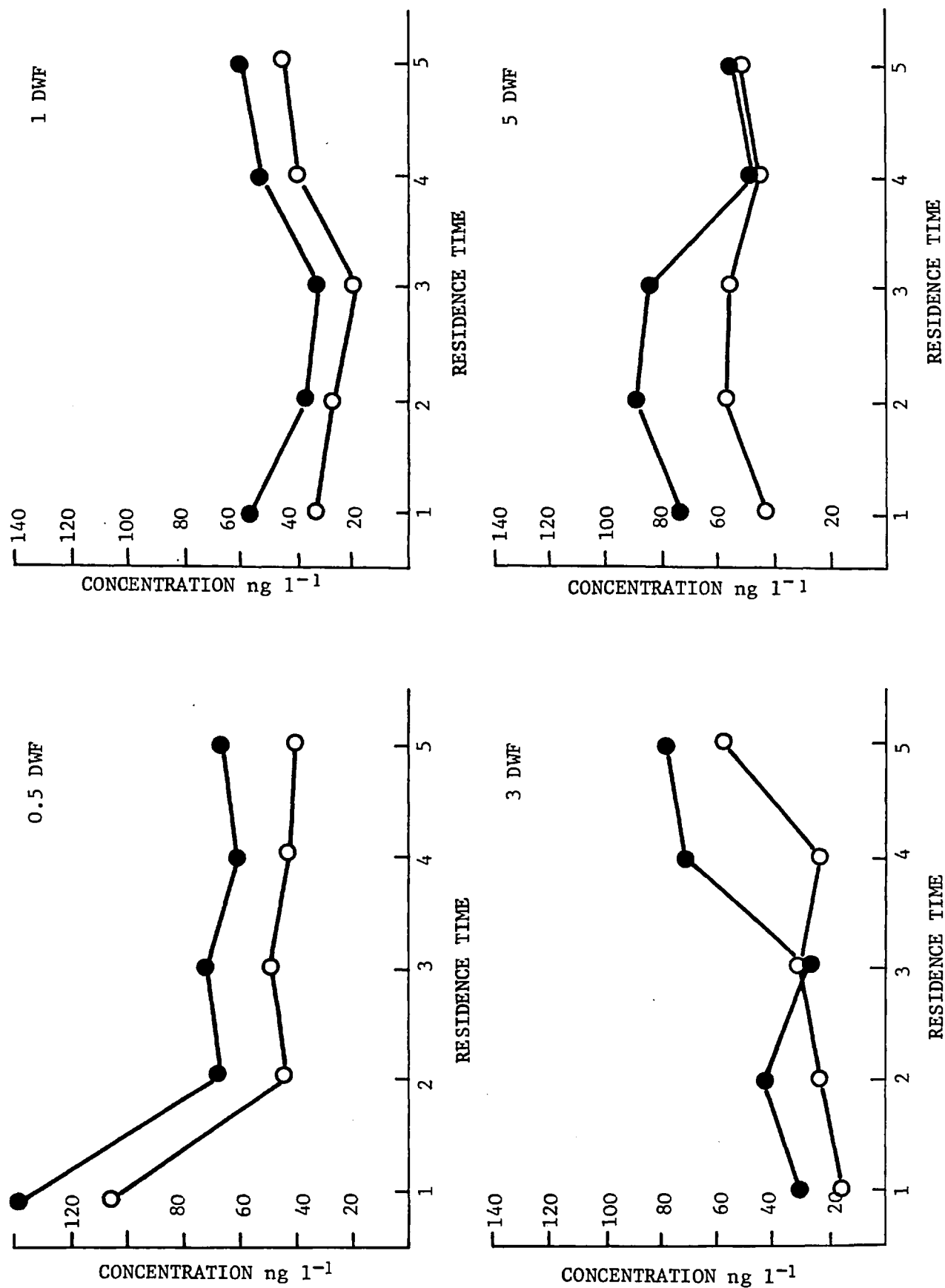


Figure 6. Typical variation in the concentrations of γ -HCH in raw sewage influent (●) to and settled sewage effluent (○) from the primary sedimentation pilot plant, without solids dosing, at different hydraulic loadings.

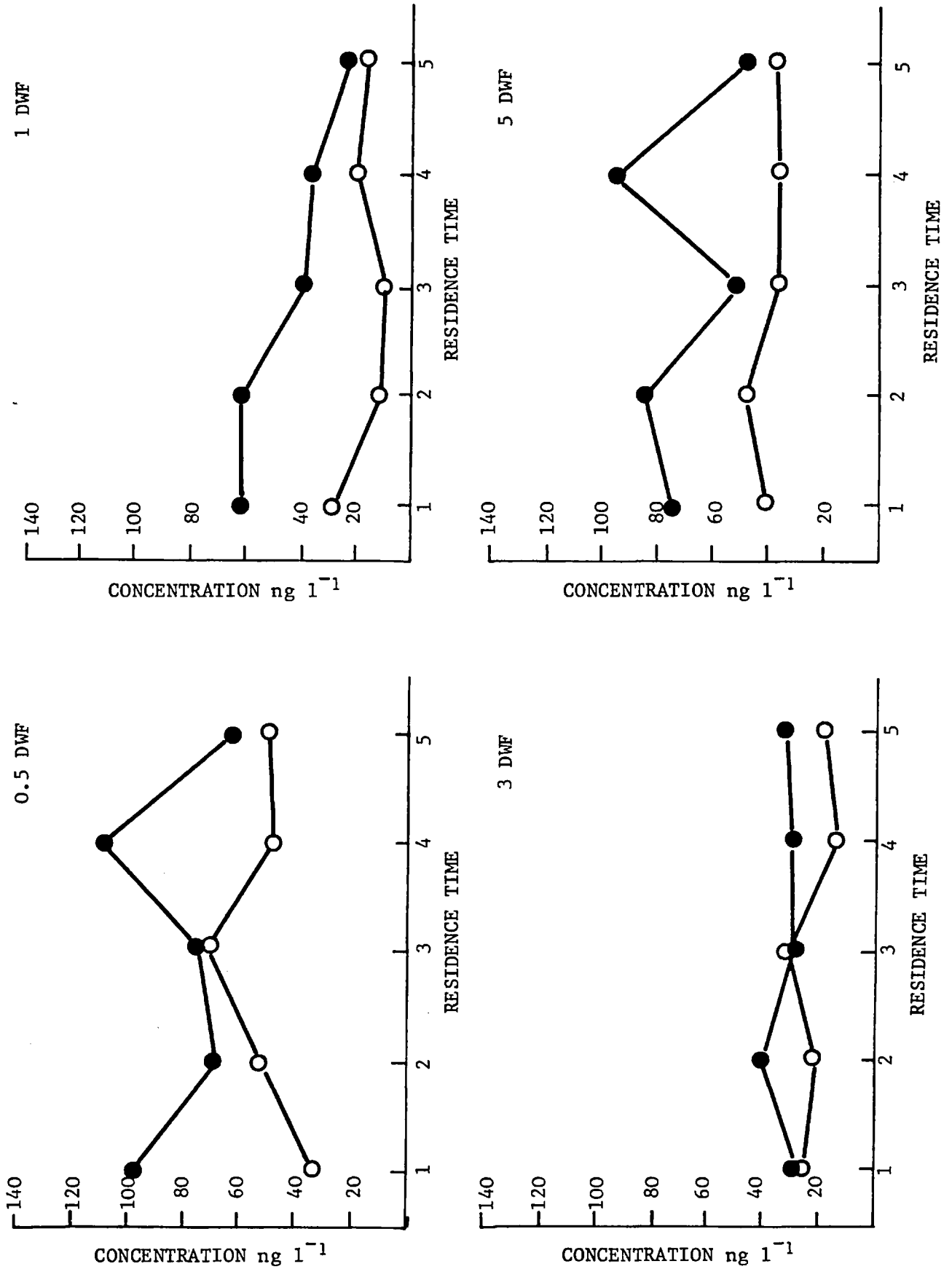


Figure 7. Typical variations in the concentrations of dieldrin in the raw sewage influent (●) to and settled sewage effluent (○) from the primary sedimentation plant, without solids dosing, at different hydraulic loadings.

might, in this case, have been the variation in the nature of the raw sewage characteristics over the period of the individual experiments and the fact that the experiments were run for 10 retention times of the sedimentation tanks at each hydraulic loading, such that any diurnal fluctuations in raw sewage characteristics would not have been experienced at the higher flow rates. This may be confirmed by examination of Table 34, where daily mean raw sewage characteristics (suspended solids, COD and flow) of the influent to the Hogsmill Valley Works during the pilot plant experimental programme are presented. During the 1 DWF experiments, daily mean suspended solids concentrations varied between 286 mg l^{-1} and 362 mg l^{-1} together with variations in COD of 474 mg l^{-1} to 554 mg l^{-1} . At this time, flow to the main works was elevated, such that raw sewage of a dilute nature was received by the pilot plant. During the 3 DWF and 5 DWF experiments, mean concentrations of suspended solids and COD did not vary excessively, with the exception of 5 DWF (ii), when a peak in suspended solids and COD was observed. During the first 0.5 DWF experiment, particularly elevated concentrations of suspended solids ($368\text{--}610 \text{ mg l}^{-1}$) and COD ($774\text{--}860 \text{ mg l}^{-1}$) were observed over the period of study; at this time one of the four primary sedimentation tanks at the main treatment works was inoperable and the contents had been pumped into the raw sewage inlet channel. During the second 0.5 DWF experiment, the main treatment works was operating normally and no excessive fluctuations in suspended solids, COD or flow were observed.

These variations in flow and suspended solids and COD loadings are, however, typical of those which may occasionally be experienced during the normal operation of full size treatment plants and cannot therefore be considered unrepresentative. Although more detailed information could be obtained concerning the effects upon micropollutant removal of these variations in raw sewage quality by increasing the sampling frequency, it is clearly impossible to predict the occurrence of such events and the extended time periods associated with the analysis of large numbers of samples for PCB and OCL are not conducive to the execution of an experimental programme of this nature.

Table 34. Variation in the quality of raw sewage influent to Hogsmill Valley water pollution works over the period of pilot plant experiments.

Pilot plant hydraulic loading		Daily mean SS (mg l ⁻¹)	Daily mean COD (mg l ⁻¹)	Daily mean flow to works l s ⁻¹
1	DWF (i)	324	480	1,056.24
		286	474	944.62
1	DWF (ii)	362	554	804.39
		363	547	740.50
5	DWF (i)	382	680	770.01
5	DWF (ii)	490	767	708.90
3	DWF (i)	328	547	694.55
3	DWF (ii)	348	580	781.70
0.5	DWF (i)	450	774	733.21
		610	994	662.38
		368	860	643.05
0.5	DWF (ii)	390	514	840.27
		360	547	654.74
		346	634	651.74

4.5 Concentrations of PCB and OCL compounds in U.K. sewage sludges

The concentration of chlorinated materials into sewage sludges during primary sedimentation suggests that the potential exists for environmental problems if these sludges are to be disposed to the agricultural land or the marine environment. A survey of these materials in U.K. sewage sludges in the "as disposed of" conditions was therefore undertaken to ascertain the potential magnitude of the problem.

4.5.1 National survey

In all 324 samples were collected from the 10 water authorities in England and Wales. A further 120 from Scotland and Northern Ireland giving a total of 444. Sewage treatment works were divided into six arbitrary classes based upon the population equivalent served and the distribution of sample collected within each region was arranged to avoid bias of sample collection towards large works only. The numbers of each sample in each category for the individual regions can be seen in Table 35. These samples were analysed using the method of McIntyre *et al.* (1980) in conjunction with the further developments outlined in Section 4.2. The results are presented on a region by region basis.

Table 35. Numbers of sludge samples to be collected from each area and from which size of works to be collected.

Population range served by works							
>500- Area	1001- 1000	5001- 5000	10,001- 10,000	50,001- 50,000	100,001- 100,000	Totals 1000,000	
Water Authorities							
Anglian	15	13	4	4	2	2	40
Northumbrian	23	10	2	4	1		40
North West	19	9	4	7	3	3	45
Severn Trent	18	13	4	6	2	2	45
Southern	9	10	3	5	2	1	30
South West	17	8	2	2	1		30
Thames	15	15	4	7	2	2	45
Welsh	23	9	3	3	1	1	40
Wessex	10	12	4	3		1	30
Yorkshire	19	13	5	7	1	1	46
Scotland	13	10	2	3	1	1	30
N. Ireland	18	6	2	3		1	30

4.5.1.1 Anglian Water Authority Area

A statistical summary of the analyses of PCB and OCL in sludges sampled from this area is presented in Table 36 and results of analyses are given in Appendix I (Table 1).

PCB concentrations varied between 0.07 and 21.5 $\mu\text{g g}^{-1}$ with a mean concentration of 0.98 $\mu\text{g g}^{-1}$ and a median value of 0.31 $\mu\text{g g}^{-1}$. The mean:median ratio of 3.16 indicates that the distribution is skewed, with a small number of relatively high values strongly influencing the mean concentration. Non-detectable concentrations were encountered on nine occasions. Concentrations of γ -HCH in these samples lay in the range 0.02 to 1.11 $\mu\text{g g}^{-1}$ with a mean of 0.21 $\mu\text{g g}^{-1}$ and a median value of 0.12 $\mu\text{g g}^{-1}$. The parameters of the distribution are again influenced by a small number of elevated values, notably those of sample numbers 1 and 32.

Aldrin was not detected in any of the 40 samples. Dieldrin was detected in all of the 40 sewage sludge samples, at concentrations of between 0.02 and 1.72 $\mu\text{g g}^{-1}$. The calculated mean value of 0.21 $\mu\text{g g}^{-1}$ is substantially higher than the median concentration of 0.12 $\mu\text{g g}^{-1}$ which is again indicative of a distribution skewed by a few relatively high values. Endrin was only detected in 17 of the samples, where concentrations varied between 0.01 and 0.20 $\mu\text{g g}^{-1}$, with a mean value of 0.07 $\mu\text{g g}^{-1}$ and a median concentration of 0.04 $\mu\text{g g}^{-1}$.

Table 36. Statistical summary of results from Anglian Water Authority Area

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.98	3.39	0.07-21.5	0.31	3.16
γ -HCH	0.21	0.26	0.02-1.11	0.12	1.75
Aldrin	-	-	-	-	-
Dieldrin	0.21	0.29	0.02-1.72	0.12	1.75
Endrin	0.07	0.05	0.01-0.20	0.04	1.75

4.5.1.2. Severn-Trent Water Authority Area

A statistical summary of the analyses of PCB and OCL in sludges sampled from this area is presented in Table 37 and results of analyses are given in Appendix I (Table 2).

PCB concentrations in these sludge samples lay between 0.06 and 2.31 $\mu\text{g g}^{-1}$ with a mean concentration of 0.17 $\mu\text{g g}^{-1}$. PCB were not detected in 13 of the 42 samples. The mean:median ratio (2.13) again is indicative of a skewed distribution, most influenced by the relatively elevated concentration measured in sample number 31. Concentrations of γ -HCH in the samples varied between 0.04 and 1.47 $\mu\text{g g}^{-1}$ with a mean value of 0.17 $\mu\text{g g}^{-1}$ and a median concentration of 0.12 $\mu\text{g g}^{-1}$. In this case, the distribution is not particularly skewed, with the mean influenced by the concentration of 1.47 $\mu\text{g g}^{-1}$ measured for sample 30. Aldrin was not detected in any of the 42 samples. Dieldrin was detected in 36 out of the 42 samples and concentrations lay between 0.01 and 0.72 $\mu\text{g g}^{-1}$ with a mean value of 0.16 $\mu\text{g g}^{-1}$. The mean:median ratio of 1.6 demonstrated that the distribution of results is slightly skewed by possibly, the values recorded for samples 11 and 28, of 0.72 $\mu\text{g g}^{-1}$ and 0.67 $\mu\text{g g}^{-1}$ respectively. Endrin was non-detectable in 21 of the 42 sludge samples and concentrations in the remaining 21 samples varied between 0.01 and 0.20 $\mu\text{g g}^{-1}$ with a mean value of 0.05 $\mu\text{g g}^{-1}$, which is again slightly influenced by the relatively high concentrations measured in samples 27 and 40.

Table 37. Statistical summary of results from Severn-Trent Water Authority Area.

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.17	0.35	0.06-2.31	0.08	2.13
γ -HCH	0.17	0.23	0.04-1.47	0.12	1.42
Aldrin	-	-	-	-	-
Dieldrin	0.16	0.19	0.01-0.72	0.10	1.60
Endrin	0.05	0.05	0.01-0.20	0.03	1.67

4.5.1.3. Thames Water Authority Area

A statistical summary of concentrations of organochlorines measured in sewage sludge samples from this area is presented in Table 38 and results of the analyses are given in Appendix I (Table 3).

PCB were detected in 38 of the 41 samples analysed, at concentrations between 0.02 and 0.54 $\mu\text{g g}^{-1}$. The similarity of the mean (0.16 $\mu\text{g g}^{-1}$) and median (0.15 $\mu\text{g g}^{-1}$) concentrations indicates that the data in this case approximate closely to a normal distribution. γ -HCH concentrations were non-detectable in 2 of the samples and could not be measured due to interference in a third. In the remaining 38 samples, concentrations varied between 0.01 and 1.31 $\mu\text{g g}^{-1}$ with a mean of 0.22 $\mu\text{g g}^{-1}$ and a median concentration of 0.13 $\mu\text{g g}^{-1}$. The mean:median ratio of 1.69 is indicative of a distribution which is skewed, probably by the results obtained for samples 22 and 24 in particular. Aldrin was only detected at low concentrations (0.01–0.06 $\mu\text{g g}^{-1}$) in 11 of the 41 samples, with a mean value of 0.02 $\mu\text{g g}^{-1}$. Although the mean:median ratio suggests the data are normally distributed, this is considered to be an artefact, owing to the small number of detectable results included in the calculations. Dieldrin was detected in 38 of the sludge samples and concentrations lay in the range 0.02 to 0.89 $\mu\text{g g}^{-1}$ with a mean value of 0.29 $\mu\text{g g}^{-1}$. The mean:median ratio of 1.12 indicates that the data approximate to a normal distribution, with perhaps the result for sample 7 biasing the mean. Endrin was detected in 34 of the sludges analysed, at concentrations of between 0.04 and 1.71 $\mu\text{g g}^{-1}$ with a mean concentration of 0.23 $\mu\text{g g}^{-1}$. The mean:median ratio of 1.36 suggests that a number of relatively high values, notably those of samples 18 and 22, have biased the distribution.

Table 38 Statistical summary of results from Thames Water Authority Area

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.15	0.11	0.02–0.54	0.15	1.00
γ -HCH	0.20	0.25	0.01–1.31	0.13	1.55
Aldrin	0.01	0.008	0.01–0.06	0.01	1.00
Dieldrin	0.27	0.29	0.02–0.89	0.12	1.03
Endrin	0.21	0.28	0.04–1.71	0.04	1.22

4.5.1.4 Welsh Water Authority Area

A statistical summary of analyses of sludge samples from this water authority area is presented in Table 39, and results of analyses are given in Appendix I (Table 4).

PCB were detected in 26 of the 40 samples analysed at concentrations varying between 0.01 and 1.12 $\mu\text{g g}^{-1}$ with a mean value of 0.13 $\mu\text{g g}^{-1}$. The mean:median ratio of 3.3 demonstrates that the mean is highly influenced by a small number of relatively high values, such as those of samples 18 and 26. γ -HCH was detected in all but 3 of the samples analysed at concentrations of between 0.01 and 70.0 $\mu\text{g g}^{-1}$, with a mean concentration of 1.92 $\mu\text{g g}^{-1}$. The elevated concentration detected in sample 27 (70.0 $\mu\text{g g}^{-1}$) has biased the mean value significantly, since the next highest concentration encountered was 1.08 $\mu\text{g g}^{-1}$. Aldrin was only detected in 5 of the 40 sludges analysed, at concentrations between 0.01 and 0.04 $\mu\text{g g}^{-1}$ with a mean value of 0.02 $\mu\text{g g}^{-1}$. Further statistical analysis was largely meaningless owing to the limited number of results. Dieldrin was detected in 30 of the sludge samples at concentrations varying between 0.01 and 0.48 $\mu\text{g g}^{-1}$, with a mean concentration of 0.08 $\mu\text{g g}^{-1}$. The mean:median ratio of 4.0 indicates that the distribution of the results is biased towards the high end, with a few relatively high values biasing the mean. Endrin concentrations varied between 0.01 and 0.39 $\mu\text{g g}^{-1}$ in 28 of the 40 samples analysed and were non-detectable in the remainder. The mean and median values of 0.07 $\mu\text{g g}^{-1}$ and 0.04 $\mu\text{g g}^{-1}$ respectively, suggest a distribution which is influenced by a limited number of relatively high concentrations.

Table 39. Statistical summary of results from Welsh Water Authority Area

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.14	0.22	0.01-0.73	0.03	4.67
γ -HCH	1.92	11.04	0.01-70.0	0.09	21.30
Aldrin	0.01	0.005	0.01-0.04	0.01	1.13
Dieldrin	0.08	0.11	0.01-0.48	0.02	4.00
Endrin	0.07	0.08	0.01-0.39	0.04	1.80

4.5.1.5. Northumbrian Water Authority Area

A statistical summary of analyses of sludges samples from this water authority area is presented in Table 40 and results of analyses are given in Appendix I (Table 5).

PCB concentrations in 39 of the samples varied between 0.01 and $0.37 \mu\text{g g}^{-1}$ with a mean value of $0.06 \mu\text{g g}^{-1}$ and were non-detectable on 8 occasions. The distribution of results is only slightly influenced by a limited number of elevated values, since the mean:median ratio is equal to 1.5. γ -HCH was detected in 41 of the 47 samples analysed at concentrations between 0.01 and $1.08 \mu\text{g g}^{-1}$ with a mean value of $0.12 \mu\text{g g}^{-1}$. The mean:median ratio of 4.0 indicates that the distribution of results is highly influenced by a number of relatively high values, notably those of samples 22 and 32. Aldrin was detected in 22 of the samples analysed, at concentrations varying between 0.01 and $0.13 \mu\text{g g}^{-1}$ with a mean value of $0.015 \mu\text{g g}^{-1}$. It is evident from the mean:median ratio of 1.5 that the distribution is not significantly skewed, with the concentration of $0.13 \mu\text{g g}^{-1}$ in sample 32 influencing the mean most strongly. Dieldrin was detected in all but 6 of the sludges analysed at concentrations between 0.01 and $5.54 \mu\text{g g}^{-1}$ with a mean concentration of $0.23 \mu\text{g g}^{-1}$. The one relatively high concentration measured in sample 18 ($5.54 \mu\text{g g}^{-1}$) has highly influenced the mean value, as confirmed by the mean:median ratio of 3.8. Endrin concentrations, detected in 31 of the 47 sludges analysed, varied between 0.02 and $0.84 \mu\text{g g}^{-1}$ with a mean concentration of $0.10 \mu\text{g g}^{-1}$. The distribution of results has been biased from the normal case, particularly by the relatively high results obtained for samples 20 and 45.

Table 40. Statistical summary of results from Northumbrian Water Authority Area.

Compound	Mean $\bar{x} \mu\text{g g}^{-1}$	Standard Deviation $\sigma_n \mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.06	0.07	0.01-0.37	0.04	1.5
γ -HCH	0.14	0.23	0.01-1.08	0.05	2.8
Aldrin	0.02	0.03	0.01-0.13	0.01	2.0
Dieldrin	0.23	0.80	0.01-5.54	0.06	3.8
Endrin	0.10	0.19	0.02-0.84	0.04	2.5

4.5.1.6. Yorkshire Water Authority Area

A statistical summary of the analysis of 47 sludges from this region is presented in Table 41 and results from the analyses are given in Appendix I (Table 6).

Concentrations of PCB in these sludges varied between 0.04 and 4.30 $\mu\text{g g}^{-1}$, with a mean value of 0.34 $\mu\text{g g}^{-1}$. The two relatively high values of 1.54 $\mu\text{g g}^{-1}$ and 4.30 $\mu\text{g g}^{-1}$ appear to be primarily responsible for the skewed nature of the distribution (mean:median 2.6). γ -HCH concentrations, detected in 33 of the sludges analysed, varied between 0.01 and 9.64 $\mu\text{g g}^{-1}$ with a mean value of 0.37 $\mu\text{g g}^{-1}$. The high mean:median ratio indicates that the distribution has been biased from the normal by the small number of relatively high concentrations recorded. Aldrin was detected in 32 of the samples analysed at concentrations varying between 0.01 and 0.21 $\mu\text{g g}^{-1}$ with a mean value of 0.03 $\mu\text{g g}^{-1}$. Again, the mean:median ratio of 2.0 is indicative of a skewed distribution. Dieldrin was detected in 46 of the sludge samples analysed, at concentrations between 0.03 and 52.94 $\mu\text{g g}^{-1}$ with a mean value of 1.72 $\mu\text{g g}^{-1}$. The distribution is highly skewed, with the high values encountered in samples 10-15 producing a mean:median ratio of 14.3. Endrin was detected in only 14 of the 47 sludge samples at concentrations varying from 0.02-0.23 $\mu\text{g g}^{-1}$ with a mean value of 0.02 $\mu\text{g g}^{-1}$. Although a skewed distribution of results is apparent, the limited number of samples with detectable concentrations is considered to be insufficient for reliable statistical evaluation.

Table 41. Statistical Summary of Results from Yorkshire Water Authority Area.

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.34	0.65	0.04-4.30	0.13	2.6
γ -HCH	0.37	1.41	0.01-9.64	0.04	9.25
Aldrin	0.03	0.04	0.01-0.21	0.01	3.0
Dieldrin	1.72	7.71	0.03-52.94	0.12	14.35
Endrin	0.03	0.05	0.01-0.23	0.01	3.0

4.5.1.7. North-West Water Authority Area

A statistical summary of the analysis of 44 sewage sludges sampled from this region is presented in Table 42 and results from analyses are given in Appendix I (Table 7).

PCB were detected in all but 4 of the samples, at concentrations of between 0.03 and 2.13 $\mu\text{g g}^{-1}$ with a mean value of 0.36 $\mu\text{g g}^{-1}$. The mean:median ratio of 2.8 indicates that the distribution is skewed by a small number of relatively high values, notably those of samples 2, 11, 18, 31 and 39. γ -HCH concentrations varied between 0.01 and 10.15 $\mu\text{g g}^{-1}$ in 43 samples and were non-detectable in the remaining 2. The mean concentration of 0.32 $\mu\text{g g}^{-1}$ and median value of 0.05 $\mu\text{g g}^{-1}$ indicate that the distribution of results is skewed, predominantly by the concentrations recorded for sample number 27. Aldrin was only detected in 10 of the 44 samples, at concentrations varying between 0.01 and 0.05 $\mu\text{g g}^{-1}$, with a mean value of 0.02 $\mu\text{g g}^{-1}$. Dieldrin was detected in all but 2 of the sludge samples analysed, at concentrations of between 0.01 and 1.61 $\mu\text{g g}^{-1}$ with a mean concentration of 0.19 $\mu\text{g g}^{-1}$. The mean:median ratio of 2.1 suggests that the distribution of results is slightly influenced by a small number of relatively elevated values, particularly those of samples 24 and 27. Endrin was only detected in 7 of the 44 samples, at concentrations varying between 0.02 and 0.13 $\mu\text{g g}^{-1}$ with a mean value of 0.07 $\mu\text{g g}^{-1}$.

Table 42. Statistical summary of results from North-West Water Authority Area.

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.33	0.49	0.03-2.13	0.13	2.5
γ -HCH	0.31	1.50	0.01-10.15	0.05	6.2
Aldrin	0.01	0.008	0.01-0.05	0.01	1.0
Dieldrin	0.18	0.27	0.01-1.61	0.09	2.0
Endrin	0.02	0.03	0.02-0.13	0.01	2.0

4.5.1.8. Wessex Water Authority Area

A statistical summary of analyses of sludges sampled from this region is presented in Table 43 and results of analyses are included in Appendix I (Table 8).

PCB were detected in 26 of the 36 sludge samples, at concentrations varying between 0.01 and 0.31 $\mu\text{g g}^{-1}$ with a mean concentration of 0.09 $\mu\text{g g}^{-1}$. The data approximate closely to being normally distributed, with a mean:median ratio of 1.1. γ -HCH concentrations varied between 0.03 and 2.05 $\mu\text{g g}^{-1}$ with a mean of 0.30 $\mu\text{g g}^{-1}$ in 31 of the samples analysed and were non-detectable in the remaining 5. The distribution is biased from the normal, predominantly by the concentrations recorded 34, 35 and 36. Aldrin was detected in only 9 of the 36 samples analysed, at concentrations between 0.01 and 0.02 $\mu\text{g g}^{-1}$ with a mean of 0.01 $\mu\text{g g}^{-1}$. Dieldrin concentrations in the 36 samples varied between 0.05 and 28.73 $\mu\text{g g}^{-1}$ with a mean value of 1.59 $\mu\text{g g}^{-1}$ and a median concentration 0.24 $\mu\text{g g}^{-1}$. The mean:median ratio of 6.6 indicates that the distribution is highly skewed, mainly by the relatively high concentrations encountered in samples 4, 24, 35 and 36. Endrin was detected in 15 of the 36 samples at concentrations varying between 0.01 and 0.08 $\mu\text{g g}^{-1}$ with a mean value of 0.03 $\mu\text{g g}^{-1}$.

Table 43. Statistical summary of results from Wessex Water Authority Area.

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.07	0.08	0.01-0.31	0.08	0.9
γ -HCH	0.26	0.41	0.03-2.05	0.09	2.9
Aldrin	0.01	0.003	0.01-0.02	0.01	1.0
Dieldrin	1.60	5.24	0.05-28.73	0.24	6.6
Endrin	0.02	0.02	0.01-0.08	0.01	2.0

4.5.1.9. South-West Water Authority Area

A statistical summary of the analysis of sewage sludge samples from this region is presented in Table 44 and results of analyses are given in Appendix I (Table 9).

PCB were detected in 24 of the samples at concentrations varying between 0.01 and 0.20 $\mu\text{g g}^{-1}$ with a mean value of 0.08 $\mu\text{g g}^{-1}$ and were not detected in the remaining 6 samples. The results are approximately normally distributed with a mean:median ratio of 1.1. γ -HCH was detected at concentrations between 0.01 and 2.86 $\mu\text{g g}^{-1}$ in 23 of the 30 sludges analysed, with a mean concentration of 0.24 $\mu\text{g g}^{-1}$ and a median value of 0.07 $\mu\text{g g}^{-1}$. The results are not normally distributed, with relatively high values encountered in samples 15 and 16. Aldrin was detected in only 10 of the 30 sludges analysed at concentrations varying between 0.01 and 0.04 $\mu\text{g g}^{-1}$ with a mean value of 0.02 $\mu\text{g g}^{-1}$. Dieldrin was detected in all but 1 of the 30 samples at concentrations in the range 0.02 to 2.93 $\mu\text{g g}^{-1}$ with a mean concentration of 0.39 $\mu\text{g g}^{-1}$. The distribution is moderately biased from the normal, principally by the concentrations measured in samples 1, 12 and 24. Endrin was detected in only 10 of the 30 sludges analysed and concentrations lay in the range 0.01 to 0.06 $\mu\text{g g}^{-1}$ with a mean value of 0.03 $\mu\text{g g}^{-1}$.

Table 44. Statistical summary of results from South-West Water Authority Area.

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.06	0.06	0.01-0.20	0.05	1.26
γ -HCH	0.19	0.51	0.01-2.865	0.06	3.1
Aldrin	0.01	0.007	0.01-0.04	0.01	1.0
Dieldrin	0.38	0.57	0.02-2.93	0.25	1.5
Endrin	0.02	0.01	0.01-0.06	0.01	2.0

4.5.1.10. Southern Water Authority Area

Only 17 samples were obtained from this region owing to operational difficulties. A statistical summary of sludge analyses for PCB and OCL is presented in Table 45, and results of analyses are given in Appendix I (Table 10).

PCB concentrations in the 17 samples varied between 0.05 and 0.42 $\mu\text{g g}^{-1}$ with a mean concentration of 0.13 $\mu\text{g g}^{-1}$ and a median value of 0.10 $\mu\text{g g}^{-1}$. The mean:median ratio of 1.3 indicates that the results approximate to a normal distribution, with few significantly elevated values. γ -HCH concentrations varied between 0.02 and 1.21 $\mu\text{g g}^{-1}$ with a mean value of 0.34 $\mu\text{g g}^{-1}$. The mean:median ratio of 1.7 suggests that the mean of the distribution has been biased by a few relatively high values, notably those of samples 4 and 7. Aldrin was only detected in 4 of the 17 samples analysed at low concentrations of between 0.02 and 0.06 $\mu\text{g g}^{-1}$. Dieldrin was detected in all 17 of the sludge samples analysed at concentrations varying between 0.01 and 0.67 $\mu\text{g g}^{-1}$ with a mean value of 0.14 $\mu\text{g g}^{-1}$ and a median concentration of 0.11 $\mu\text{g g}^{-1}$. The data approximate to a normal distribution, with a mean:median ratio of 1.27. Endrin was only detected in one of the 17 samples analysed, at the minimum detectable concentration of 0.01 $\mu\text{g g}^{-1}$.

Table 45. Statistical Summary of results from Southern Water Authority Area.

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.13	0.09	0.05-0.42	0.10	1.30
γ -HCH	0.34	0.33	0.02-1.21	0.20	1.70
Aldrin	0.02	0.01	0.02-0.06	0.02	2.00
Dieldrin	0.14	0.15	0.01-0.67	0.11	1.27
Endrin	-	-	-	-	-

4.5.1.11. Scottish Area Sludge Samples

A statistical summary of the analyses of sewage sludges sampled from this region is presented in Table 46 and results of analyses are given in Appendix I (Table 11).

PCB were detected in all 30 of the sewage sludges analysed, at concentrations of between 0.12 and 6.63 $\mu\text{g g}^{-1}$, with a mean value of 0.76 and a median concentration of 0.51 $\mu\text{g g}^{-1}$. The mean is biased by a small number of relatively elevated values, particularly those of samples 19, 23, 24 and 26. γ -HCH was also detected in all of the samples analysed, at concentrations varying between 0.03 and 0.65 $\mu\text{g g}^{-1}$ with a mean of 0.09 and a median concentration of 0.06 $\mu\text{g g}^{-1}$. The data are moderately skewed from the normal distribution, predominantly by the relatively high values recorded for samples 3 and 10. Aldrin was detected at low concentrations in 24 of the samples analysed, between 0.01 and 0.09 $\mu\text{g g}^{-1}$, with a mean of 0.04 $\mu\text{g g}^{-1}$ and a median concentration also of 0.04 $\mu\text{g g}^{-1}$. Dieldrin was detected in all 30 sewage sludges analysed, at concentrations varying between 0.03 and 0.14 $\mu\text{g g}^{-1}$ with a mean value of 0.06 $\mu\text{g g}^{-1}$ and a mean concentration also of 0.06 $\mu\text{g g}^{-1}$. Endrin was detected in only 19 of the 30 samples analysed, at concentrations varying between 0.01 and 0.39 $\mu\text{g g}^{-1}$ with a mean value of 0.12 $\mu\text{g g}^{-1}$ and a median concentration of 0.10 $\mu\text{g g}^{-1}$. The data approximate to a normal distribution, with a mean: median ratio of 1.20.

Table 46. Statistical summary of results from Scottish Area.

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.76	1.20	0.12-6.63	0.51	1.49
γ -HCH	0.09	0.12	0.03-0.65	0.06	1.50
Aldrin	0.03	0.02	0.01-0.09	0.03	1.00
Dieldrin	0.06	0.03	0.03-0.14	0.06	1.00
Endrin	0.08	0.09	0.01-0.39	0.06	1.33

4.5.1.12. Northern Ireland Area Samples

A statistical summary of the analyses of 30 sewage sludges sampled from this region is presented in Table 47 and results of analyses are given in Appendix I (Table 12).

PCB were detected in all 30 of the sludges analysed at concentrations varying between 0.02 and 1.07 $\mu\text{g g}^{-1}$ with a mean value of 0.22 $\mu\text{g g}^{-1}$ and a median concentration of 0.18 $\mu\text{g g}^{-1}$. The mean:median ratio of 1.22 suggested that the data are moderately close to being normally distributed, with perhaps the values recorded for samples 7 and 22 being relatively high. γ -HCH was detected in 26 of the samples analysed, at concentrations in the range 0.01 to 0.14 $\mu\text{g g}^{-1}$ with a mean value of 0.03 $\mu\text{g g}^{-1}$ and a median concentration of 0.01 $\mu\text{g g}^{-1}$. The relatively high concentration measured in sample 28 appears to have biased the mean. Aldrin was detected in only 11 of the 30 samples analysed, at concentrations of between 0.01 and 0.03 $\mu\text{g g}^{-1}$. Dieldrin concentrations in the 30 sludge samples varied between 0.05 and 0.97 $\mu\text{g g}^{-1}$ with a mean concentration of 0.17 $\mu\text{g g}^{-1}$ and a median value of 0.11 $\mu\text{g g}^{-1}$. The distribution is biased from the normal, predominantly by the concentrations recorded for samples 27, 28 and 29. Endrin was detected in only 8 of the 30 sewage sludges analysed, at concentrations varying between 0.01 and 0.16 $\mu\text{g g}^{-1}$.

Table 47. Statistical summary of results from Northern Ireland.

Compound	Mean $\bar{x}$ $\mu\text{g g}^{-1}$	Standard Deviation σ_n $\mu\text{g g}^{-1}$	Range $\mu\text{g g}^{-1}$	Median $\mu\text{g g}^{-1}$	Mean:Median ratio
PCB	0.22	0.22	0.02-1.07	0.18	1.22
γ -HCH	0.02	0.03	0.01-0.14	0.01	2.00
Aldrin	0.01	0.005	0.01-0.03	0.01	1.00
Dieldrin	0.17	0.22	0.05-0.97	0.11	1.50
Endrin	0.02	0.04	0.01-0.16	0.01	2.00

4.5.2. Variations in time of the concentrations of persistent organochlorine compounds in sewage sludges from Beckton, Haverhill and Loughborough Sewage Treatment Works

To evaluate the variation in PCB and OCL concentrations with time, three sewage treatment works were sampled on a regular basis. Concentrations of PCB, γ -HCH, aldrin, dieldrin and endrin measured in 18 discrete monthly samples from each of these sewage treatments works are included in Tables 48, 49 and 50. PCB concentrations in concentrations in Beckton sludges varied between $0.11 \mu\text{g g}^{-1}$ and $0.93 \mu\text{g g}^{-1}$ over the sampling period with a mean concentration of $0.26 \mu\text{g g}^{-1}$. Concentrations of γ -HCH varied between 0.01 and $0.21 \mu\text{g g}^{-1}$ over the sampling period with a mean value of $0.10 \mu\text{g g}^{-1}$. Aldrin was only detected in 6 samples at concentrations of 0.01 to $0.04 \mu\text{g g}^{-1}$. Dieldrin concentrations varied between 0.21 and $0.91 \mu\text{g g}^{-1}$ with a mean of $0.60 \mu\text{g g}^{-1}$. Endrin was only detected in one sample (October 1981) at a concentration of $0.02 \mu\text{g g}^{-1}$.

Table 48. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Beckton Sewage Treatment Works

Sample Date	Total Solids Concentration $\mu\text{g l}^{-1}$	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
14.4.81	45.72	0.13	0.15	ND ¹	0.89	ND
20.5.81	21.77	0.37	0.10	ND	0.86	ND
16.6.81	23.09	0.19	0.13	ND	0.71	ND
13.7.81	19.30	0.28	0.08	ND	0.75	ND
17.8.81	23.48	0.25	0.21	ND	0.56	ND
14.9.81	28.72	0.11	0.19	ND	0.70	ND
12.10.81	27.04	0.15	0.09	ND	0.91	0.02
16.11.81	26.93	0.31	0.17	ND	0.77	ND
18.12.81	71.32	0.17	0.12	0.01	0.27	ND
17.1.82	30.76	0.11	0.01	ND	0.29	ND
15.2.82	18.11	0.29	0.03	0.01	0.68	ND
15.3.82	26.73	0.20	0.03	ND	0.67	ND
15.4.82	23.09	0.14	0.04	0.01	0.76	ND
17.5.82	23.79	0.23	0.12	0.02	0.81	ND
14.6.82	24.90	0.22	0.20	ND	0.33	ND
20.7.82	24.96	0.32	0.81	0.01	0.32	ND
12.8.82	47.41	0.28	0.14	ND	0.39	ND
14.9.82	13.18	0.93	0.03	0.04	0.21	ND

Table 49. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Haverhill Sewage Treatment Works

Sample Date	Total Solids Concentration $\mu\text{g l}^{-1}$	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
17.4.81	291.72	0.09	0.27	ND	0.05	0.04
15.5.81	312.14	0.07	0.30	ND	0.03	0.03
20.6.81	279.77	ND ¹	0.25	ND	0.04	ND
18.7.81	304.90	ND	0.61	ND	0.03	0.08
15.8.81	279.48	0.09	0.43	ND	0.12	ND
19.9.81	231.98	0.12	0.18	ND	0.31	ND
16.10.81	274.17	0.07	0.53	ND	0.20	ND
14.11.81	301.58	0.03	0.39	ND	0.11	0.03
18.12.81	307.62	0.08	0.08	ND	0.03	0.16
13.1.82	261.30	ND	0.02	ND	0.02	0.19
16.2.82	259.94	ND	0.04	ND	0.02	0.11
15.3.82	317.61	ND	0.04	ND	0.01	0.12
15.4.82	281.50	0.07	0.07	0.01	0.02	0.09
17.5.82	296.24	0.06	0.05	ND	0.03	0.15
12.6.82	200.80	0.29	0.02	0.02	0.04	ND
19.7.82	344.40	0.45	0.05	0.02	0.05	0.01
17.8.82	300.30	0.13	0.11	0.01	0.06	ND
14.9.82	316.20	0.07	0.09	0.01	0.19	ND

Table 50. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Loughborough Sewage Treatment Works

Sample Date	Total Solids Concentration $\mu\text{g l}^{-1}$	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
15.4.81	45.24	0.19	0.01	ND	0.16	ND
11.5.81	34.67	0.51	0.03	ND	0.09	ND
15.6.81	37.56	0.17	0.03	ND	0.14	0.04
17.7.81	34.52	0.29	0.07	ND	0.30	0.02
12.8.81	38.76	0.22	0.21	ND	0.16	0.06
14.9.81	28.89	0.23	0.09	ND	0.07	ND
12.10.81	10.01	0.34	0.03	ND	0.11	0.01
16.11.81	32.58	0.13	0.08	ND	0.18	ND
18.12.81	21.25	0.10	0.02	ND	0.07	0.41
19.1.82	25.64	0.19	ND	0.02	0.14	ND
15.2.82	22.78	0.04	ND	ND	0.02	ND
16.3.82	38.05	0.20	0.03	ND	0.11	0.06
15.4.82	46.37	0.21	0.03	0.03	0.16	1.17
17.5.82	21.42	ND	0.23	ND	ND	ND
14.6.82	15.96	0.19	0.02	0.03	0.15	ND
19.7.82	47.31	0.18	0.01	0.04	0.10	ND
16.8.82	26.45	0.29	0.04	ND	0.21	ND
13.9.82	25.54	0.31	0.07	0.01	0.16	ND

1 Not detected ($<0.01 \mu\text{g g}^{-1}$)

Concentrations of PCB in the sludges sampled from Haverhill sewage treatment works varied between 0.03 and 0.45 $\mu\text{g g}^{-1}$ with a mean value of 0.12 $\mu\text{g g}^{-1}$ and were non-detectable on five occasions. γ -HCH concentrations lay in the range 0.02 to 0.61 $\mu\text{g g}^{-1}$ with a mean of 0.20 $\mu\text{g g}^{-1}$. Aldrin was only detected on five occasions, at concentrations of 0.01 to 0.02 $\mu\text{g g}^{-1}$. Dieldrin concentrations varied between 0.01 and 0.31 $\mu\text{g g}^{-1}$ with a mean value of 0.08 $\mu\text{g g}^{-1}$ over the sampling period. Endrin was not detected on seven occasions and concentrations varied between 0.01 and 0.19 $\mu\text{g g}^{-1}$, with a mean value of 0.09 $\mu\text{g g}^{-1}$, over the remaining 11 samples.

In sludges from Loughborough sewage treatment works, PCB concentrations varied between 0.04 and 0.51 $\mu\text{g g}^{-1}$ with a mean value of 0.22 $\mu\text{g g}^{-1}$ and one non-detectable result in May 1982. γ -HCH concentrations were non-detectable on two occasions and, in the remaining 16 samples, varied between 0.01 and 0.23 $\mu\text{g g}^{-1}$ with a mean concentration of 0.06 $\mu\text{g g}^{-1}$. Aldrin concentrations were non-detectable on 13 occasions and varied between 0.01 and 0.04 $\mu\text{g g}^{-1}$ in the remaining 5 samples. Concentrations of dieldrin lay in the range 0.02 to 0.30 $\mu\text{g g}^{-1}$ with a mean of 0.14 $\mu\text{g g}^{-1}$ and one non-detectable result. Endrin was only detected on seven occasions and concentrations varied between 0.01 and 1.17 $\mu\text{g g}^{-1}$ with a mean value of 0.25 $\mu\text{g g}^{-1}$.

Examination of the results presented as time series in Figures 8, 9 and 10 reveals possible trends in the data for some determinands over the eighteen month sampling period. In the Beckton sewage sludge samples distinct peaks in concentration are evident for γ -HCH over the sampling period, around August to November 1981 and over May to June 1982. A broadly similar trend is also apparent for dieldrin, where peaks in concentration occur around September to November 1981 and March to May 1982. No apparent trend exists for concentrations of PCB. In the Haverhill sewage sludges, γ -HCH and dieldrin concentrations again exhibit similar trends, with elevated concentrations in the period July to December 1981. PCB concentrations displayed a small increase over this period and a well-defined peak over the period May to September 1982, when no similar increases were observed for γ -HCH and dieldrin.

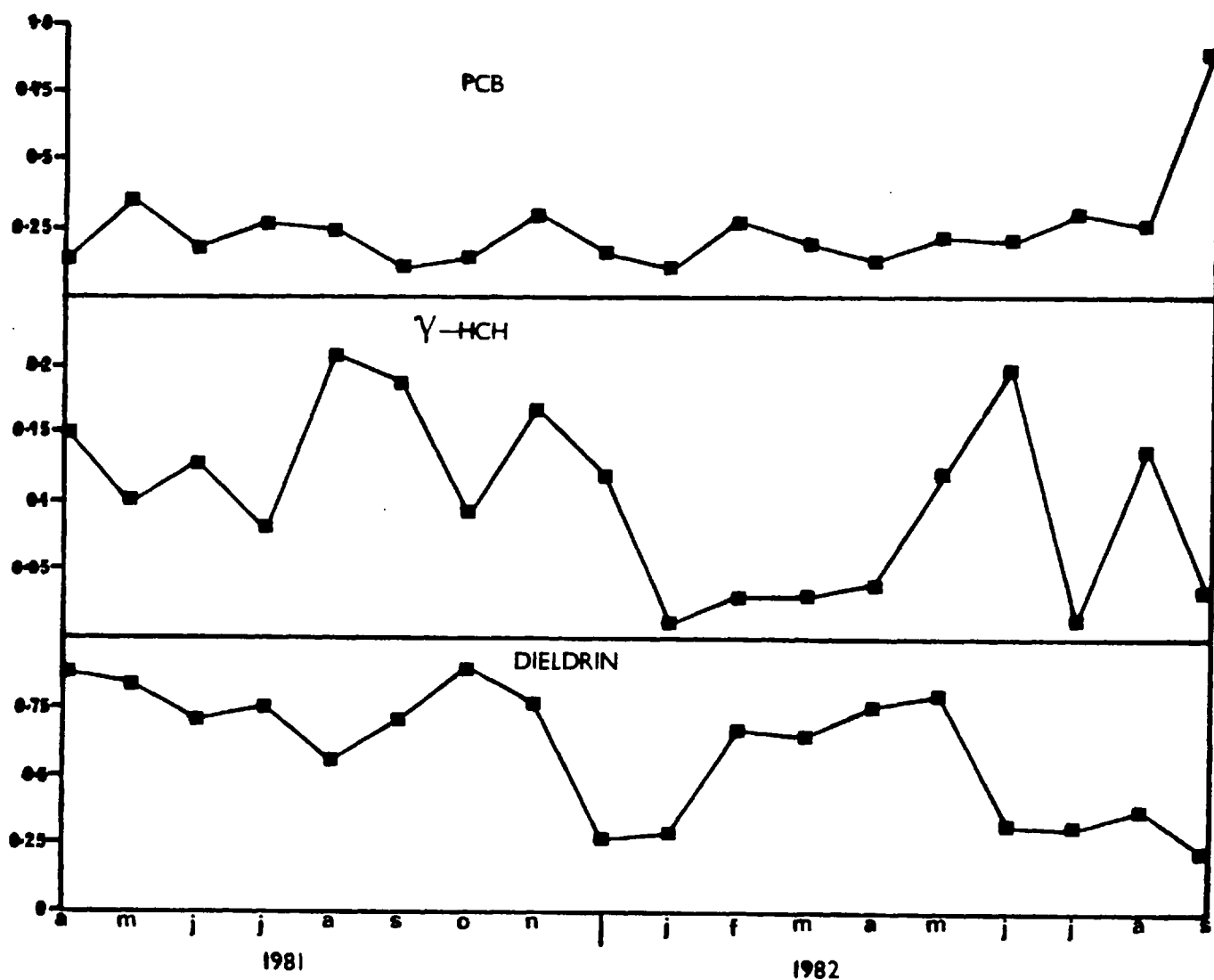
concentration $\mu\text{g g}^{-1}$ 

Figure 8. Variation with time of concentrations of persistent organochlorine compounds in sewage sludge samples from Beckton Sewage Treatment Works

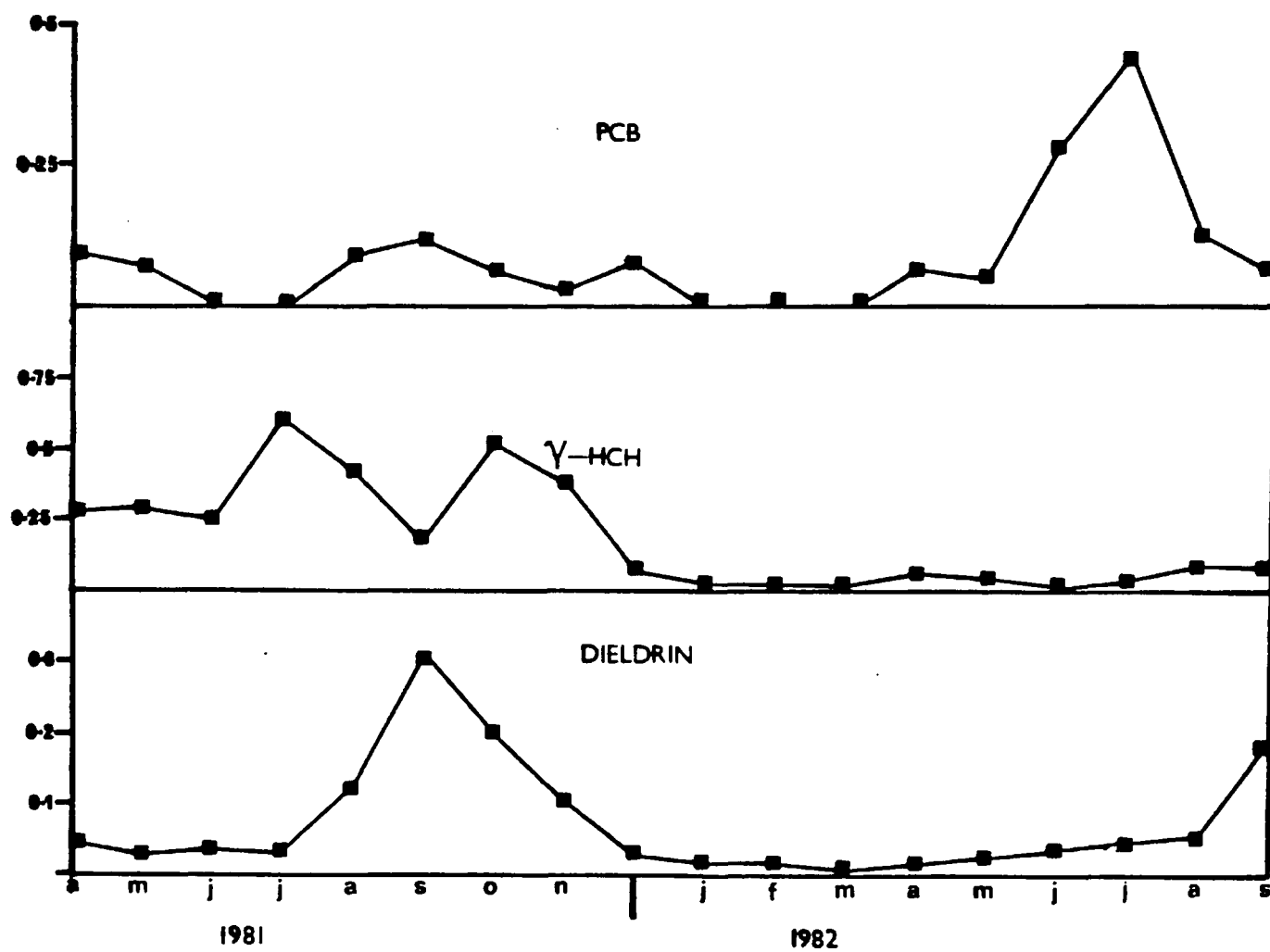
concentration $\mu\text{g g}^{-1}$ 

Figure 9. Variation with time of concentrations of persistent organochlorine compounds in sewage sludge samples from Haverhill Sewage Treatment Works

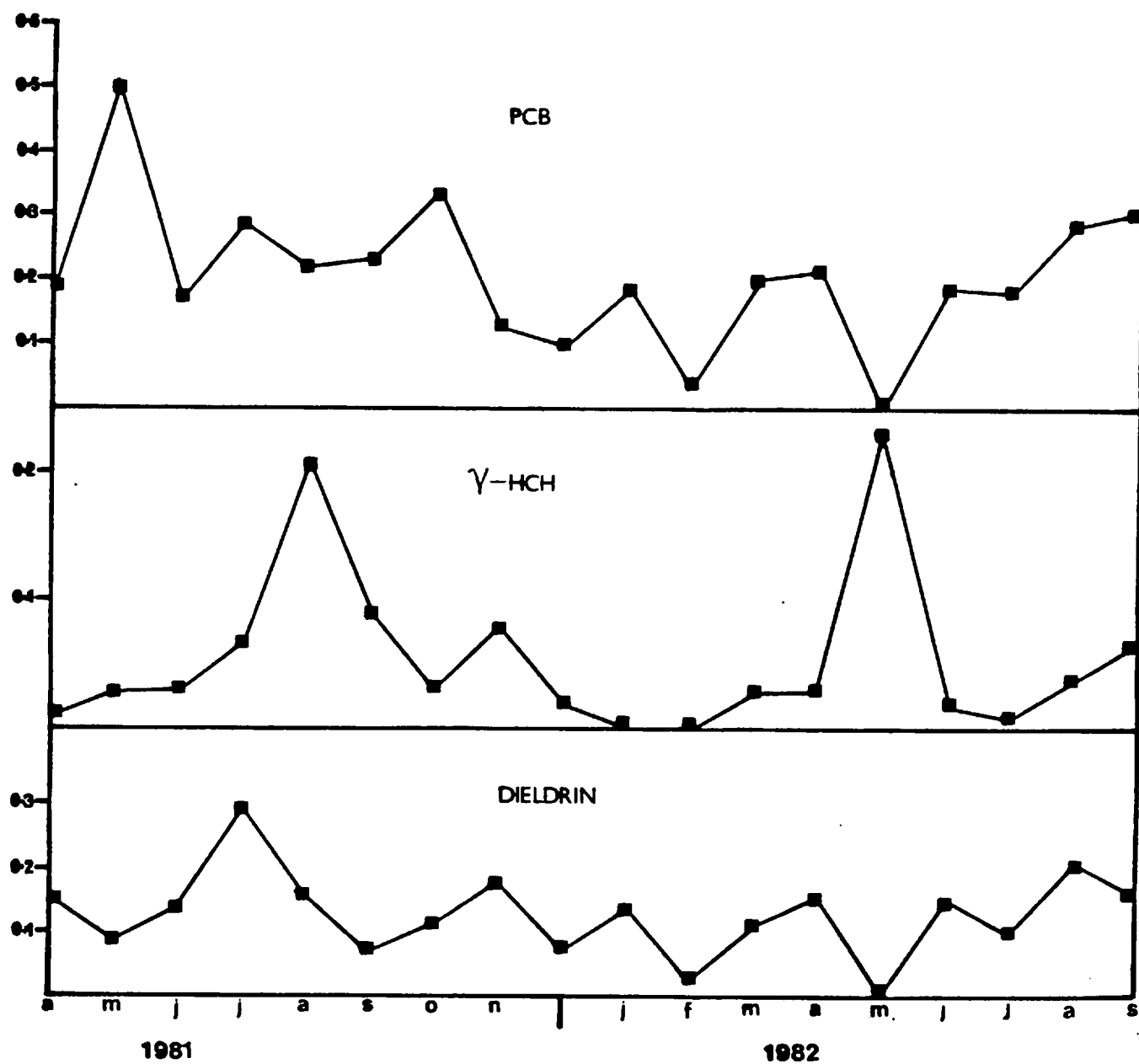
concentration $\mu\text{g g}^{-1}$ 

Figure 10. Variation with time of concentrations of persistent organochlorine compounds in sewage sludge samples from Loughborough Sewage Treatment Works

γ -HCH concentrations in the sewage sludges sampled from Loughborough exhibited two distinct peaks over June to October 1981 and April to July 1982. A minor peak in dieldrin concentrations was also observed over June to September 1981. Elevated PCB concentrations also occurred in May 1981 and October 1981, during the same overall period of elevated γ -HCH and dieldrin concentrations.

In general, no significant trend for any determinand at any of the works studied is evident.

4.5.3. Statistical analysis of PCB and OCL concentrations in UK sewage sludges

4.5.3.1. Distribution of PCB and OCL concentrations in UK sewage sludges

A statistical summary of the results from the analysis of sewage sludge samples from 444 UK treatment plants is included in Table 51.

Table 51. Statistical summary of concentrations of persistent organochlorine compounds in UK sewage sludges

Compound	Mean conc. $\mu\text{g g}^{-1}$	Standard Deviation $\mu\text{g g}^{-1}$	Percentiles, $\mu\text{g g}^{-1}$			Range $\mu\text{g g}^{-1}$
			5	50 (median)	95	
PCB	0.34	1.23	0.03	0.14	0.95	0.01-21.5
γ -HCH	0.41	3.58	0.01	0.09	0.67	0.01-70.00
Aldrin	0.03	0.03	0.01	0.02	0.09	0.01-00.21
Dieldrin	0.50	3.07	0.02	0.13	0.97	0.01-52.94
Endrin	0.11	0.16	0.01	0.06	0.30	0.01-00.71

PCB concentrations exhibited a mean value of $0.34 \mu\text{g g}^{-1}$, a standard derivation of $1.23 \mu\text{g g}^{-1}$ and a 50th percentile value (median) of $0.14 \mu\text{g g}^{-1}$. The data are not normally distributed, as confirmed by both the mean:median ratio (2.4) and by the observation that the majority of the distribution does not lie between $\pm 3 \sigma$ of the mean value. γ -HCH concentrations varied between 0.01 and $70.0 \mu\text{g}$

g^{-1} with a mean value of $0.41 \mu\text{g g}^{-1}$, a standard derivation (σ_n) of $3.58 \mu\text{g g}^{-1}$ and a 50th percentile value (median) of $0.09 \mu\text{g g}^{-1}$. The mean:median ratio of 4.6 indicates that the data are abnormally distributed. Aldrin concentrations in the 444 sludge samples were generally low, with a mean of $0.03 \mu\text{g g}^{-1}$, a standard deviation of $0.03 \mu\text{g g}^{-1}$ and a 50th percentile value (median) of $0.02 \mu\text{g g}^{-1}$. The limited range in the concentrations measured (0.01 - $0.21 \mu\text{g g}^{-1}$) and the relatively large number of non-detectable values preclude a comprehensive appreciation of the statistical distribution of the results. Concentrations of dieldrin varied between 0.01 and $52.94 \mu\text{g g}^{-1}$ with a mean value of $0.50 \mu\text{g g}^{-1}$, a standard deviation of $3.07 \mu\text{g g}^{-1}$ and a 50th percentile (median) of $0.13 \mu\text{g g}^{-1}$. The mean:median ratio (3.8) indicates that the data do not approximate to a normal distribution. Endrin concentrations measured in the sludge samples lay in the more limited range 0.01 to $1.71 \mu\text{g g}^{-1}$, with a mean value of $0.11 \mu\text{g g}^{-1}$, a standard deviation of $0.16 \mu\text{g g}^{-1}$ and a 50th percentile concentration (median) of $0.06 \mu\text{g g}^{-1}$. The data are not normally distributed, as is confirmed by the mean:median ratio of 1.8.

In an attempt to define more closely the form of the results, data from analyses for the five determinands were plotted on log-normal probability graphs which are illustrated in figures 11 to 15. It is evident that the data approximate tolerably well to straight lines on these plots and it therefore appears that the results of this survey are best described by the log-normal distribution form.

4.5.3.2. Analysis of variance between sewage sludge types

In order to evaluate any possible differences in the concentrations of persistent organochlorine substances between the various types of sludges sampled (whether primary, mixed primary/secondary biological sludges or digested sludges), the data acquired from analysis of the 444 sludge samples were divided on the basis of the three aforementioned sludge types and analysis of variance was conducted for each determinand. No significant differences were observed between the means, at the 95% significance level, of any of the five determinands for the three sludge types, with the exception of dieldrin concentrations in digested sludges. This result was found subsequently to be caused by three particularly elevated concentrations recorded for digested sludges obtained from treatment

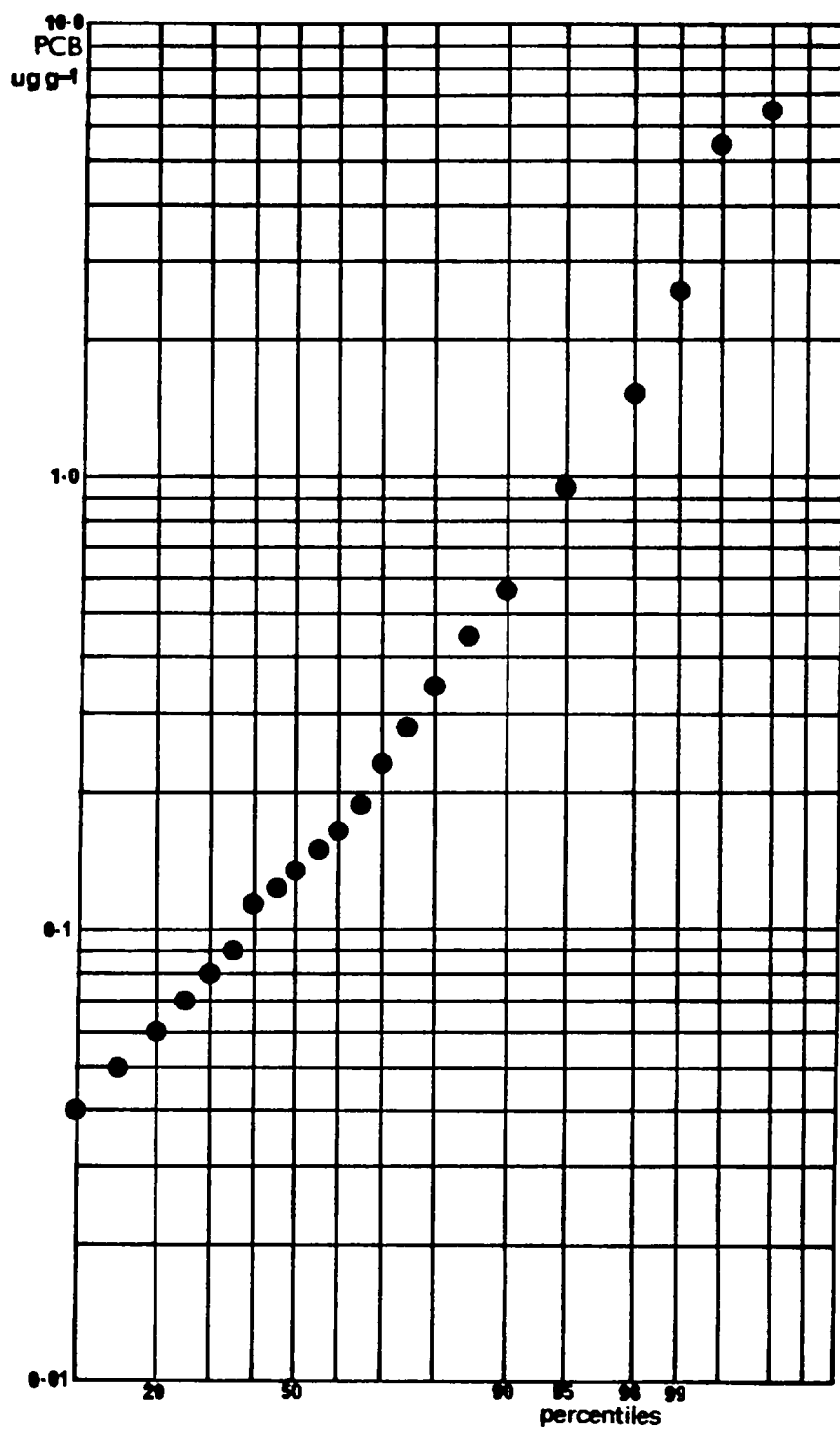


Figure 11. Log-normal probability plot of PCB concentrations in U.K. sewage sludges.

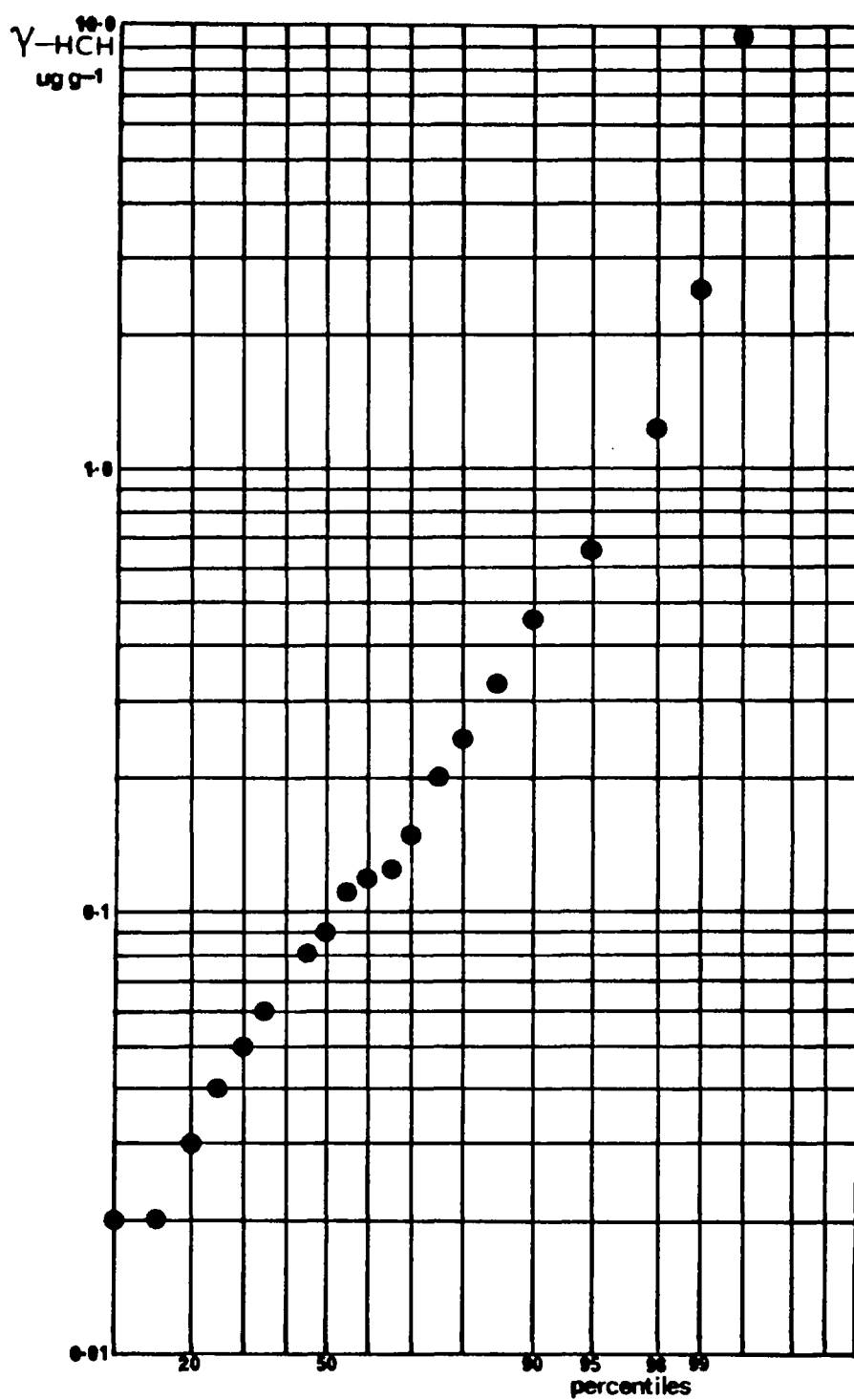


Figure 12. Log-normal probability plot of γ -HCH concentrations in U.K. sewage sludges.

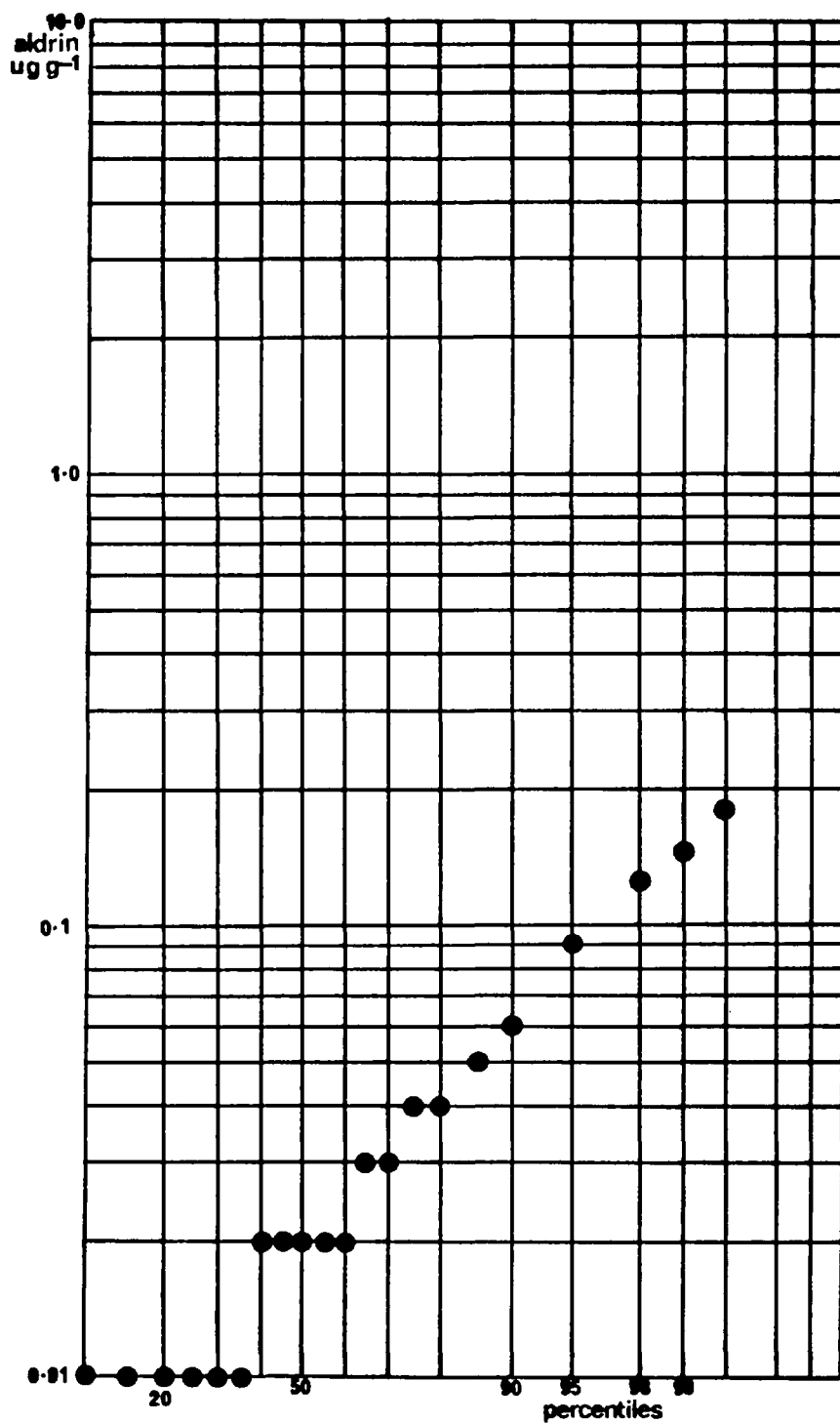


Figure 13. Log-normal probability plot of aldrin concentrations in U.K. sewage sludges.

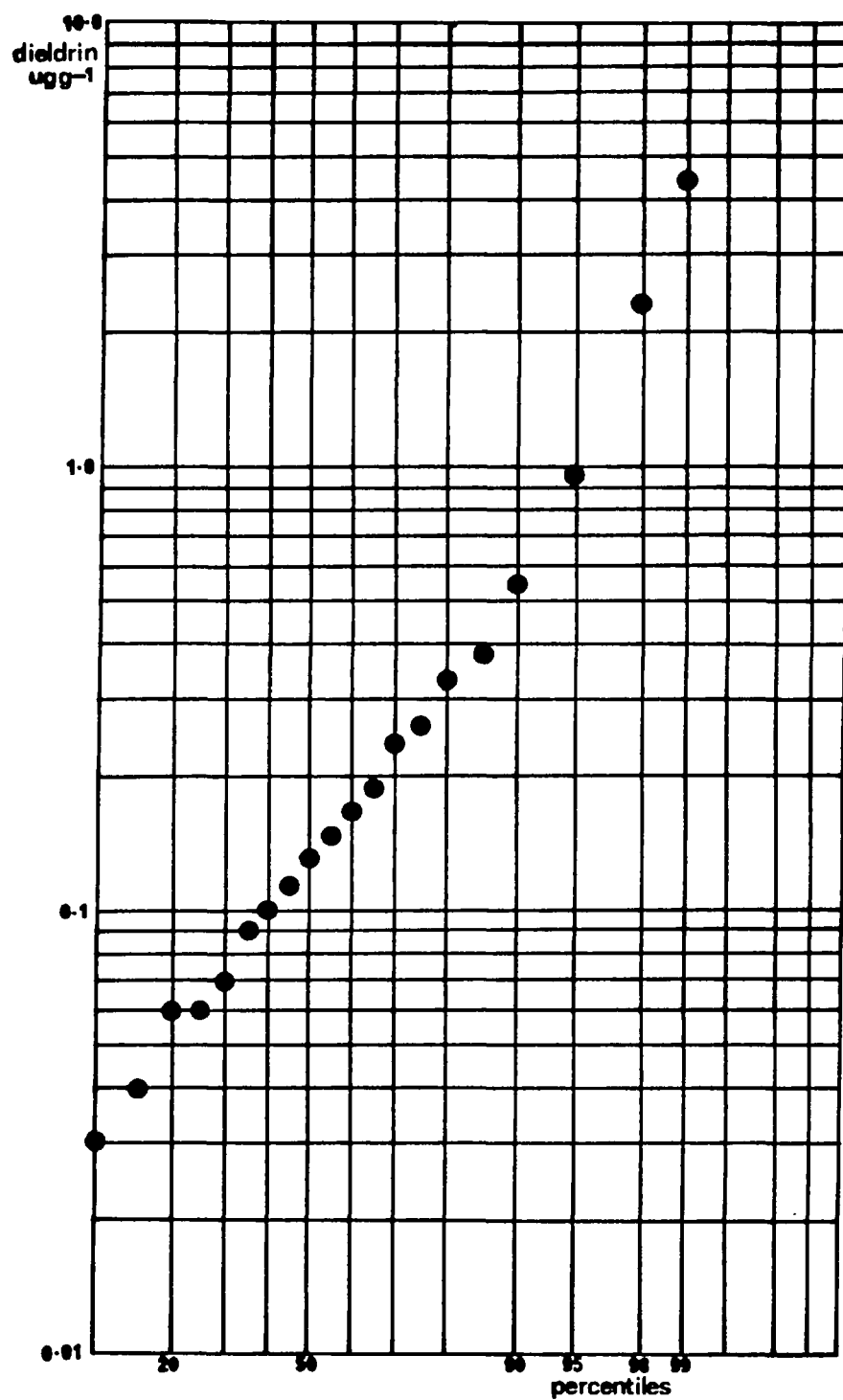


Figure 14. Log-normal probability plot of dieldrin concentrations in U.K. sewage sludges.

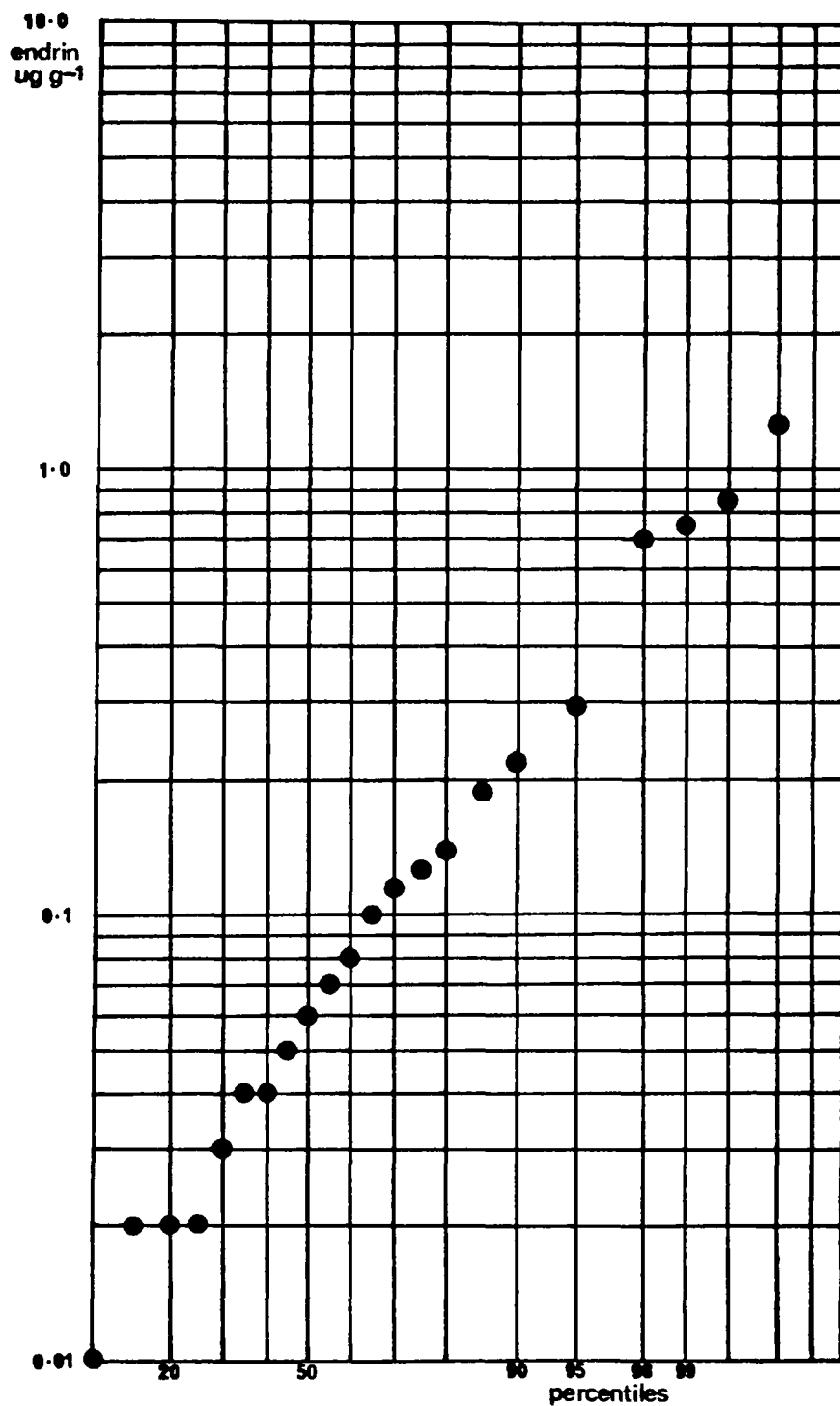


Figure 15. Log-normal probability plot of endrin concentrations in U.K. sewage sludges.

works known to receive trade effluents containing dieldrin. Since the difference between the means could not therefore be attributed to sludge treatment factors, the test was repeated with the exclusion from the calculations of the three elevated values and a negative result was obtained.

4.5.3.3. Correlation Analysis of PCB and OCL Concentrations in UK Sewage Sludges

Simple linear correlation analyses between determinand concentration, relative proportions of trade effluent received and the population served by the respective 444 sewage treatment works sampled were carried out for each determinand. Little or no correlation was observed between any of the compounds considered and either of the two sewage parameters; typical examples of correlations obtained for PCB, γ -HCH, aldrin, dieldrin and endrin with trade effluent received and population served are included in Table 52. The correlation coefficients (r) vary between -0.021 and $+0.180$, indicating that little or no correlations exist between the variables of determinand concentration, industrial effluent received and population served.

Table 52. Correlation analysis of determinands with industrial effluent received and population served.

Compound	Correlation Coefficients (r)	
	Industrial Effluent	Population
PCB	-0.010	-0.013
γ -HCH	-0.013	-0.018
Aldrin	-0.021	$+0.002$
Dieldrin	$+0.029$	-0.017
Endrin	$+0.180$	$+0.176$

4.5.3.4. Serial correlation analysis of Beckton, Haverhill and Loughborough samples

In order to elucidate any underlying trends in the monthly data from analysis of sewage sludges taken from these three locations, a serial correlation (autocorrelation) analysis was conducted, according to the method described by Chatfield (1975). The autocorrelation plots (correlograms) for these data are presented in figures 16, 17 and 18 where the autocorrelation coefficients (r_k) are plotted against corresponding time lags (k) in the raw data. Five different time lags are included in the graphs, since the maximum number recommended should be $< N/4$ (where N = number of raw data points). It is possible, from the shape and form of the correlograms, to assess the nature of the variations inherent in the raw data. However, unless the correlograms conform closely to a small number of pre-established forms, interpretation of the underlying trends in the time series data is difficult.

For the samples obtained from the three sewage treatment works over the 18-month period, correlograms have been derived only for PCB, γ -HCH and dieldrin, since low and non-detectable concentrations of aldrin and endrin were observed frequently.

PCB concentrations in Beckton sewage sludges exhibited a random time series distribution, with autocorrelation coefficients (r_k) of 0 at all time lags (k). A similar pattern was also obtained for γ -HCH concentrations, where little or no correlation between successive values at different time lags is evident. Dieldrin concentrations displayed a somewhat different correlogram, the form of which suggests a degree of short-term correlation, i.e. one for which an observation above the mean tends to be followed by one or more further observations above the mean, and similarly for observations below the mean. This may also be termed as an autoregressive time series. For the Haverhill samples, both the PCB and dieldrin concentrations exhibit a degree of short term correlation, as do those of γ -HCH, although to a lesser degree. Concentrations of PCB, γ -HCH and dieldrin in the Loughborough samples approximate closely to a random series, with the autocorrelation coefficients of all these determinands at different time lags tending to zero.

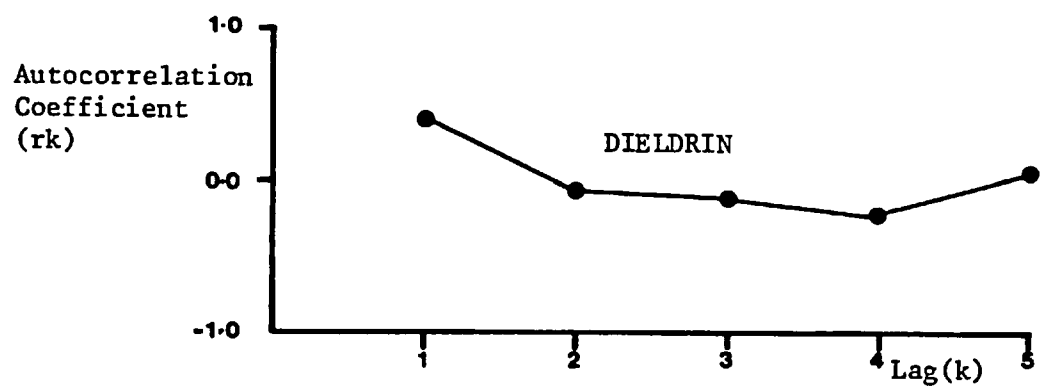
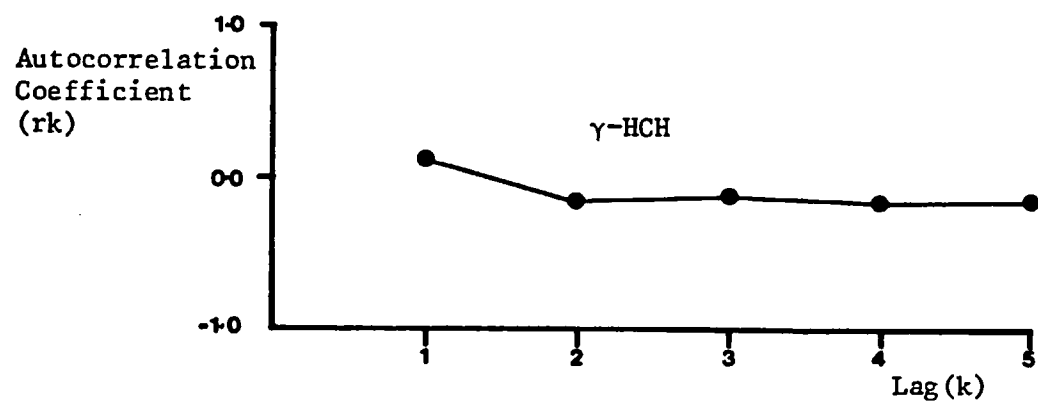
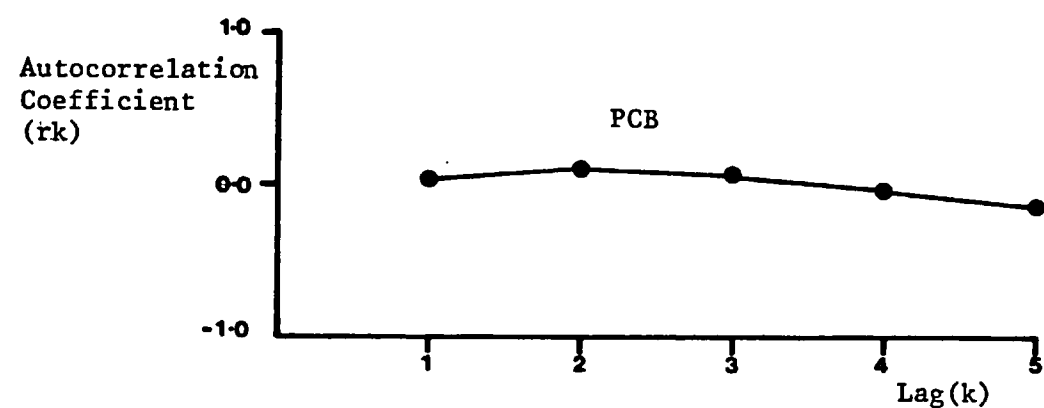


Figure 16. Autocorrelation plots for monthly data from Beckton sewage sludge samples

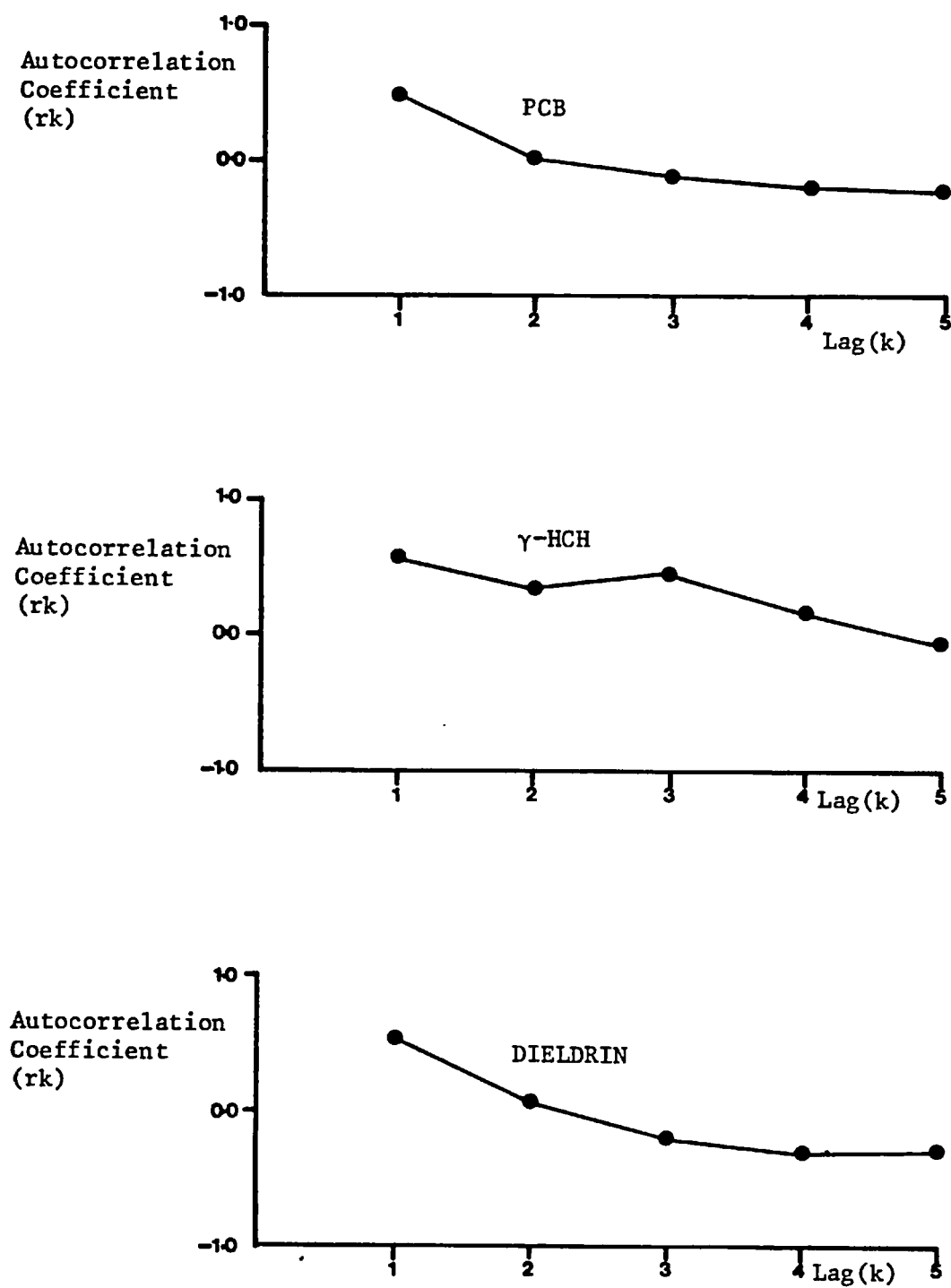


Figure 17: Autocorrelation plots for monthly data from Haverhill sewage sludge samples

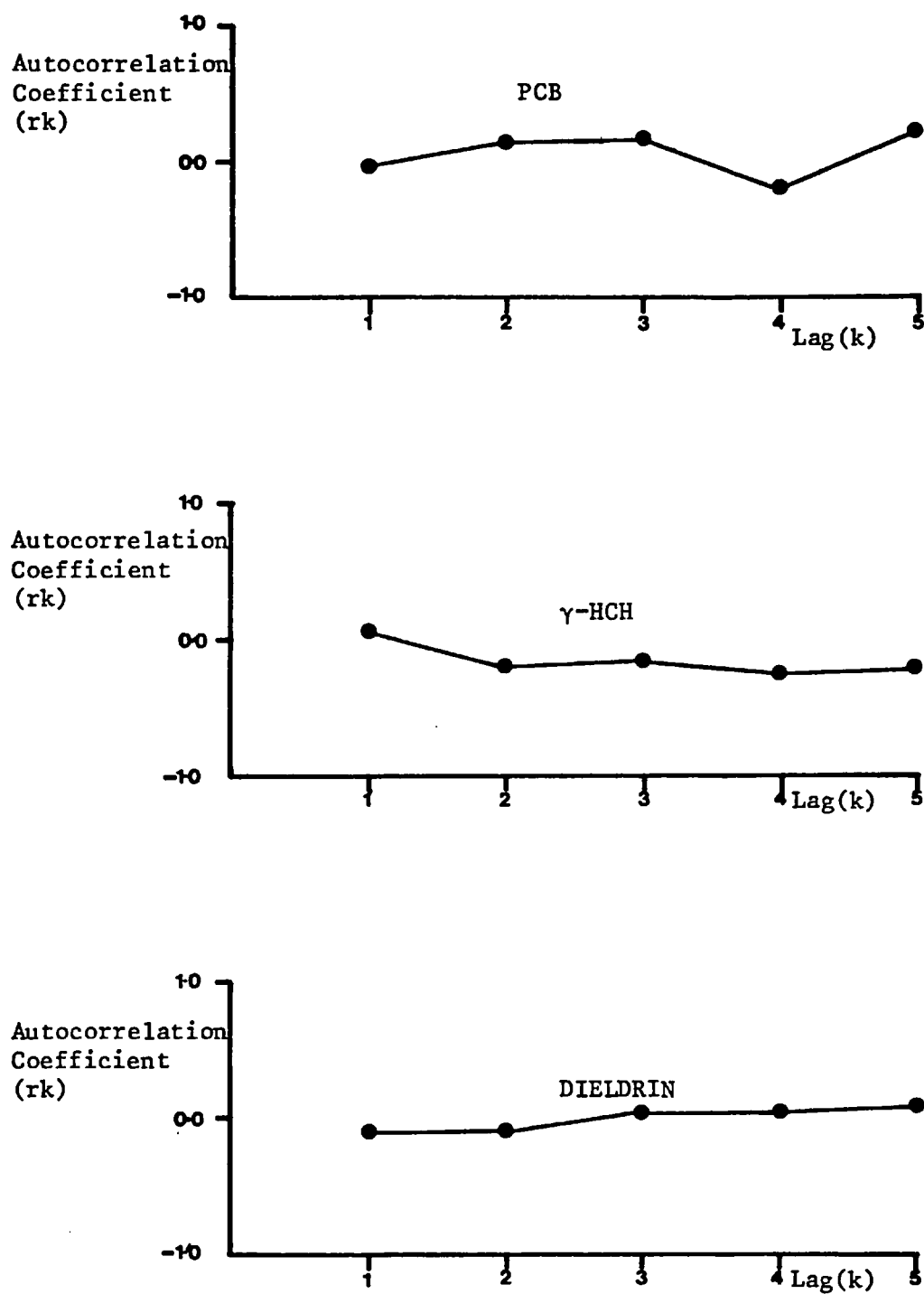


Figure 18. Autocorrelation plots for monthly data from Loughborough sewage sludge samples

Although some general indications are present as to the trends in the time series data for the sewage sludge samples from the three works, insufficient raw data points are available to achieve a comprehensive analysis. Since only 5 time lags may be properly extracted from the available data, the power of the statistical analysis is substantially reduced.

4.6. Survey of the concentrations of PCB, γ -HCH, aldrin, dieldrin and endrin in a subsample of UK sewage sludges using packed and capillary column gas chromatography and capillary column gas chromatography-mass spectrometry

Although elementary packed column gas chromatographic (PGC) techniques have been employed to good effect for the determination of PCB and OCL in a large number of UK sewage sludges, more advanced analytical techniques, including capillary column gas chromatography (CGC) and capillary column gas chromatography-mass spectrometry (GC-MS) may be applied in order to resolve problems which may arise in the detection of PBC and OCL in particularly complex sample matrices. These latter two techniques, however, are not readily applicable to the routine analysis of large numbers of samples owing to constraints of cost and time involved in each analysis. In order to assess the suitability of these advanced techniques for the determination of PCB and OCL compounds in sewage sludge extracts, a subsample of sludges which had displayed complex PGC chromatograms and, in some cases, elevated concentrations of PCB and certain OCL, were selected for analysis by PGC, CGC and GC-MS techniques. Therefore 20 samples were selected on the basis of their complexity and elevated PCB and aldrin concentrations and a further 20 complex samples were taken with above-average concentrations of γ -HCH, dieldrin and endrin. All sample extracts analysed by the three techniques were identical in form, except where dilution of extracts was required in order to maintain determinand concentrations within the calibrated working range of the analytical instruments. The instrumental and experimental conditions have been described previously (section 3.3.5.).

The results of the analyses for PCB are presented in Table 53. Concentrations determined by the three analytical techniques displayed broad similarities, and varied between 0.04 and 8.04 $\mu\text{g g}^{-1}$

Table 53. PCB concentrations in selected sewage sludges determined by packed and capillary column gas chromatography and by capillary column gas chromatography-mass spectrometry

Sample Number	Packed Column GC $\mu\text{g l}^{-1}$	Capillary Column GC $\mu\text{g l}^{-1}$	Capillary Column GC-MS $\mu\text{g l}^{-1}$
1	0.23	0.17	0.26
2	5.00	3.49	3.27
3	0.20	0.36	0.41
4	0.16	0.04	0.05
5	1.74	0.78	0.65
6	5.45	5.10	5.04
7	1.58	1.95	2.27
8	1.08	1.16	1.02
9	6.15	8.05	5.63
10	0.52	0.59	0.64
11	0.49	0.50	0.47
12	8.04	10.85	11.01
13	0.80	0.94	0.62
14	2.20	3.07	2.41
15	1.72	1.87	1.31
16	0.08	0.10	ND*
17	0.82	0.94	0.76
18	0.04	0.05	ND
19	0.87	0.76	0.71
20	0.30	0.37	0.26

* not detected.

for PGC analyses, between 0.04 and 10.85 $\mu\text{g g}^{-1}$ for CGC analyses and between 0.05 and 11.01 $\mu\text{g g}^{-1}$ for GC-MS analyses. In the greater majority of the samples, lower concentrations were recorded using GC-MS than with the other two analytical systems. However, CGC analyses, in overall terms, compared favourably with analyses conducted by GC-MS. PCB were not detected by GC-MS in samples 16 and 18, although concentrations of 0.08 and 0.10 $\mu\text{g g}^{-1}$ were recorded by PGC and CGC respectively. At these concentrations, a combination of background interference from ions of similar masses to PCB isomers eluting into the mass spectrometer and baseline noise effectively obscured the

detection signal due to PCB. The generally higher concentrations measured by PGC are almost certainly due to the inferior resolution of packed columns and the quantification of co-eluting substances together with PCB peaks on the gas chromatograms. In general, the results obtained from the three analytical techniques compared tolerably well.

Results for the analysis of γ -HCH in 20 samples using the three analytical systems are included in Table 54. Concentrations

Table 54. γ -HCH concentrations in selected sewage sludges determined by packed and capillary column gas chromatography and by capillary column gas chromatography-mass spectrometry

Sample Number	Packed Column GC $\mu\text{g l}^{-1}$	Capillary Column GC $\mu\text{g l}^{-1}$	Capillary Column GC-MS $\mu\text{g l}^{-1}$
1	1.84	1.25	0.97
2	1.40	1.01	1.14
3	1.36	0.91	0.86
4	2.51	1.88	1.89
5	3.20	2.29	2.42
6	0.19	ND*	ND
7	1.37	1.25	1.07
8	0.34	ND	ND
9	0.39	ND	ND
10	1.99	1.61	1.53
11	0.27	ND	ND
12	0.13	ND	ND
13	0.09	0.07	0.10
14	0.17	0.13	0.19
15	0.26	0.08	0.08
16	0.39	0.25	0.20
17	0.49	0.30	0.33
18	0.14	0.12	0.09
19	0.29	0.28	0.22
20	0.20	0.16	0.17

* not detected.

detected varied between 0.09 and 3.20 $\mu\text{g g}^{-1}$ for PGC analyses, between 0.07 and 2.29 $\mu\text{g g}^{-1}$ for CGC analyses, with five non-detectable results, and between 0.08 and 2.42 $\mu\text{g g}^{-1}$ for GC-MS analyses, also with five non-detectable results. Concentrations measured by PGC were, on average significantly higher than those recorded using either CGC or GC-MS, and in some instances, γ -HCH was found to be absent from the extracts when analysed by CGC and GC-MS; viz samples 6, 8, 9, 11 and 12. As with PCB analyses, CGC and GC-MS analyses compared favourably within the limits of analytical error. The possible sources of error in the quantification of γ -HCH by PGC may arise from the fact that this compound elutes from the packed column in the 'early' region of the chromatogram and was frequently observed to be only partially resolved from surrounding gas chromatographic peaks. It would appear, therefore, that co-eluting peaks may have resulted in over-calculation of γ -HCH concentrations.

Aldrin was only detected in a limited number of the samples taken for analysis of PCB and this compound and the results are presented in Table 55. Concentrations varied between 0.01 and 0.63 for PGC analyses, between 0.01 and 0.14 $\mu\text{g g}^{-1}$ for CGC analyses and between 0.05 and 0.08 $\mu\text{g g}^{-1}$ for GC-MS analyses. Aldrin was measured in

Table 55. Aldrin concentrations in selected sewage sludges determined by packed and capillary column gas chromatography and by capillary column gas chromatography-mass spectrometry

Sample Number	Packed Column GC $\mu\text{g l}^{-1}$	Capillary Column GC $\mu\text{g l}^{-1}$	Capillary Column GC-MS $\mu\text{g l}^{-1}$
1	0.10	ND*	ND
3	0.05	0.01	ND
5	0.01	ND	ND
6	0.25	0.02	ND
11	0.07	ND	ND
12	0.06	0.04	0.05
14	0.06	ND	ND
15	0.63	0.14	0.08

* not detected.

8 samples by PGC, but these results were not reflected in the CGC and C-MS analyses, where aldrin was not detected in samples 1, 5, 11 and 14 by CGC and was only detected in samples 12 and 15 by GC-MS. Problems in the quantification of this compound by PGC and CGC arise from its occurrence in sludge samples at low concentrations and the fact that the chromatographic peak due to aldrin elutes amongst a number of early PCB peaks and is, in most cases, only partially resolved from more intense surrounding peaks. Therefore, co-elution of aldrin with interfering peaks may result in its over-estimation by PGC.

Table 56. Dieldrin concentrations in selected sewage sludges
determined by packed and capillary column gas chromatography
and by capillary column gas chromatography-mass spectrometry

Sample Number	Packed Column GC $\mu\text{g l}^{-1}$	Capillary Column GC $\mu\text{g l}^{-1}$	Capillary Column GC-MS $\mu\text{g l}^{-1}$
1	7.34	3.84	4.07
2	0.68	0.34	0.26
3	28.00	15.52	16.75
4	4.17	3.54	2.97
5	13.8	11.95	9.83
6	2.80	1.18	1.59
7	3.91	4.32	2.19
8	1.76	ND*	ND
9	0.87	0.63	0.66
10	11.71	6.32	7.11
11	2.11	1.87	1.97
12	5.27	2.12	2.63
13	4.33	4.14	4.02
14	6.50	4.29	3.71
15	1.88	1.36	1.09
16	1.55	1.04	1.17
17	0.10	0.11	ND
18	1.09	0.70	0.72
19	3.75	3.46	3.97
20	0.79	ND	ND

* not detected.

Dieldrin concentrations measured by the three analytical techniques are included in Table 56 and varied between 0.10 and 28.00 $\mu\text{g g}^{-1}$ for PGC analyses, between 0.11 and 15.52 $\mu\text{g g}^{-1}$ for the GC-MS analyses. As with the analysis of samples for γ -HCH, concentrations of dieldrin determined by PGC were significantly higher on average than those measured by either CGC or GC-MS. On two occasions (samples 8 and 20) dieldrin was found to be absent from the sample extracts by CGC and GC-MS although chromatographic peaks of the correct retention times had been observed in the PGC analyses. Results of the CGC and GC-MS analyses were found to agree particularly well, with the exception of sample 17, where the concentration of dieldrin measured by both PGC and CGC techniques was not detected by GC-MS. It therefore appeared, from these analyses, that dieldrin analysis by PGC was subject to similar limitations as those of γ -HCH, although to a lesser degree, such that the presence of co-eluting substances in the gas chromatogram resulted in enhancement of detector signals and consequent over-estimation of concentrations in the extracts.

Concentrations of endrin were measured in only 7 of the 20 samples by PGC but these results, contained in Table 57, were not confirmed by either CGC or GC-MS analyses. The ranges in concentrations

Table 57. Endrin concentrations in selected sewage sludges determined by packed and capillary column gas chromatography and by capillary column chromatography-mass spectrometry

Sample Number	Packed Column GC $\mu\text{g l}^{-1}$	Capillary Column GC $\mu\text{g l}^{-1}$	Capillary Column GC-MS $\mu\text{g l}^{-1}$
3	2.50	1.51	1.37
5	1.16	0.88	0.28
10	0.82	0.37	0.42
11	0.09	ND	ND
16	1.04	0.29	ND
18	0.69	ND	ND
19	0.04	0.01	ND

* not detected.

measured by the three methods were 0.04 to 2.50 $\mu\text{g g}^{-1}$ for PGC, 0.01 to 1.51 $\mu\text{g g}^{-1}$ for CGC and 0.28 to 1.37 $\mu\text{g g}^{-1}$ for GC-MS. In samples 3, 5 and 10, endrin was observed to be present at significantly lower concentrations when measured by CGC and GC-MS, than when measured by the PGC technique. The agreement between CGC and GC-MS analyses, observed in the analysis of other compounds, was not evident in this case, particularly for sample 5, where a significantly lower concentration of endrin was measured by GC-MS as compared to CGC, and for sample 16, where endrin was not detected by the GC-MS technique. Reasonable agreement was observed for CGC and GC-MS analyses, however, in samples 3 and 10.

It is evident, from comparison of the results obtained during this exercise by using PGC, CGC and GC-MS techniques, that, where complex samples are analysed, more powerful, advanced analytical techniques may be required in order to give an accurate measure of determinand concentration. However, drawbacks exist with the practical application of such techniques.

5. DISCUSSION

The development and modification of analytical procedures for the determination of persistent organochlorine micropollutants in waste water and waste water sludge samples have enabled the detection and quantification of PCB and four organochlorine insecticides (γ -HCH, aldrin, dieldrin and endrin) in a range of sewage sludges. The availability of multi-residue analytical methods is of importance for studies of this nature since it is possible to maximise the amount of information obtained concerning the presence and concentrations of organic microcontaminants in waste water samples, which is of paramount concern where the methods employed are relatively costly, in terms of time, equipment, materials and labour.

Increased awareness of the contamination of water by a large variety of synthetic organic chemicals and the associated potential risks to public health have resulted in the introduction of water quality criteria and standards in some developed countries for concentrations of specific organic compounds (World Health Organisation, 1971; Council of the European Communities, 1976; United States Environmental Protection Agency, 1975). The establishment of such standards for water is in most cases governed by the availability of information concerning the toxicity of a certain substance to either aquatic organisms (in the case of surface waters) or to laboratory test animals (in the case of water for direct human consumption). In addition, the availability of analytical methods for the determination of designated compounds in water, to the required degree of accuracy, sensitivity and confidence, is a prerequisite. Of concern also, is the presence of organic contaminants in waste waters and particularly in waste water sludges which are disposed to agricultural land or marine sites, where the potential for further contamination of the environment exists. At present, legislation or guidelines concerned with the presence of potentially harmful concentrations of organic micropollutants in sewage sludges are virtually non-existent, such that it is difficult to draw firm conclusions from the data acquired in analytical surveys. However, comparison of results with those obtained in previous studies and from other countries, and with the ranges of concentrations of similar organic substances reported in other environmental samples, will be useful in forming an assessment of whether concern is justified and the implications for sewage sludge disposal mechanisms and practices.

Polychlorinated biphenyls and the organochlorine insecticides γ -HCH and dieldrin have been found to be common micro-contaminants of UK sewage sludge. Aldrin and endrin were observed to occur less frequently in sewage sludges and at concentrations close to the detection limit of the analytical method, which may be explained by the limited use of these two compounds in the UK. In the case of aldrin, the reported epoxidation of this compound to dieldrin in aquatic media may also explain the low concentrations observed (Moriarty, 1975).

Although information pertaining to concentrations of persistent organochlorine compounds in sewage sludges is limited both in terms of the number of surveys conducted and numbers of samples analysed, it is of value here to compare results of the present survey with those of previous exercises carried out in the UK and other countries. Table 58 includes a summary of relevant data arising from previous work. Mean concentrations of PCB measured during this survey are broadly comparable with earlier results obtained in the UK in 1979 (McIntyre & Lester, 1982) and 1972-1974 (Water Research Centre, 1973, 1974). γ -HCH and dieldrin concentrations recorded in the 1979 UK work are approximately one-half those detected in this survey, although this may be accounted for by the relatively small number of samples analysed. The results of an analytical survey conducted during 1978 in the Netherlands (Fieggan, 1981) exhibit lower mean concentrations of both γ -HCH and dieldrin, but higher average concentrations of PCB. A survey in Sweden of 146 sewage sludges in 1971 (de Haan, 1976) reported lower concentrations of γ -HCH and significantly higher mean concentrations of dieldrin and PCB. Concentrations of PCB and OCL reported from minor surveys in Canada, Japan and the USA (Liu *et al.*, 1975; Shannon *et al.*, 1976; Kowase *et al.*, 1977; Furr *et al.*, 1976; Babish *et al.*, 1981) vary considerably in the range 0.02 to 16.04 $\mu\text{g g}^{-1}$ for individual compounds and therefore results from all these surveys appear to be broadly comparable.

The results of the eighteen month survey of sewage sludges from Beckton, Haverhill and Loughborough sewage treatment works have established typical variations in the concentrations of PCB and OCL with time. The serial correlation analyses of these data have been largely inconclusive owing to the relatively small number of samples taken. There are, however, some indications that short-term corre-

Table 58. Comparison of Persistent Organochlorine Micropollutant Concentrations in Sewage Sludges

Country	Year	Number of Samples	Concentration $\mu\text{g g}^{-1}$						Reference
			γ -HCH	Dieldrin	PCB	HCB	Aldrin	Endrin	
UK	1982	445	0.41	0.50	0.34	–	0.03	0.11	This work
Netherlands	1978	202	0.025	0.018	0.950	0.031	–	–	Fiegggen (1981)
Sweden	1971	146	0.28	3.90	1.50	–	–	–	DeHaan (1976)
UK	1979	40	0.21	0.28	0.16	–	–	–	McIntyre & Lester (1982)
UK	1972–1974	22	–	–	0.04–2.68	–	–	–	WRC (1973; 1974)
USA	1973	14	–	0.31	5.15	–	–	–	Furr <u>et al.</u> (1976)
USA	1973	21	–	0.91	9.11	–	16.2	–	16.04 Babish <u>et al.</u> (1981)
Japan	1974	7	–	–	6.35	–	–	–	Kowase <u>et al.</u> (1977)
Canada	1973	40	0.02–0.08	0.04–0.08	–	–	0.02–0.24	–	0.21–0.71 Liu <u>et al.</u> (1975)
Canada	1976	24	–	–	6.26	–	–	–	Shannon <u>et al.</u> (1976)

lations may exist for certain compounds, particularly for PCB and dieldrin in the Haverhill samples. It is not possible to state this with certainty, again owing to the limited data.

The statistical distribution of PCB and OCL has been found to approximate to the log-normal distribution form principally because of the relatively large number of low concentrations observed, which are also influenced by the necessary imposition of an analytical detection limit, and the small number of extreme concentrations encountered. No major differences in concentrations are evident between the various geographical regions in the UK, with the exception of the rural areas of Wales, the South-West and parts of East Anglia, where consistently low concentrations of organochlorine compounds were observed. On a few occasions, particularly high concentrations of PCB and OCL were observed, notably those of dieldrin in several samples from Yorkshire Water Authority region and sporadic occurrences of relatively high concentrations of PCB and γ -HCH in samples obtained from other Water Authority areas. In a number of cases, the occurrence of these elevated concentrations may be related to known industrial uses of certain compounds (such as dieldrin usage for moth-proofing operations in the textile industry and the use of γ -HCH in timber preservation) in particular areas. However, there were also a limited number of cases in which elevated concentrations of determinands occurred in sewage sludges sampled from sewage treatment works receiving domestic sewage. It is possible in these cases, that disposal of surplus residues from household applications may be responsible. No clear indications of a relationship between industrial effluent discharges to sewage treatment works and concentrations of PCB or OCL in the resultant sewage sludges were obtained from the correlation analyses carried out; it appears therefore that a significant proportion of persistent organochlorine residues in raw sewage, and subsequently sewage sludges, may arise from domestic uses and human excretion.

Although insignificant differences were found statistically between the concentrations of PCB and OCL in sludges which had undergone different types of treatment, i.e. sludge treatment was demonstrated to have had no effect upon persistent organochlorine residues, it should be emphasized that the sludges were grab samples and any potential differences in concentrations arising from treatment

almost certainly would have been obscured by confounding variables, such as the spatial variation in residue concentrations. However, this may be an important area of interest, since it has been postulated recently that anaerobic processes may be significant in the preliminary biotransformation of organic molecules, prior to biodegradation (Kobayashi & Rittmann, 1982). The possibility therefore exists for the potential amelioration of organochlorine residues in sewage sludges prior to disposal, by means of anaerobic sludge digestion.

Legislation pertaining to organic contaminants in sewage sludge is limited to the USA, where a maximum concentration of 10 mg kg^{-1} of PCB has been proposed for sewage sludges which are applied to certain types of arable land (United States Environmental Protection Agency, 1979). It is further recommended that any sludge which exceeds the established limit should be incorporated into the soil and not merely spread at the surface. The rationale underlying the guideline is that the Food and Drug Administration tolerance limits for PCB in animal feeds and milk should not be exceeded as a result of the ingestion of PCB-contaminated feed or soil. No guidelines exist for concentrations of other organic residues, although there is a general recommendation that sludge containing 'high' concentrations of potentially toxic organics should be disposed by other means. Although this guideline will contribute to the limitation of PCB residues in animal products, no consideration is given to the possible accumulation of such compounds in the soil to toxic levels, as a result of continued application of sludges containing PCB concentrations below the established limit. Similarly, where it is recommended that sludges with PCB contents greater than the limit should be incorporated into the soil, no account is taken of the possible return to surface layers of PCB-contaminated sludge/soil mixtures by such soil organisms as earthworms.

From the results of the present monitoring exercise, it is apparent that concentrations of persistent organochlorine residues in UK sewage sludges are generally low, with the exception of a few isolated cases in which elevated concentrations occur as the result of specific industrial activity. Short-term incidences of elevated concentrations of PCB or OCL in sewage sludges may be of lesser environmental concern, although the absence of established regular monitoring regimes will limit reporting of their occurrence. Where elevated con-

centrations of organic contaminants are known to occur regularly in certain sewage sludges, it would therefore be advantageous to employ disposal mechanisms which limit both the availability of residues to food chains and the potential for further environmental contamination. Routine analytical monitoring may also be required in some circumstances, in order to detect sporadic occurrence of elevated concentrations.

Although packed column gas chromatography (PGC) has been used with considerable success for the determination of PCB and OCL in environmental samples, the resolving power of a packed gas chromatographic column is somewhat limited where extremely complex sample matrices, such as sewage sludges, are to be analysed. On a number of occasions, during the analysis of UK sewage sludges for PCB and OCL, complex gas chromatographic profiles were generated by the PGC, such that the accurate quantification of particular determinands was precluded by the elution of interfering, co-chromatographed substances. In such cases, there exist three possible choices. By the use of more stringent sample clean-up procedures, the concentrations of interfering substances may be reduced to acceptable levels prior to analysis; a gas chromatographic system of higher resolution and separation power may be employed; or by the use of alternative detection systems, the specificity of the analytical method towards the determinands may be optimised. More rigorous clean-up methods entail an increase in overall analysis time and the possible introduction of errors due to losses in the greater number of sample handling operations and contamination, and for these reasons, such solutions are generally regarded as unacceptable, except where no other alternative exists. The use of capillary column gas chromatography (CGC) offers an acceptable solution, where the resolution of the analysis may be greatly increased, together with improvements in sensitivity, thus resulting in superior chromatographic separation of the determinands from interfering substances. At present, electron-capture detection is the optimum system for use in the analysis of halogenated organic compounds, because of its high sensitivity and good specificity. An acceptable alternative to this procedure lies in the deployment of combined gas chromatography-mass spectrometry, with detection achieved by monitoring selected characteristic ions present in the mass spectra of the individual determinands (selected ion monitoring).

The problems associated with the quantification of PCB and OCL in complex sample matrices have been addressed by use of advanced analytical techniques (CGC and GC-MS) in conjunction with elementary PGC, for the detection of these organochlorine compounds in particularly difficult sample extracts. On the basis of the results obtained (Section 4.6) it is evident that CGC and GC-MS are superior to PGC both in terms of specificity and accurate quantification. In a limited number of cases, problems were also encountered with GC-MS analyses, where low concentrations of PCB or certain OCL could not be detected owing to interference by co-eluting ions of similar m/z value. Since the mass spectrometer could only be operated at a unit mass resolution of 1000 for these analyses, interference of this nature may occur where complex samples contain high molecular weight components which produce intense fragment ions across a broad m/z range. Possible solutions to this problem include high-resolution single ion monitoring techniques, which were not available during these investigations, or the use of negative ion chemical ionisation mass spectrometry, which has been used to good effect for the analysis of chlorinated organics at trace concentrations in complex samples, but which has not yet been thoroughly evaluated (Dougherty *et al.*, 1972). Analyses by CGC and GC-MS compared favourably and it would appear that CGC may be the preferred method since, although more costly to operate than PGC, the quality of information acquired is similar to that of GC-MS, on a quantitative basis, and analysis times are similar to those of PGC, whilst being shorter and less expensive than GC-MS. However, where elevated concentrations are observed in certain samples which cannot be measured effectively using solely gas chromatographic methods, GC-MS techniques represent the only available procedure for reliable identification and quantification of PCB and OCL.

During the course of the primary sedimentation pilot plant experiments, acceptable removals of suspended solids from the raw sewage influent were achieved and it was apparent that the sedimentation tanks were performing satisfactorily with those predicted by three theoretical models is presented in Table 59. It is evident that the average removal efficiencies recorded in these experiments compare well with the values predicted for full-scale tanks by the three theoretical methods, the results lying within the range of the values at lower hydraulic loadings derived by Tebbut and Christoulas (1975).

Table 59. Comparison of suspended solids removal efficiencies achieved by the primary sedimentation pilot plant at different hydraulic loadings and those predicted by theoretical models.

Flow rate 1 s ⁻¹	Retention Time (h)	Surface loading rate (m ³ m ⁻² d ⁻¹)	Removal predicted by Escritt (1972) (%)	Removal predicted by Smith (1969) (%)	Removal predicted by Tebbutt and Christoulas (1975) (%)	This study (%)
0.055	4.6	6.1	74.2	77.7	64.2	75.0
0.111	2.3	12.2	73.2	73.7	57.1	63.6
0.333	0.8	36.6	66.2	59.4	61.9	63.7
0.555	0.5	61.0	60.1	43.9	52.8	59.1

and Smith (1969) and the perhaps somewhat optimistic values predicted for higher hydraulic loadings by Escritt (1972), which have been discussed previously by Anderson (1981). Similarly, the suspended solids removal efficiencies attained at varying influent suspended solids loading, which was a prime variable studied during these experiments, compare favourably with those derived by Tebbut and Christoulas (1975) and those of Anderson (1981), as do the values obtained for non-settleable solids.

The removals of PCB and OCL during the primary sedimentation pilot plant experiments have been found to be variable, although broadly within the range of those reported from previous studies. This is confirmed by the data included in Table 60, where the results of the present preliminary and subsequent pilot plant experiments are compared with results from other authors. No comparative data were available for aldrin and endrin and only one previous study had addressed the removal of γ -HCH during sewage treatment. In general terms, the results obtained during this study are lower on average than those reported elsewhere, although most are based upon the acquisition of a limited quantity of data and have not been derived

Table 60. Comparison of PCB and OCL removals achieved in the primary sedimentation pilot plant with those reported for full scale primary sedimentation plants.

Removal Efficiency %					Reference
γ -HCH	Aldrin	Dieldrin	Endrin	PCB	
-	-	-	-	86.0	Dube <u>et al.</u> (1974)
-	-	-	-	71.0	Berg and Peoples (1977)
-	-	-	-	66.0	Shannon <u>et al.</u> (1976)
-	-	41-54	-	33-75	McIntyre <u>et al.</u> (1981a)
44.0	-	-	-	-	Harper <u>et al.</u> (1977)
-	-	<95	-	-	Bernholz <u>et al.</u> (1971)
-	-	99	-	-	Wilroy (1963)
-	-	-	-	48-69	This study (preliminary)
14.3	4.7-	18.5-	9.2-	9.2-	This study (subsequent)
49.4	80.0	62.2	62.5	41.0	

from treatment plants operated under such a wide variety of solids and hydraulic loadings. In addition, in all but the studies reported by Harper et al. (1977) and McIntyre et al. (1981a), concentrations detected by other workers in raw sewage influents to primary sedimentation tanks were up to five orders of magnitude above those encountered here, and in the case of textile factory effluents considered by Wilroy (1963), raw waste water concentrations of dieldrin were approximately 30 mg l^{-1} . It is perhaps misleading in this respect to consider only the percentage removals of PCB and OCL, and more accurate to compare residual concentrations in settled sewage effluent from primary sedimentation processes, particularly where varying influent concentrations have been reported, since it is apparent that primary sedimentation of raw waste water may be incapable of achieving removal of PCB and OCL down to a certain critical concentration in the settled sewage effluent, which may depend upon the initial influent concentration.

The mechanism of removal of PCB and OCL during primary sedimentation has not been finally clarified by these studies, although there exist strong indications of the possible mode of behaviour of these compounds in waste water. Examination of the results of the primary sedimentation pilot plant study has revealed that there were no apparent relationships observed between the removal of PCB and OCL and the solids and hydraulic loadings applied to the pilot plant, nor was there any firm relationship between the variation in influent concentrations of two undosed compounds (γ -HCH and dieldrin) and their removal during primary sedimentation, within the range of concentrations detected. A consideration of the results obtained from the jar test experiments, together with those from the pilot plant experiments is of value in attempting to elucidate the possible factors influencing removal.

Significant removal of PCB and OCL from raw sewage was effected by centrifugation during the jar tests, with no effects observed due to the presence of varying concentrations of solids, surfactants or grease. Since centrifugation cannot be equated with the more quiescent conditions of sedimentation, non-settleable solids would have been removed from the raw sewage using this procedure, which may explain in part, the discrepancy in the removals of PCB and OCL between the jar tests and the primary sedimentation pilot plant

studies. Further explanation of the removals observed during primary sedimentation may be derived from the jar test results where determinand concentrations were varied. With increasing concentrations of PCB and OCL, it was observed that greater concentrations of determinands were present in the centrifuged samples (particularly γ -HCH and dieldrin), which indicates that these quantities were almost certainly present in the dissolved form or associated with a particularly fine particle size fraction. Therefore, removal of these two forms of PCB and OCL from raw sewage during primary sedimentation may be variable and incomplete and not closely related to the removal of suspended solids, since the particle size fraction with which the determinands may be associated will comprise a small proportion of the total suspended solids burden. It is also probable, that, in the ng l^{-1} range of concentrations at which the determinands occurred in raw sewage during this study, relatively small proportions were associated with settleable solids, whereas at higher concentrations, in the $\mu\text{g l}^{-1}$ range and above, the solubility limit in raw sewage would be attained and the remainder of the determinands would be distributed broadly across the particle size range, thus resulting in elevated removals during primary sedimentation.

As discussed previously, the removal efficiencies per se of persistent organochlorine compounds during primary sedimentation are not as important as the absolute resultant concentration in the primary sludge and settled sewage, since a central aim of sewage treatment is the production of sludges and final effluents of acceptable quality for subsequent disposal to the environment; an overall 90% removal of a particular micropollutant, if present in raw sewage at substantial concentrations, may still result in unacceptable concentrations in the final effluent and would produce unacceptable concentrations in sewage sludges, unless attenuated by further treatment procedures. A typical example may occur in the case of aldrin, dieldrin and endrin, where a draft proposal has been promulgated for the establishment of a quality objective for these three compounds of 5 ng l^{-1} in surface waters (Commission of the European Communities, 1979). Clearly, such a stringent standard would place much emphasis on the need for efficient removal during sewage treatment, particularly at those treatment plants which discharge final effluents to watercourses where dilution is limited.

It is evident that sewage treatment processes are capable of removing, either by chemical and biological degradation or physical means, varying proportions of organic contaminants from waste waters. Whilst the removal of organic substances is deemed desirable when sewage effluents are discharged to rivers in which re-use is practised, the accumulation of potentially toxic organic residues in sewage sludges which are disposed to the environment also gives rise to concern.

Where sewage effluents are discharged to rivers which are abstraction sources it is possible that factors such as hydraulic dilution and natural adsorption and degradation processes in the water course may attenuate concentrations of organic contaminants such that conventional treatment at a subsequent abstraction point will produce potable water of an acceptable quality. In this case it would be difficult to justify installation of treatment processes for more efficient removal of organics unless the natural environment of the water course were proven to be affected. However, if potable water of sufficient quality was not consistently produced, it may be debateable whether to upgrade water or waste water treatment processes, although in this case the effect of organic micropollutants upon the water course may be the deciding factor in favour of employing waste water treatment for organic contaminant removal. This somewhat superficial situation will be complicated in reality by the temporal fluctuations in such parameters as flow and the organic content of effluent discharges, the natural flow and adsorptive and degradative capacities of rivers, and the number and complexities of individual point and non-point discharges, coupled with the possible variations in the effectiveness of treatment processes.

It is evident, therefore, that the removal of organic contaminants from waste water is desirable for many reasons. However, of the processes both natural and anthropogenic which may be applied, degradation by either biological or chemical means would appear to be most expedient, since physical removal of organic compounds in essentially unchanged chemical form may result in concern over the possible effects of the final disposal of such residues in the environment.

6. CONCLUSIONS

- 6.1. Following modifications, previously developed analytical methods for the determination of PCB and OCL in sewages and sewage sludges, using packed column electron-capture gas chromatography, were found to be suitable for the determination of the organochlorine insecticides aldrin and endrin in samples of raw sewage, primary sludge and digested primary sludge.
- 6.2. Analysis of 444 sewage sludge samples in the as disposed form from UK sewage treatment works has revealed that PCB, γ -HCH and dieldrin occurred most frequently and at the highest concentrations, whilst aldrin and endrin occurred less frequently and generally at concentrations near to the analytical detection limit. Data from analysis of the 444 sludge samples were not normally distributed, but were found to approximate to the log-normal distribution form.
- 6.3. With certain exceptions, concentrations of persistent organochlorine compounds did not correlate with proportions of trade effluent discharged to individual sewage treatment works, or with the magnitude of population served by the treatment plants. Apart from the occurrence of low concentrations of PCB and OCL in sludge samples obtained from treatment plants serving rural areas, no relationship was observed between concentrations and geographical location.
- 6.4. No significant differences between the concentrations of organochlorine micropollutants in various types of sludges were observed, when extreme values (the highest 5%), which were not considered to be treatment-related, were excluded from the calculations.
- 6.5. Concentrations of PCB, γ -HCH and dieldrin in eighteen discrete monthly sewage sludges samples from Beckton, Haverhill and Loughborough sewage treatment works were generally low and did not vary markedly over the period of study. Aldrin and endrin occurred infrequently and at concentrations close to the detection limit. A serial correla-

tion analysis of these data yielded additional information in the case of PCB and dieldrin concentrations in the Haverhill samples where a degree of short-term correlation between successive values above and below the mean was evident.

- 6.6. Capillary column gas chromatographic and capillary column gas chromatography-mass spectrometric techniques were found to be suitable, and in most cases superior, for the analysis of PCB, γ -HCH, aldrin, dieldrin and endrin in a subsample of 20 UK sewage sludges, when compared with elementary packed column gas chromatographic techniques. For the greater majority of samples, the increased resolution and specificity of the two advanced analytical techniques facilitated the detection the PCB and OCL in particularly complex sample matrices and reduced the level of interferences such that more accurate measures of determinand concentration were achieved.
- 6.7. A preliminary pilot plant experiment to investigate the behaviour of persistent organochlorine compounds during primary sedimentation of raw sewage, with the pilot plant operating under three different hydraulic loading regimes, revealed that PCB removals were highest under 1 DWF conditions (69%), lowest under 3 DWF conditions (48%) and intermediate (58%) under a typical diurnal variable flow regime.
- 6.8. A series of jar test experiments, undertaken to investigate possible factors which may influence the partitioning of PCB, γ -HCH, aldrin, dieldrin and endrin between solid and liquid phases in raw sewage, demonstrated that varying concentrations of solids, surfactants, grease and determinands did not affect the distribution of PCB and OCL between the two phases and that significant removal of these compounds from raw sewage accompanied the removal of solids by centrifugation.

- 6.9. A further series of pilot plant experiments, conducted over a range of solids and hydraulic loading conditions, have indicated that variable percentage removals of PCB, γ -HCH, aldrin, dieldrin and endrin occurred and that these were largely independent of variations in flow and suspended solids loadings.
- 6.10. An evaluation of the performance of the pilot scale sedimentation plant in terms of suspended solids and COD removal at varying hydraulic loadings indicated that the plant was operating in an acceptable manner and that the removals of suspended solids at different flow rates were within the range of those predicted by available theoretical models.
- 6.11. A comparison of the results of the pilot plant experiments with those obtained from the jar tests have revealed that a significant proportion of PCB and OCL present in raw sewage may be associated with non-settleable solids which are not removed under the normally prevailing conditions of primary sedimentation, but which were removed by centrifugation procedures during the jar test experiments. It was also concluded that a portion of the PCB and OCL in raw sewage may be in the dissolved form.
- 6.12. There appeared to be little relationship between the concentrations of γ -HCH and dieldrin, which were not dosed into the raw sewage influent to the primary sedimentation pilot plant during the experimental programme, and the removal efficiency of these two compounds. An exception occurred in the case of dieldrin, where particularly low influent concentrations on one occasion were poorly removed, coincidentally with a low removal of suspended solids.
- 6.13. The removal of persistent organochlorine micropollutants during waste water treatment and their distribution between sewage sludges and final effluents prior to disposal to the environment constitutes an important area of study in the control of organic micropollutants in the hydrological cycle. The two major areas of concern are, currently, the attenuation of organic contaminants in sewage effluents

which are discharged to re-use rivers and the presence of organic micropollutants in sewage sludges which are disposed to agricultural land and the possible effects of these disposal operations in the environment. It is envisaged that the control of organic substances during waste water treatment processes and sludge disposal operations will become more important in the future, with the impending promulgation of new legislation in Europe and the U.S.A.

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APPENDIX I

Table 1. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Anglian Water Authority Area

Sample Number	Total Solids Concentration g l ⁻¹	Concentration µg g ⁻¹				
		PCB	γ-HCH	Aldrin	Dieldrin	Endrin
1	44.54	1.05	1.11	ND ¹	0.26	ND
2	39.80	1.11	0.21	ND	0.55	ND
3	51.16	0.35	0.11	ND	0.71	ND
4	22.14	0.20	0.09	ND	0.52	ND
5	40.62	0.63	0.18	ND	0.35	ND
6	45.42	0.17	0.14	ND	0.11	ND
7	40.57	0.53	0.25	ND	0.09	ND
8	42.33	0.50	0.06	ND	1.72	0.2
9	41.36	0.72	0.18	ND	0.12	ND
10	289.47	0.16	0.03	ND	0.03	ND
11	318.79	ND ¹	0.02	ND	0.06	ND
12	249.32	ND	0.02	ND	0.07	ND
13	349.71	ND	0.03	ND	0.06	ND
14	334.66	0.07	0.02	ND	0.04	ND
15	72.91	0.56	0.04	ND	0.09	0.06
16	50.10	0.47	0.05	ND	0.11	ND
17	54.47	0.95	0.26	ND	0.26	ND
18	281.79	0.36	0.06	ND	0.11	ND
19	329.83	0.14	0.60	ND	0.04	ND
20	276.47	ND	0.03	ND	0.02	ND
21	82.59	ND	0.09	ND	0.06	ND
22	26.24	0.46	0.10	ND	0.08	ND
23	4.48	ND	0.04	ND	0.02	ND
24	33.16	ND	0.18	ND	0.17	0.03
25	61.55	21.5	0.21	ND	0.39	0.02
26	66.36	0.58	0.12	ND	0.13	0.10
27	61.73	0.50	0.20	ND	0.15	0.02
28	6.35	0.37	0.04	ND	0.10	ND
29	263.79	0.34	0.07	ND	0.14	0.01
30	308.27	0.08	0.03	ND	0.03	0.02
31	297.44	0.31	0.17	ND	0.21	0.04
32	267.93	0.09	1.10	ND	0.13	ND
33	10.87	ND	0.34	ND	0.29	0.03
34	28.42	5.58	0.19	ND	0.12	0.18
35	43.95	0.18	0.18	ND	0.47	0.09
36	40.84	0.24	0.16	ND	0.11	0.05
37	42.61	ND	0.54	ND	0.15	0.05
38	72.30	0.32	0.44	ND	0.10	0.13
39	42.52	0.11	0.66	ND	0.10	0.05
40	23.30	0.45	0.11	ND	0.14	0.03

1 Not detected (<0.01 µg g⁻¹)

Table 2. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Severn-Trent Water Authority Area.

Sample Number	Total Solids Concentration g l ⁻¹	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
1	21.40	0.44	0.05	ND ¹	0.11	ND
2	28.89	0.22	0.21	ND	0.16	0.06
3	44.90	0.14	0.13	ND	0.10	ND
4	20.49	0.07	0.07	ND	0.14	0.01
5	57.40	0.39	0.20	ND	0.21	ND
6	34.16	ND	0.06	ND	0.19	ND
7	64.50	0.14	0.13	ND	0.08	ND
8	18.95	0.08	0.04	ND	0.14	0.02
9	24.40	0.29	0.06	ND	0.72	ND
10	23.87	0.20	0.09	ND	0.27	0.03
11	44.76	0.11	0.37	ND	0.72	0.02
12	29.94	0.19	0.26	ND	0.17	ND
13	40.91	0.11	0.09	ND	0.20	0.03
14	40.40	0.44	0.23	ND	0.19	ND
15	293.76	0.07	0.07	ND	0.34	0.01
16	402.93	ND	0.17	ND	0.13	ND
17	271.68	ND	0.27	ND	0.39	0.02
18	294.72	0.07	0.14	ND	0.10	0.03
19	314.95	0.29	0.15	ND	0.37	0.01
20	261.77	ND	ND	ND	ND	ND
21	407.29	ND	0.05	ND	ND	ND
22	294.75	0.12	0.19	ND	ND	ND
23	260.14	ND	ND	ND	ND	ND
24	372.66	0.17	ND	ND	ND	ND
25	273.29	0.11	0.23	ND	0.02	ND
26	281.27	0.10	0.24	ND	0.24	0.07
27	327.63	ND	ND	ND	0.01	0.17
28	31.65	0.23	0.13	ND	0.67	0.04
29	45.06	0.07	0.17	ND	0.33	0.04
30	37.60	0.11	1.47	ND	0.19	0.09
31	7.31	2.31	0.28	ND	0.03	0.05
32	781.90	ND	0.08	ND	0.10	0.02
33	874.10	ND	0.12	ND	0.04	0.02
34	267.45	0.07	0.10	ND	0.02	ND
35	241.79	ND	0.04	ND	ND	ND
36	317.61	ND	0.16	ND	0.03	ND
37	58.61	0.13	0.10	ND	0.13	0.04
38	272.56	0.06	0.06	ND	0.01	ND
39	907.50	ND	0.10	ND	0.02	ND
40	264.92	0.08	0.41	ND	0.04	0.20
41	329.74	0.15	0.08	ND	0.03	0.05
42	27.82	ND	0.33	ND	0.06	ND

1 Not detected ($<0.01 \mu\text{g g}^{-1}$)

Table 3. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Thames Water Authority Area.

Sample Number	Total Solids Concentration g l ⁻¹	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
1	372.0	0.29	0.24	0.01	0.32	0.10
2	41.4	0.08	0.26	ND	0.33	0.17
3	327.1	0.10	0.21	ND	0.26	0.13
4	51.8	0.18	0.37	0.02	0.05	0.20
5	27.7	0.18	0.01	ND	0.35	ND
6	386.9	ND ¹	0.58	ND	0.54	0.19
7	212.3	ND	0.12	ND	0.89	0.12
8	19.5	0.14	0.12	ND	0.15	0.06
9	29.0	0.05	0.37	ND	0.33	0.14
10	224.5	0.06	0.65	ND	0.02	0.07
11	33.6	0.26	0.02	ND	0.37	0.21
12	227.3	0.12	0.14	0.01	0.60	ND
13	35.1	0.15	0.01	0.01	0.33	0.14
14	25.9	0.23	0.10	ND	0.30	0.27
15	311.3	0.04	0.10	ND	0.21	ND
16	339.6	0.04	0.02	ND	0.39	0.04
17	270.9	0.16	ND	ND	ND	0.06
18	39.2	0.35	0.01	ND	0.22	1.71
19	527.5	0.15	0.11	0.02	0.47	ND
20	29.1	0.54	0.04	0.06	0.63	0.27
21	20.7	0.16	0.03	ND	ND	0.13
22	31.6	0.33	0.67	ND	0.57	0.67
23	20.8	0.09	0.05	0.02	0.25	0.09
24	288.5	0.11	1.31	ND	0.32	0.11
25	102.0	0.16	0.02	ND	0.28	0.07
26	880.0	0.06	ND	ND	0.10	ND
27	25.6	ND	0.06	ND	0.08	0.07
28	324.5	0.06	0.06	ND	0.22	0.08
29	338.7	0.40	0.07	ND	0.11	ND
30	54.7	0.11	0.16	ND	0.18	0.22
31	221.4	0.29	0.19	0.01	0.15	0.43
32	40.4	0.18	*	ND	*	*
33	61.5	0.18	0.27	0.02	0.25	0.20
34	22.2	0.11	0.03	ND	0.18	0.14
35	44.0	0.06	0.18	ND	0.16	0.25
36	42.4	0.05	0.33	ND	0.14	0.33
37	30.9	0.20	0.05	ND	0.10	0.24
38	34.1	0.16	0.24	ND	0.50	0.38
39	42.5	0.12	0.35	ND	0.34	0.20
40	36.4	0.02	0.30	0.02	0.19	0.14
41	23.1	0.24	0.38	0.01	0.06	0.23

¹ Not detected ($<0.01 \mu\text{g g}^{-1}$)

* Quantification impossible owing to gross interference

Table 4. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Welsh Water Authority Area

Sample Number	Total Solids Concentration g l ⁻¹	Concentration µg g ⁻¹				
		PCB	γ-HCH	Aldrin	Dieldrin	Endrin
1	18.9	0.21	0.04	ND	0.06	0.13
2	19.6	ND	0.12	ND	0.22	ND
3	64.3	ND	0.52	ND	0.27	0.21
4	50.2	ND	0.08	ND	0.18	0.20
5	17.7	ND	0.01	ND	0.02	0.04
6	40.8	ND	ND	ND	ND	0.22
7	34.6	0.22	0.03	ND	0.14	ND
8	23.3	0.11	0.34	ND	0.15	0.11
9	31.8	0.25	0.11	ND	0.19	0.07
10	10.9	0.30	0.11	0.04	0.48	0.07
11	27.4	0.15	0.09	ND	ND	ND
12	50.5	0.25	0.30	ND	0.08	0.11
13	875.9	0.08	0.16	ND	0.04	ND
14	747.2	ND	0.02	ND	0.02	ND
15	832.7	ND	0.02	ND	ND	ND
16	58.92	0.13	0.02	ND	0.13	0.04
17	865.1	ND	0.04	ND	0.01	ND
18	793.4	0.73	0.10	0.02	0.01	ND
19	841.5	ND	0.19	ND	0.01	ND
20	884.6	ND	0.06	ND	ND	0.01
21	890.9	ND	0.01	ND	0.09	0.10
22	847.3	0.03	0.01	ND	0.01	0.02
23	43.2	0.45	ND	ND	ND	0.14
24	54.9	1.12	0.15	ND	0.01	0.39
25	46.5	0.17	0.61	ND	0.01	0.07
26	4.5	0.47	0.02	0.02	0.04	0.20
27	18.2	0.16	70.00	0.01	ND	0.02
28	22.2	ND	ND	ND	ND	ND
29	59.7	0.06	0.31	ND	0.40	0.16
30	55.5	0.03	0.59	ND	0.32	0.07
31	44.8	0.05	0.09	0.01	0.11	0.11
32	35.1	0.03	1.08	ND	0.11	0.14
33	830.2	ND	0.17	ND	ND	ND
34	884.0	0.01	0.09	ND	ND	0.07
35	891.1	0.03	0.05	ND	0.01	0.02
36	860.0	0.04	0.08	ND	0.02	0.03
37	882.7	ND	0.67	ND	ND	ND
38	847.8	0.02	0.06	ND	0.02	0.02
39	799.4	0.11	0.01	ND	0.07	0.05
40	871.7	0.07	0.47	ND	0.05	0.04

1 Not detected (<0.01 µg g⁻¹)

Table 5. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Northumbrian Water Authority Area.

Sample Number	Total Solids Concentration g l ⁻¹	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
1	699.0	0.19	0.01	ND ¹	0.14	ND
2	256.0	0.20	0.16	0.11	0.07	0.11
3	391.0	ND	0.02	ND	0.03	ND
4	38.3	0.04	0.37	ND	0.50	ND
5	116.7	0.11	ND	0.09	ND	ND
6	54.0	0.04	0.01	0.01	0.25	ND
7	86.0	0.03	0.17	ND	0.19	0.12
8	21.4	0.17	0.03	0.01	ND	ND
9	17.9	0.03	0.13	0.01	0.03	0.07
10	50.5	ND	0.59	ND	0.52	ND
11	93.1	0.04	0.06	0.01	0.05	0.06
12	51.7	0.03	ND	ND	0.08	0.14
13	351.0	0.03	0.07	ND	0.05	ND
14	237.0	0.02	0.03	ND	0.16	ND
15	110.0	0.09	0.04	0.02	0.08	0.09
16	21.2	0.04	0.17	0.01	0.16	0.27
17	76.0	ND	0.08	0.06	0.03	0.04
18	68.6	0.04	0.22	0.01	5.54	ND
19	71.6	0.04	0.04	0.01	0.17	0.07
20	26.7	0.06	0.31	0.03	0.11	0.84
21	104.5	ND	0.03	ND	0.03	0.02
22	120.0	0.04	1.00	ND	0.01	ND
23	85.0	0.05	0.06	ND	0.03	0.04
24	58.2	0.05	0.06	ND	0.49	0.72
25	41.2	0.03	0.02	ND	ND	ND
26	26.5	ND	0.41	ND	0.06	0.17
27	77.0	0.02	0.27	0.01	0.03	0.02
28	606.0	ND	0.06	ND	0.09	ND
29	19.1	0.14	0.02	ND	0.16	0.07
30	67.4	0.06	ND	ND	ND	ND
31	56.7	0.06	0.08	0.01	0.16	0.14
32	34.6	0.05	1.08	0.13	0.02	0.06
33	530.0	0.02	0.01	0.01	0.05	0.04
34	183.0	0.15	0.12	ND	0.10	0.04
35	100.0	0.02	0.01	ND	0.03	0.04
36	220.0	0.04	0.02	0.01	0.08	ND
37	20.0	ND	ND	ND	0.06	0.11
38	102.2	ND	ND	ND	ND	0.02
39	245.0	0.11	0.03	0.01	0.08	0.06
40	66.7	0.06	0.28	ND	0.10	0.08
41	22.1	0.09	ND	ND	0.25	0.13
42	38.5	0.37	0.01	0.02	0.16	0.04
43	52.0	0.10	0.08	0.02	0.24	0.10
44	40.8	0.06	0.02	0.09	0.09	0.17
45	47.7	0.16	0.15	0.01	0.03	0.77
46	17.0	0.04	0.03	ND	ND	ND
47	57.4	0.05	0.17	ND	0.07	0.11

1 Not detected ($<0.01 \mu\text{g g}^{-1}$),

Table 6. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Yorkshire Water Authority Area

Sample Number	Total Solids Concentration g l ⁻¹	Concentration µg g ⁻¹				
		PCB	γ-HCH	Aldrin	Dieldrin	Endrin
1	57.6	0.44	ND ¹	0.01	1.22	ND
2	48.5	0.15	0.20	ND	0.06	0.01
3	182.0	0.12	0.06	0.03	0.01	ND
4	39.9	0.04	ND	ND	0.10	ND
5	49.9	0.08	0.01	0.01	0.06	ND
6	146.0	0.05	0.03	ND	0.11	ND
7	58.9	0.11	0.15	ND	0.16	ND
8	18.0	4.30	2.36	0.01	0.50	ND
9	31.1	0.13	0.02	0.21	0.10	ND
10	362.0	0.10	0.46	ND	1.45	ND
11	884.0	0.29	ND	0.12	4.71	ND
12	66.7	0.16	0.11	0.01	1.86	0.02
13	462.0	0.66	0.36	0.04	52.94	0.23
14	60.2	0.90	0.14	0.04	4.06	0.20
15	231.0	0.10	0.10	0.18	3.64	0.17
16	152.0	0.19	0.02	0.06	0.10	ND
17	335.0	0.22	0.18	0.04	0.45	0.12
18	617.0	0.12	ND	ND	2.20	ND
19	289.0	0.14	ND	0.07	0.17	0.17
20	17.6	0.11	ND	ND	0.04	ND
21	107.0	0.21	ND	0.05	0.26	ND
22	31.3	0.08	ND	ND	0.03	ND
23	70.7	0.24	0.02	0.01	0.46	ND
24	106.4	0.67	ND	0.15	0.45	0.02
25	62.0	0.10	ND	0.01	0.04	ND
26	58.2	0.09	0.13	0.02	0.24	ND
27	18.1	0.15	0.23	ND	0.28	ND
28	34.7	0.06	0.07	ND	0.16	ND
29	39.6	0.16	0.28	0.01	0.05	ND
30	68.6	0.25	0.41	0.01	0.44	0.01
31	29.9	0.07	ND	ND	0.04	ND
32	177.0	0.19	0.04	0.05	0.06	0.05
33	61.6	0.10	0.18	ND	0.50	ND
34	47.4	0.07	0.06	ND	0.04	ND
35	1.5	0.11	0.16	0.02	0.14	ND
36	24.4	0.42	0.02	0.03	0.09	ND
37	86.5	0.10	9.64	0.02	0.13	ND
38	70.6	0.14	ND	0.01	ND	ND
39	27.0	0.34	0.07	0.06	0.43	0.03
40	38.3	1.54	0.59	0.02	0.21	0.01
41	347.0	0.16	0.28	0.02	0.18	ND
42	31.3	0.17	0.02	0.04	0.20	0.01
43	48.5	0.22	0.10	0.01	0.52	ND
44	32.7	0.80	0.64	ND	0.15	ND
45	42.1	0.12	ND	0.01	0.25	ND
46	55.8	0.16	0.07	ND	0.25	ND
47	620.0	0.69	ND	0.02	1.31	0.02

1 Not detected (<0.01 µg g⁻¹)

Table 7. Concentrations of persistent organochlorine compounds in sewage sludges sampled from North-West Water Authority Area

Sample Number	Total Solids Concentration g l ⁻¹	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
1	10.41	ND ¹	0.32	ND	0.25	ND
2	22.54	2.13	0.04	ND	0.09	0.04
3	13.47	0.15	0.02	0.02	0.07	ND
4	29.72	ND	0.01	0.03	0.32	ND
5	21.31	0.32	0.04	ND	0.21	ND
6	19.00	0.21	0.02	0.01	0.06	0.06
7	29.83	0.07	0.06	ND	0.28	ND
8	9.58	ND	ND	ND	0.03	ND
9	37.72	0.12	0.02	ND	0.14	ND
10	17.22	0.06	0.03	ND	0.12	ND
11	27.19	1.46	0.02	0.02	0.05	ND
12	37.50	0.09	0.02	ND	0.07	0.02
13	19.85	0.17	0.01	ND	0.45	ND
14	30.96	0.08	0.05	ND	0.12	ND
15	36.55	0.09	0.24	ND	0.06	ND
16	22.94	0.32	0.06	ND	0.08	ND
17	10.24	0.04	0.14	ND	0.20	0.13
18	40.25	1.46	0.13	ND	0.20	0.10
19	22.47	0.15	0.05	0.04	0.09	ND
20	4.40	0.15	0.17	ND	0.07	ND
21	30.28	0.09	0.12	0.01	0.26	0.13
22	31.48	0.07	0.02	ND	ND	ND
23	33.03	0.35	0.19	ND	ND	ND
24	18.27	0.62	0.08	ND	0.89	ND
25	58.42	0.10	0.12	ND	0.32	ND
26	88.63	0.17	0.14	0.05	0.13	ND
27	41.52	0.35	10.15	ND	1.61	ND
28	12.38	ND	0.26	ND	0.23	ND
29	33.38	0.08	0.04	ND	0.10	ND
30	22.88	0.17	0.03	ND	0.04	0.04
31	26.73	1.56	ND	ND	0.14	ND
32	20.78	0.46	0.03	ND	0.27	ND
33	45.22	0.40	0.02	ND	0.05	ND
34	20.78	0.05	0.02	ND	0.02	ND
35	26.98	0.03	0.11	0.01	0.02	ND
36	18.00	0.07	0.09	ND	0.01	ND
37	27.93	0.11	0.05	0.01	0.03	ND
38	29.93	0.04	0.08	0.03	0.07	ND
39	32.36	1.35	0.05	ND	0.06	ND
40	43.66	0.94	0.09	ND	0.05	ND
41	321.73	0.13	0.04	ND	0.54	ND
42	297.61	0.12	0.06	ND	0.08	ND
43	342.79	0.08	0.05	ND	0.07	ND
44	284.62	0.07	0.14	ND	0.04	ND

1 Not detected ($<0.01 \mu\text{g g}^{-1}$)

Table 8. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Wessex Water Authority Area

Sample Number	Total Solids Concentration g l ⁻¹	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
1	20.60	0.05	0.09	ND	0.18	0.02
2	60.33	ND ¹	0.14	ND	0.06	ND
3	42.71	ND	0.32	0.01	0.24	ND
4	27.75	0.27	0.13	ND	28.73	0.04
5	80.00	0.14	0.09	ND	0.18	ND
6	39.50	0.06	ND	ND	0.11	ND
7	30.00	0.02	0.21	ND	0.24	0.05
8	24.00	0.09	0.73	ND	0.98	0.04
9	43.80	0.07	0.14	0.01	0.61	0.07
10	37.30	0.11	0.05	ND	0.25	ND
11	25.24	0.29	ND	ND	0.34	ND
12	43.80	0.09	0.14	0.01	0.41	ND
13	20.10	0.31	ND	0.01	0.74	ND
14	36.00	0.08	0.08	0.02	0.31	ND
15	34.48	0.01	0.06	ND	0.66	0.02
16	51.55	0.04	0.11	0.02	0.09	0.08
17	20.80	0.02	0.04	ND	0.28	0.07
18	55.54	0.12	0.09	ND	0.38	ND
19	323.00	0.08	0.04	ND	0.20	0.01
20	148.50	0.10	ND	ND	0.34	ND
21	120.40	0.14	0.13	ND	0.21	ND
22	187.60	0.03	0.09	ND	0.19	ND
23	289.00	0.01	0.06	ND	0.13	0.03
24	84.05	ND	0.30	ND	2.05	0.01
25	107.40	0.08	0.23	ND	0.18	0.02
26	305.70	ND	0.03	ND	0.06	ND
27	203.70	ND	0.04	0.01	0.16	ND
28	536.60	ND	ND	ND	0.05	ND
29	171.70	0.07	0.75	ND	0.16	0.02
30	263.10	ND	0.26	ND	0.50	ND
31	109.30	ND	0.16	ND	0.17	ND
32	292.30	ND	0.54	0.01	0.25	ND
33	184.40	0.05	0.17	ND	0.20	ND
34	209.80	ND	1.04	0.02	0.18	ND
35	93.50	0.07	1.11	ND	15.51	0.01
36	88.20	0.12	2.05	ND	2.15	0.04

1 Not detected ($<0.01 \mu\text{g g}^{-1}$)

Table 9. Concentrations of persistent organochlorine compounds in sewage sludges sampled from South-West Water Authority Area

Sample Number	Total Solids Concentration g l ⁻¹	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
1	212.40	0.18	0.05	0.01	2.93	ND
2	19.56	0.07	ND	0.02	0.06	ND
3	366.10	0.01	0.01	ND	0.05	ND
4	306.50	0.02	0.01	ND	0.18	ND
5	255.20	ND ¹	ND	ND	0.11	ND
6	286.30	ND	ND	ND	0.12	0.03
7	265.60	0.14	ND	ND	0.09	ND
8	37.80	0.08	0.03	0.03	0.25	0.06
9	89.80	0.01	0.15	ND	0.31	0.04
10	208.80	0.03	ND	0.01	ND	0.01
11	81.02	0.20	0.08	ND	0.27	0.02
12	196.90	0.07	0.18	ND	1.53	ND
13	53.19	0.02	ND	ND	0.29	0.02
14	384.00	0.04	0.02	ND	0.02	ND
15	124.90	0.09	2.86	ND	0.19	ND
16	212.20	0.11	0.63	ND	0.32	0.03
17	259.10	0.05	0.06	ND	0.42	ND
18	178.60	0.01	0.13	ND	0.31	ND
19	212.90	0.09	0.07	ND	0.83	0.02
20	308.50	ND	0.03	0.02	0.06	ND
21	381.90	0.03	0.06	0.01	0.05	ND
22	132.90	ND	0.17	ND	0.30	ND
23	157.20	0.10	ND	ND	0.14	ND
24	41.44	0.10	0.04	0.01	1.09	ND
25	67.11	0.01	0.40	ND	0.37	0.05
26	14.75	0.06	0.09	0.04	0.39	ND
27	260.70	0.05	0.05	0.01	0.20	ND
28	134.60	ND	0.21	0.01	0.22	ND
29	199.10	0.08	0.06	ND	0.13	0.01
30	396.40	0.20	0.15	ND	0.20	ND

1 Not detected ($<0.01 \mu\text{g g}^{-1}$)

Table 10. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Southern Water Authority Area

Sample Number	Total Solids Concentration g l ⁻¹	Concentration µg g ⁻¹				
		PCB	γ-HCH	Aldrin	Dieldrin	Endrin
1	70.56	0.14	0.14	0.02	0.14	ND ¹
2	30.10	0.07	0.16	0.04	0.17	0.01
3	99.99	0.07	0.03	ND	0.01	ND
4	53.34	0.28	1.21	ND	0.19	ND
5	66.91	0.10	0.32	ND	0.01	ND
6	84.30	0.17	0.63	ND	0.17	ND
7	17.45	0.12	0.87	ND	0.11	ND
8	19.31	0.42	0.20	0.06	0.15	ND
9	24.00	0.15	0.56	0.03	0.05	ND
10	64.00	0.08	0.57	ND	0.07	ND
11	88.60	0.07	0.02	ND	0.03	ND
12	540.10	0.08	0.17	ND	0.11	ND
13	138.60	0.08	0.25	ND	0.16	ND
14	887.90	0.17	0.06	ND	0.07	ND
15	393.80	0.12	0.19	ND	0.67	ND
16	317.50	0.09	0.20	ND	0.08	ND
17	279.60	0.05	0.14	ND	0.16	ND

1 Not detected (<0.01 µg g⁻¹)

Table 11. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Scotland

Sample Number	Total Solids Concentration g l ⁻¹	Concentration µg g ⁻¹				
		PCB	γ-HCH	Aldrin	Dieldrin	Endrin
1	32.00	0.93	0.05	ND ¹	0.05	0.10
2	34.00	0.34	0.05	0.03	0.04	ND
3	40.32	0.51	0.65	0.04	0.07	ND
4	86.29	0.29	0.03	0.04	0.14	0.13
5	56.40	0.57	0.07	0.03	0.07	0.07
6	253.07	0.51	0.04	0.04	0.07	ND
7	405.36	0.34	0.06	0.02	0.08	ND
8	10.20	0.61	0.06	0.01	0.06	0.06
9	35.46	0.54	0.09	0.06	0.05	ND
10	32.03	0.72	0.26	0.04	0.05	0.16
11	38.77	0.59	0.08	0.04	0.06	0.19
12	38.24	0.24	0.04	0.03	0.06	ND
13	72.97	0.15	0.08	0.04	0.06	0.04
14	83.56	0.74	0.05	0.06	0.04	0.10
15	55.79	0.29	0.06	0.04	0.07	0.08
16	321.64	0.26	0.08	0.05	0.07	0.29
17	197.40	0.17	0.04	0.05	0.06	ND
18	13.00	0.22	0.03	0.03	0.07	0.07
19	60.37	1.02	0.03	0.09	0.13	0.39
20	26.49	0.23	0.09	ND	0.05	0.10
21	20.96	0.16	0.07	0.04	0.06	0.22
22	168.70	0.47	0.11	ND	0.06	0.11
23	38.63	2.59	0.04	ND	0.04	0.01
24	266.07	6.63	0.11	ND	0.03	0.02
25	456.49	0.12	0.03	0.03	0.04	ND
26	332.01	1.02	0.14	0.05	0.09	ND
27	32.50	0.80	0.03	0.02	0.04	0.05
28	41.24	0.70	0.07	ND	0.06	0.07
29	30.92	0.40	0.06	0.02	0.03	ND
30	93.07	0.62	0.05	0.03	0.03	ND

1 Not detected (<0.01 µg g⁻¹)

Table 12. Concentrations of persistent organochlorine compounds in sewage sludges sampled from Northern Ireland

Sample Number	Total Solids Concentration g l ⁻¹	Concentration $\mu\text{g g}^{-1}$				
		PCB	γ -HCH	Aldrin	Dieldrin	Endrin
1	57.00	0.30	0.02	0.01	0.10	ND
2	10.50	0.19	0.01	ND ¹	0.07	ND
3	50.60	0.03	0.01	ND	0.12	ND
4	51.03	0.11	0.01	ND	0.17	ND
5	200.00	0.24	0.03	0.01	0.11	ND
6	60.00	0.02	0.02	ND	0.12	ND
7	64.00	0.62	0.01	0.01	0.08	ND
8	11.20	0.13	0.02	0.02	0.07	ND
9	100.00	0.19	0.01	0.02	0.10	ND
10	470.00	0.05	0.01	ND	0.11	ND
11	18.00	0.25	ND	ND	0.12	ND
12	84.00	0.45	0.01	0.02	0.11	ND
13	34.00	0.09	0.01	ND	0.09	0.01
14	7.70	0.03	0.02	ND	0.09	ND
15	25.00	0.02	0.05	ND	0.15	ND
16	21.20	0.05	0.03	ND	0.13	0.01
17	49.00	0.46	ND	0.03	0.07	ND
18	61.00	0.19	0.01	ND	0.07	0.16
19	24.00	0.23	0.03	ND	0.11	0.08
20	113.00	0.39	ND	ND	0.06	ND
21	18.70	0.23	0.01	ND	0.10	0.01
22	73.00	1.07	0.02	0.02	0.23	0.15
23	34.00	0.07	0.01	ND	0.09	ND
24	7.96	0.15	0.01	0.01	0.05	ND
25	211.00	0.16	0.01	ND	0.08	ND
26	188.00	0.07	0.02	ND	0.21	0.02
27	57.00	0.37	0.07	0.01	0.77	ND
28	73.00	0.11	0.14	ND	0.97	ND
29	46.00	0.28	0.05	0.02	0.62	0.01
30	28.00	0.11	ND	ND	0.07	ND

* Not detected ($<0.01 \mu\text{g g}^{-1}$)