Studies at Iona Island Municipal Sewage Treatment Plant: Development of an Analytical Method for Wastewater Analysis

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STUDIES AT IONA ISLAND MUNICIPAL SEWAGE TREATMENT PLANT: DEVELOPMENT OF AN ANALYTICAL METHOD FOR WASTEWATER ANALYSIS

by

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ABSTRACT

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Twelve samples of Iona Island sewage, collected in the period March through September 1982, were processed by liquid/liquid extraction and analysed by GC-FID and GC-MS. About one hundred neutral or acidic compounds were tentatively identified. From this list ten base/neutral and three acidic compounds were selected as spiking standards. As internal standards the compounds 1, 2 - dibromo - 4, 5 - dimethoxybenzene and methyl margarate were selected. Response factors were measured by GC-FID on all the spiking standards relative to each internal standard.

Recovery studies were conducted on the spiking standards injected into tap water. Continuous liquid/liquid extraction with methylene chloride at pH 10 followed by further extraction at pH 2, was succeeded by extraction with diethyl ether in a separatory funnel. Recoveries of the spiking standards from tap water were measured as 100 ± 5% by this procedure.

The method was used to quantify two samples of raw and two samples of treated sewage. Fatty acids followed by phthalic acid and aromatic monobasic acids were the major components of the raw and treated sewage. The only chlorinated compounds detected were two isomers of dichlorobenzene, trichloropropene and an unidentified compound of apparent molecular weight 218, all of which were present in the raw sewage in the 1 to 10 µg/l range.

It is extremely unlikely that acute toxicity to fish is associated with the organics based on studies to date. However, until fish tissue samples from exposure tests are analysed, it is not possible to decide whether bioaccumulation of toxic substances may be a problem.

Key words: Domestic sewage, disinfection, chlorinated organics, analytical methods development, hydrocarbons, phenols, Fraser estuary.

RESUME

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Le présent rapport porte sur le traitement de douze échantillons d'eaux usées recueillis à l'île Iona de mars à septembre 1982. L'extraction et l'analyse ont été réalisées par chromatographie de partage liquide-liquide et à l'aide d'un CPG-DIF et d'un CPG-SM respectivement. On a identifié environ 100 composés acides ou neutres dont 10 composés basiques ou neutres et trois composés acides ont été choisis comme étalons mobiles. Le dibromo-1,2 dimethoxy-4,5 benzène et le méthylmargarate constituent les étalons stationnaires.

On a mené des études de récupération des étalons mobiles injectés dans de l'eau de robinet. L'extraction continue liquide-liquide avec du chlorure de méthyl à un pH 10 et un pH 2 a été suivie d'une extraction avec de l'éther de diéthyle dans une ampoule à décanter. La récupération des étalons mobiles de l'eau de robinet à l'aide de cette méthode s'élevait à 100 ± 5%.

On a utilisé cette procédure pour quantifier deux échantillons d'eaux usées brutes et deux échantillons traités. Des acides gras, suivis d'acide phthalique et d'acides aromatiques monobasiques étaient les principales composantes des eaux usées traitées et non traitées. Deux isomères du dichlorobenzène, du trichlorobenzène et d'un composé chlorés non identifié d'un poids moléculaire apparent de 218 constituaient les seuls composés chlorés détectés. Tous étaient présents dans les eaux usées non traitées en proportion variant de 1 à 10 ug/L.

Il est peu probable que la toxicité aiguë sur les poissons soit associée avec les composés organiques étudiés jusqu'à maintenant. Toutefois, il est impossible de déterminer si la biaccumulation des substances toxiques constitue un problème avant l'analyse des échantillons de tissus de poissons soumis à des tests d'exposition.

Mots-clés: eaux usées domestiques, assainissement, composés organiques chlorés, mise au point de méthodes analytiques, hydrocarbures, phénols,estuaire du Fraser

INTRODUCTION

In July 1980 the West Vancouver Laboratory of the Department of Fisheries and Oceans recognized the existence of severe environmental stress in fish populations resident near a municipal sewage outfall at Iona Island in the Fraser River estuary. The Iona Island Sewage Treatment plant is operated by the Greater Vancouver Regional District. It provides primary treatment to predominantly household sewage collected from a wide area in Greater Vancouver and the University Endowment Lands. Approximately 10% by volume of the plant inflow is industrial wastewater coming from various sources including an oil refinery, a number of hospitals and the University of British Columbia. The discharged wastewater flows down a channel in the tidal mudflats beside a rock jetty and disperses into the waters covering Sturgeon Bank. It was observed that very low oxygen levels prevailed in the water along the drainage channel and adjacent waters during hot summer days with little wind action. At various stages of the tidal cycle, components of the marine foodchain were severely stressed. Seagulls and blue herons were observed feeding on flounders and other fish swimming at the water surface. The condition is usually ameliorated with the influx of oxygenated water on the rising tide. Various aspects of the problem have been described elsewhere (Birtwell et al. 1982).

A study of the uptake of contaminants from Iona Island sewage by juvenile chinook salmon commenced in 1982. The salmon were maintained in troughs containing various dilutions (0, 1, 5, 15%) of untreated sewage. Qualitative and quantitative analytical methods were required for sewage analysis during the fish exposure studies. This report describes the tests performed in laboratory work leading to the development of a quantitative analytical method for organics. The qualitative and quantitative data obtained on the chemical composition of the wastewater during April through September 1982 are presented.

METHODS

EXTRACTION

In preliminary tests, clean water samples (1 L) were spiked with known concentrations of standard neutral and acidic components typical of municipal wastewaters. The list of spiking standards selected is shown in Table 1. The pH was adjusted to 10 and the samples extracted with 3 x 100 mL portions of dichloromethane, diethyl ether or combinations thereof in separatory funnels. The combined extracts were dried over anhydrous sodium sulphate, spiked with an internal standard and concentrated to 1 mL in a Kuderna-Danish apparatus. The water samples were then acidified to pH 2 and re-extracted as before. These extracts were similarly spiked with the internal standard and concentrated.

To avoid problems with emulsions that formed when applying the above extraction procedure to Iona sewage, further studies were conducted with liquid/liquid extractors for downward or upward displacement. The geometry of the apparatus dictated the extraction of 1800 mL wastewater samples. Uniformity of extraction was assisted by placing a small stirring bar magnet in the bottom of the extraction apparatus and stirring the solvent phase. Base/neutral compounds were extracted at pH 10 using dichloromethane and acidic compounds were removed later at pH 2 using either dichloromethane or dichloromethane/ether (4:1). Finally, the spent wastewater samples were shaken in a separatory funnel with diethyl ether and this extract combined with the extract from the acidic fraction. The combined extract was concentrated in a Kuderna-Danish apparatus, methylated with diazomethane and concentrated to 1 mL for analysis. Solvent blanks were run using Burdick and Jackson pesticide-grade solvents to check for interfering substances.

A variety of compounds was examined for suitability as internal standards. The list included 2, 4 - dibromophenol, p - bromophenol, 2, 6 - difluorobenzoic acid, methyl margarate and 1, 2 - dibromo - 4, 5 - dimethoxybenzene. The latter two were selected because they were free from interferences with the major components of the sewage.

ORGANICS ANALYSIS

Extracts were analysed by GC-FID using a Hewlett-Packard 5700 F.I.D. gas chromatograph equipped with a capillary inlet system and a 30 m x 0.32 mm Durabond-5 fused silica capillary column purchased from J and W Scientific Incorporated. The oven temperature commenced at 80°C and was programmed to 280°C at 8°C/min. The carrier gas was helium at a flow rate of 1.5 cc/min.

Extracts were also analysed using a Hewlett-Packard 5992 B GC-MS desk-top instrument fitted with a capillary column injection system (and a cryogenic cooling device). The oven temperature was programmed from 80°C to 290°C at 10°C/min. The ion source was maintained at 70 electron volts and the instrument was operated in the peakfinder mode using perfluorotributylamine for automatic internal calibration. A fused silica capillary column, identical to that used on the GC-FID instrument, was employed. Mass spectra were identified by manual searching of the Eight Peak Index Aldermaston file and the EPA-NIH file. All major components identified were confirmed by comparing their mass spectra and retention times with those of standard compounds.

SPIKING STANDARDS AND QUANTIFICATION

A number of samples of treated sewage were collected and qualitatively analysed during March, April and May, 1982. The compounds identified in the base/neutrals fractions are listed in Table 2 and those in the acidic fractions in Table 3. A summary was prepared of the frequency of occurrence of these compounds in the total of nine samples that were analysed and this is shown in Table 4. Thirteen compounds, representative of the major classes of organics in these samples, were selected as spiking standards (See Table 4). A solution in methylene chloride was prepared such that 5 mL injected into a 1 - L wastewater sample gave a spiking concentration of 100 ppb. The compounds 1, 2 - dibromo-4, 5 - dimethoxybenzene and margaric acid were selected as internal standards. These were prepared separately in dichloromethane solution at a concentration equivalent to that of the spiking standards. However, the internal standards were added to the base/neutral and acid extracts after solvent extraction and prior to concentration. Margaric acid was added to the base/neutral fraction in the form of the methyl ester.

Extraction efficiency was determined by spiking tapwater and sewage samples with the standard spiking mixture and preparing base/neutral and acid extracts as described earlier. The percent recovery was estimated by comparing the GC-FID integrals for each compound in these extracts with a known amount of the internal standards.

Quantification of sewage components was achieved by interfacing the GC-FID output to a Hewlett-Packard 79850 A LC terminal, which was used as an electronic integrator. Response factors relative to the internal standards 1, 2 - dibromo - 4, 5 dimethoxybenzene or margaric acid were determined for each spiking standard by analysing the spiking solution. Response factors for sewage components were assumed to be the same as those of the spiking standards belonging to the same class. For example, all alkylbenzenes were assumed to have the same response factor as the spiking standard n - butylbenzene. Similarly, cresol and xylenol were assumed to have the same response factor as phenol. The validity of these assumptions was tested by comparing the concentrations of the two internal standards to each other. Since both values are known, and one standard is used as a reference, then any bias in the system should be apparent. The quantitative results of analysing six sewage samples are given in Table 5. In Table 6, the composition of the organic fractions of the sewage samples is given in terms of the various classes of compounds present.

FINAL ANALYTICAL METHOD

Base/neutral fraction:

The pH of the sewage sample is adjusted to 10.0 and an 1800 mL aliquot transferred to a liquid/liquid extractor for downward displacement. Extraction with dichloromethane continues for 16 - 20 h, using a small stirring bar magnet on the bottom of the extractor. The solvent remaining in the bottom of the extractor is separated and added to the extract, which is then spiked at the 100 ppb level with the two internal standards 1, 2 - dibromo - 4, 5 - dimethoxybenzene and methyl margarate. The extract is concentrated to approximately 1 mL in a Kuderna-Danish apparatus in preparation for analysis.

Acid Fraction:

The pH of the base/neutral extracted sewage sample is adjusted to 2.0

and the extraction repeated using a mixture of dichloromethane and ether (4:1). The solvent is removed from the extractor and added to the extract. The spent sewage sample is then shaken with ether (2 x 75 mL) in a separatory funnel and the ether combined with the dichloromethane/ether extracts. After spiking with margaric acid and 1, 2 - dibromo - 4, 5 - dimethoxybenzene, the solution is concentrated to approximately 1 mL using a rotary evaporator.

Prior to analysing the sewage extracts, the retention times of the spiking standard compounds and their response factors relative to 1, 2 - dibromo - 4, 5 - dimethoxybenzene are entered into the memory of the integrator. The retention time and concentration (100 ppb) of the internal standard are also entered. The integrator then prints out directly the concentration in ppb of each sewage component identified in the search routine.

RESULTS

Compounds identified qualitatively in twelve sewage base/neutral fractions are summarized in Table 2 and the corresponding list of compounds in eleven acidic fractions is given in Table 3. It was apparent that some compounds - most notably paraffinic hydrocarbons and phenols - were distributed between both fractions. Many other minor components were detected. For some of these the mass spectra were not recognized while many others were present at concentrations below the detection limit for the mass spectrometer. One unidentified chlorinated compound, present in incoming sewage, had an apparent molecular weight of 218 and, judging by the ratio of the P to the P+2 peaks, contained one chlorine atom. The only other chlorinated compounds detected in Iona Island sewage samples were trichloropropene and two isomers of dichlorobenzene.

The acidic fractions contained large amounts of normal saturated C^5 to C^{24} fatty acids and the unsaturated palmitoleic, oleic and linoleic fatty acids. Also detected was a series of aromatic acids and another of dicarboxylic acids. Phthalic acid was often present in high concentrations and traces of cyclohexanecarboxylic acid were also commonly detected.

The data in Table 4, show that the base/neutral fractions were more heterogeneous than the acidic fractions in terms of the number of classes of organic compounds present. The only commonly occurring basic (nitrogenous) compounds were nicotine and caffeine. Phenols were not detected in all samples and had a marked tendency to partition between the base/neutral and the acid fractions. Low concentrations of a variety of alkylbenzenes with two, three or four carbon substituents were commonly present. Other frequently detected neutral components were a series of monoterpenes, the fecal sterols cholesterol and coprostanol, phthalate esters and benzyl alcohol.

The compounds listed in Table 1 were selected as spiking standards since all, except for phenylbutyric acid, were commonly detected in sewage and representative of the various compound classes known to be present. The four paraffinic hydrocarbons were available as a mixture and were used as such in preparing spiking solutions.

Recovery studies were conducted on mixtures of various chemicals spiked into tap water. Extractions performed in separatory funnels yielded recoveries in excess of 85%. However, the emulsification problems encountered in attempting to recover such compounds from spiked sewage samples negated that extraction procedure. Use of the liquid/liquid extractors for downward displacement avoided problems due to emulsification. Recoveries of 100 ± 5% from spiked tap water were achieved by this method after the introduction of a stirring bar magnet and a final ether extraction of the spent water sample in a separatory funnel.

Extracts of six sewage samples were quantified using the response factors measured for the compounds listed in Table 1. The results are presented in Table 5. Two of these samples were the acid fractions of weekly composites collected while fish exposure studies were in progress. The base/neutral fractions corresponding to these were contaminated by several abnormal compounds which, on the basis of their mass spectra, appeared to be organosilicones. These were therefore not analysed quantitatively.

The quantitative composition of the sewage samples is presented according to chemical class in Table 6. Clearly, the most plentiful components of sewage are the fatty acids. However, phthalic acid was also frequently observed at relatively high concentrations. Many aliphatic hydrocarbons were present and an attempt was made to sum the signals from compounds of this class. However, the concentrations shown in Table 6 are low because they do not take into account the "envelope" caused by the presence of unresolved complex mixtures of lubricating oil and various petroleum fractions. The presence of such unresolved matter was readily seen on the GC-FID charts and less easily observed by GC-MS.

The data in Table 5 and 6 indicate that the pair of samples collected on September 15 were more concentrated than those collected on August 19. There was poor correlation between the compositions and the relative concentrations of grab samples of raw sewage and treated sewage, collected on the same day. There were also marked differences between the composition of a grab sample of raw sewage and a composite sample of treated sewage collected over the preceeding 24 h period.

Only three chlorinated organics were detected in the study and these were all present in incoming sewage at levels near the detection limit of about 1 µg/l.

DISCUSSION

Based on analyses of the twelve samples collected in the period March to September 1982, the most frequently observed organic components of Iona

Island sewage were paraffin hydrocarbons, fatty acids, α - terpineol, phenylacetic acid, alkylbenzenes and fecal sterols. The sterols were probably present in all samples. However, on two occasions at least, the GC-MS data scan was terminated before these slow-eluting compounds had entered the detector.

Phenylacetic acid and the closely related benzoic, phenylpropionic, hydroxybenzoic, hydroxyphenylacetic and hydroxyphenylpropionic acids are likely to be among the end products of protein metabolism in humans and are chemically related to the amino acids tyrosine and phenylalanine. The presence of these compounds in primary treated domestic sewage plant effluents was noted in an earlier study (Pitt et al. 1975) using high-resolution ion-exchange chromatography.

A commonly detected component of the base/neutral fraction of Iona Island Sewage was α - terpineol. This substance is the major ingredient of pine oil and is widely used in household cleansing formulations such as "Mr. Clean" because of its germicidal properties. Other frequently observed components included the basic substances nicotine and caffeine, associated with tobacco and coffee grounds respectively, and salicylic acid, which is closely related to the common drug aspirin.

All the base/neutral fractions analysed showed the presence of a complex series of paraffin hydrocarbons indicated by sharp, distinct peaks situated on a baseline hump. The latter is caused by a complex of branched-chain hydrocarbons, such as are present in lubricating oils. Such mixtures are difficult to integrate. The values quoted in Table 6 are therefore low estimates of the total aliphatic hydrocarbons since they do not include an integral for the material in the baseline hump. The subject of hydrocarbon residues in domestic wastewaters discharged to the ocean has been recently studied in Southern California (Eganhouse and Kaplan 1982a, b) and in Seattle (Barrick 1982). In Southern California, sewage treatment plants were estimated to discharge 17.4 x 10^3 tonnes of hydrocarbons per year to the ocean, or about 4.9 g per capita per day. The Westpoint primary treatment plant in Seattle, which processes about 380 million L/d (100 million U.S. gallons), is estimated to discharge 475 metric tonnes/year of aliphatic hydrocarbons, or 2.6 gm per capita per day, to Puget Sound.

A series of C_2 to C_4 alkylbenzenes was also commonly detected in Iona Island treated sewage samples. Such compounds are used commercially as fuel components or in petroleum-based solvents and may therefore originate from both industrial and domestic use. The alkylbenzenes show prominent molecular ions and therefore appear to be present in higher concentration than is actually the case. Analysis by GC-FID shows that these compounds are present around the ppb level or below in Iona Island sewage. Similar compounds have been detected in domestic wastewater from Southern California treatment plants (Eganhouse and Kaplan 1982b).

A mixture of phenol with cresols and xylenols was often detectable in Iona Island sewage samples at relatively low levels. These compounds may derive from oil refinery wastewaters or from the application of wood preservatives such as cresote oil. Benzyl alcohol and phthalic acid were commonly seen, the latter in surprisingly high concentrations. This compound has also been identified in primary sewage in a previous study (Pitt et al. 1975).

Dicarboxylic acids were occasionally present in Iona Island sewage at fairly low concentrations and have been reported in one other study (Garrison et al. 1975). Naphthalene, methylnaphthalene and dimethylnaphthalene were also identified in Iona Island wastewater, in Southern California wastewaters (Eganhouse and Kaplan 1982b) and in Seattle Westpoint treatment plant wastewater (Barrick 1982). Sometimes grab samples contained unusual compounds. On one occasion dimethylbarbitone, related to the barbiturate drugs, was detected. Incidents involving a chemical being flushed down a sewer are more likely to be revealed in grab samples than in composite samples as a result of lower dilutions.

During the study period only three chlorinated organic compounds were observed in the extractable fraction. All of these were observed in raw sewage and thus are unrelated to the chlorine disinfection process. Other chlorinated organics may well be present below the level of detection in the mass spectrometer. Whether any of these compounds are significant to aquatic organisms will be determined by analysis of fish tissue samples accumulated during exposure studies. The results of such analyses may indicate selective bioaccumulation of chlorinated organic compounds to concentrations several orders of magnitude greater than the levels present in the wastewater. Thus, although the present data seem to indicate that disinfection with chlorine at Iona Island Sewage Treatment Plant does not result in the formation of deleterious chlorinated organics, the final verdict on this question must await the analytical results of fish tissue samples.

It was useful to have both internal standards present in each extract as this allowed a check on the concentrations of the unknowns in the sewage. Large discrepancies in the values would indicate possible instrumental problems and the results would not be acceptable. The final volume of the concentrated extracts was not critical since the concentration of the internal standards was known to be 100 ppb relative to the original water or sewage sample.

In the selected analytical method, the final extraction of the wastewater sample with ether in a separatory funnel was introduced to recover the last traces of hydrocarbons, which are ineffectively recovered using methylene chloride.

The concentrations of individual components of Iona Island sewage are anticipated to show wide variability because of the diluting effect of storm water during episodes of heavy rainfall. Results, such as those summarized in Table 6, need to be interpreted in the light of the amount of rainfall in the week prior to sampling. The first half of August 1982, was relatively wet, whereas the first half of September was dry.

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Table 1. Spiking standards for recovery studies

Base/Neutrals	RT min.
Phenol	3.21
n - Butylbenzene	4.34
Naphthalene	6.52
α - Terpineol	6.60
C_{12} - Hydrocarbon	6.68
C_{13} - Hydrocarbon	8.45
Nicotine	9.35
C_{14} - Hydrocarbon	10.19
C_{16} - Hydrocarbon	13.44
* 1, 2 - Dibromo-4,5 - dimethoxybenzene	14.94
Caffeine	17.18
Acids	
Benzoic	4.96
Phenylpropionic	8.09
Phenylbutyric	8.24
* Margaric	19.39

* Internal standard

Table 2. Compounds identified in base/neutral fractions of treated Iona Island sewage samples:

Tetrachloroethylene
Xylene
Methylcyclohexane
Xylene
Butylcellosolve
2 - Cyclohexen-1-ol
Cyclohex-2-enone
Trichloropropene
C_3 - Alkylbenzene
C_3 - Alkylbenzene
Phenol
C_3 - Alkylbenzene
C_{10} - Hydrocarbon
Dichlorobenzene
C_3 - Alkylbenzene

C_4 - Alkylbenzene
Dichlorobenzene
Benzyl alcohol
C_4 - Alkylbenzene

Methylnaphthalene
C_{13} - Hydrocarbon
Methylnaphthalene
Nicotine
C_{14} - Hydrocarbon
Dimethylnaphthalene
C_{15} - Hydrocarbon
C_{16} - Hydrocarbon
Diethyl phthalate
N,N - Dimethyllauramide
Tributylphosphate
C_{17} - Hydrocarbon
* Dibromodimethoxybenzene
Myristic acid
* d_{10} - Anthracene
C_{18} - Hydrocarbon
Caffeine
C_{19} - Hydrocarbon
unknown organochlorine (M^+ 218)
Phthalate ester

Table 2. (cont'd)

C_4 - Alkylbenzene

C_4 - Alkylbenzene

Cresol

C_4 - Alkylbenzene

Cresol

C_{11} - Hydrocarbon

Phenylethanol

Ethyl phenol

Xylenol

C_4 - Alkylbenzene

Camphor

Borneol

Menthol

Naphthalene

α - Terpineol

Hydroxyacetophenone

C_{12} - Hydrocarbon

2 - Phenoxyethanol

Carvone

Palmitic acid

* Methyl margarate

C_{21} - Hydrocarbon

C_{23} to C_{28} - Hydrocarbon

Coprostanol

Cholesterol

* Internal standard

Table 3. Compounds identified in acid fractions of Iona Island composite samples.

Valeric acid
Caproic acid
2 - Cyclohexen-1-one
2 - Furancarboxylic acid
Phenol
C_{10} - Hydrocarbon
Heptanoic acid

Succinic acid
Cyclohexanecarboxylic acid
Cresol
Benzoic acid
Caprylic acid
Ethyl phenol
Glutaric acid
Phenylacetic acid
Salicylic acid
C_{12} - Hydrocarbon
Xylenol
p - Toluic acid
Pelargonic acid

Lauric acid
Azelaic acid
C_{16} - Hydrocarbon
C_{17} - Hydrocarbon
* Dibromodimethoxybenzene
Myristic acid
*d_{10} - Anthracene
Pentadecanoic acid
Caffeine
C_{19} - Hydrocarbon
Palmitoleic acid
Palmitic acid
Phthalate ester
* Margaric acid
Linoleic acid
Oleic acid
Stearic acid
C_{21} - Hydrocarbon
Eicosenoic acid
Arachidic acid
Behenic acid

Table 3. (cont'd)

Phenylpropionic acid
3 - Methyladipic acid
Adipic acid
C_{13} - Hydrocarbon
Capric acid
Pimelic acid
Hydroxybenzoic acid
Cinnamic acid
Hydroxyphenylacetic acid
C_{14} - Hydrocarbon
Suberic acid
Dimethyl barbitone
Hydroxyphenylpropionic acid
Phthalic acid
C_{15} - Hydrocarbon
Phthalic acid isomer

Lignoceric acid

* Internal standard

Table 4. Frequency of occurrence in Iona Island sewage samples

PHENOLS	
Phenol	4
Cresol	6
Xylenol	4
Ethylphenol	1
ALKYLBENZENES	
Xylene	7
C_3 Alkylbenzenes	11
C_4 Alkylbenzenes	10
ALIPHATIC HYDROCARBONS	
C_{10} to C_{28} Hydrocarbons	12
AROMATIC ALCOHOLS	
Benzyl alcohol	6
2-Phenoxyethanol	8
MISCELLANEOUS	
Sterols	(9)
Naphthalenes	8
Tributyl phosphate	1
Cyclohexanes	5
Phthalate esters	9
MONOTERPENES	
Camphor	5
Borneol	8
Menthol	8
α-Terpineol	12
NITROGENOUS COMPOUNDS	
Nicotine	10
Caffeine	10
Dimethylbarbitone	1
N,N - Dimethyl lauramide	1
CHLORINATED COMPOUNDS	
Dichloro benzene	8
Trichloropropene	1
Unknown M^+218	2
TOTAL SAMPLES	12

AROMATIC ACIDS	
Benzoic acid	9
Phenylacetic acid	11
Phenylpropionic acid	9
Salicylic acid	9
Hydroxybenzoic acid	2
Hydroxyphenylacetic acid	1
Hydroxyphenylpropionic acid	1
p-Toluic acid	1
Cinnamic acid	4
DICARBOXYLIC ACIDS	
Succinic acid	5
Glutaric acid	3
Adipic acid	3
Pimelic acid	3
Suberic acid	3
Azelaic acid	5
3 methyladipic	3
Phthalic acid	5
Fatty acids	11
TOTAL SAMPLES	11

Table 5. Iona sewage test results. Results shown are ppb.

Compound	G.C. Ret. Time (min)	Pre chlor grab Aug. 19/82	Post chlor grab Aug. 19/82	Fish test comp. June 18-25	Fish test comp. June 25 - July 10	Pre chlor grab Sept. 15/82	Post chlor comp. Sept. 15/82
Xylene	2.15	12	31			110	44
Xylene	2.35	7	20			59	N.Q.
Trimethyl Benzene	3.47	2	8			NIL	123
Dichloro Benzene	3.72	2	NIL			NIL	N.Q.
Benzyl Alcohol	4.00	41	64			26	58
P - Cresol	4.69	7	29			NIL	N.Q.
Benzoic Acid	4.99	59	25	59	11	96	194
C8:0 Fatty Acid	5.41	79	49	110	27	174	446
Menthol	6.32	5	NIL	N.T	N.T	29	+r
Phenyl Acetic Acid	6.41	88	61	33	32	140	344
α - Terpineol	6.68	21	49	N.T.	N.T.	94	191
Phenyl Propionic Acid	8.12	29	20	7	TRACE	48	79
Methyl Naphthalene	8.44	5	NIL	N.T.	N.T.	10	NIL
Hydrocarbon C13	8.50	14	7	N.T.	N.T.	29	63
C10:0 Fatty Acid	8.93	70	56	126	21	243	198
Nicotine	9.38	5	12	N.T.	N.T.	19	29
Hydrocarbon	9.39	7	6	5	NIL	N.Q.	N.Q.
Hydrocarbon C14	10.22	8	6	2	NIL	22	58
Hydrocarbon	10.81	10	9	NIL	NIL	N.Q.	N.Q.
Phthalic Acid	11.35	562	739	2487	1979	55	2258
Hydrocarbon C15	11.89	10	13	3	4	31	59
C12:0 Fatty Acid	12.32	179	87	197	49	210	354
Phthalate	13.23	15	22	N.T.	N.T.	NIL.	NIL
Hydrocarbon C16	13.45	6	12	N.T.	N.T.	9	61
Hydrocarbon	13.46	11	12	6	NIL	N.Q.	N.Q.
C13:0 Fatty Acid	13.66	31	25	13	NIL	NIL	90
Hydrocarbon	14.51	8	7	NIL	NIL	N.Q.	N.Q.
C14:0 Fatty Acid	15.28	325	107	367	84	307.	543.
C15:0 Fatty Acid	16.22	36	18	19	NIL	19	36
C15:0 Fatty Acid	16.34	38	15	36	NIL	N.Q.	N.Q.
C15:0 Fatty Acid	16.38	23	24	6	NIL	20	39
C15:0 Fatty Acid	16.75	80	31	82	29	57	89
Hydrocarbon	16.84	2	NIL	N.T.	N.T.	NIL	NIL
Hydrocarbon	16.97	2	NIL	N.T.	N.T.	NIL	NIL
Caffeine	17.21	2	2	N.T.	N.T.	14	41
C16:0 Fatty Acid	17.59	51	23	16	NIL	N.Q.	N.Q.
C16:1 Fatty Acid	17.84	188	15	145	NIL	110	76
C16:0 Fatty Acid	18.25	1457	530	1800	519	1214	973

Table 5 (cont'd)

Compound	G.C. Ret. Time (min)	Pre chlor grab Aug. 19/82	Post chlor grab Aug. 19/82	Fish test comp. June 18-25	Fish test comp. June 25 - July 10	Pre chlor grab Sept. 15/82	Post chlor comp. Sept 15/82
Phthalate	18.50	15	54	N.T.	N.T	52	37
C17:0 Fatty Acid	19.02	32	17	19	NIL	11	20
C18:2 Fatty Acid	20.23	72	NIL	123	163	48	59
C18:1 Fatty Acid	20.36	342	23	651	69	66	100
C18:1 Fatty Acid	20.41	103	77	233	196	60	95
C18:0 Fatty Acid	20.70	584	223	528	172	352	628
C20:1 Fatty Acid	21.02	22	NIL	2	178	NIL	59
C22:1 Fatty Acid	22.61	22	24	29	NIL	12	19
C22:0 Fatty Acid	22.90	22	15	17	NIL	19	41
Di-octyl Phthalate	23.11	6	NIL	49	NIL	N.Q.	N.Q.
Succinic Acid	3.98					33	NIL
Dimethyl Phenol	5.40					174	N.Q.
Salicylic Acid	6.75					46	162
Carvone	8.50					16	10
Naphthalene	7.30					15	NIL
Dimethyl Naphthalene	11.30					11	NIL

NQ = Not quantified
NT = Not tested

Table 6. Composition of Iona Sewage Samples by Chemical Class *.

Chemical Class	Pre chlor grab Aug. 18/82	Post chlor grab Aug. 19/82	Fish test comp. June 18-25	Fish test comp. June 25 - July 10	Pre chlor grab Sept. 15/82	Post chlor comp. Sept. 15/82
Benzene hydrocarbons	21	59	NT	NT	169	167+
Aliphatic hydrocarbons	81	72	NT	NT	91+	241+
Naphthalenes	5	NIL	NT	NT	36	NIL
Phenols	7	29	NT	NT	174	NQ
Monoterpenes	26	49	NT	NT	139	201
Dichlorobenzene	2	NIL	NT	NT	NIL	NQ
Benzyl alcohol	41	64	NT	NT	26	58
Phthalate esters	36	76	49+	NIL+	52+	37+
Fatty acids	3756	1359	4519	1507	2922	3775
Aromatic acids	176	106	99	43	330	779
Nicotine	5	12	NT	NT	19	29
Caffeine	2	2	NT	NT	14	41
Phthalic acid	562	739	2487	1979	55	2258
Succinic acid	NQ	NQ	NQ	NQ	33	NIL

* in μg/L
NT = not tested
NQ = not quantified