

Assessment of treatment options for removal of chlorthal from groundwater

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Contents

Appendices

List of Tables

List of Figures

Summary

i Reasons

South Staffordshire Water have identified an issue with the compound chlorthal in some ground water supplies. Chlorthal is a persistent metabolite of the herbicide chlorthal-dimethyl (dimethyl tetrachloroterephthalate) which is no longer approved for use in the EU. South Staffordshire Water therefore requires an assessment of a range of different treatment options for removal of chlorthal from groundwater.

ii Objectives

To use laboratory and pilot scale tests to assess the potential for a defined range of treatment options to remove or degrade chlorthal in groundwater. The processes to be evaluated were treatment by granular activated carbon (GAC), Ultraviolet (UV) light irradiation, UV with hydrogen peroxide, ozonation, and ozonation with hydrogen peroxide.

iii Benefits

The study will aid strategic planning for future treatment options at Slade Heath and other groundwater sites where chlorthal is present.

iv Conclusions

GAC is capable of removing chlorthal, however it would have a small capacity to breakthrough at the regulatory limit of 0.1 µg/l, requiring frequent regeneration. For this reason, GAC is not considered a practicable option for this application.

Addition of powdered activated carbon (PAC) could be considered together with a suitable PAC removal process (e.g. ultrafiltration membrane), however the implications for operating costs are likely to make use of PAC inappropriate.

UV irradiation is only capable of significant removal of chlorthal by degradation when used in combination with a relatively high dose of hydrogen peroxide. The estimated energy requirement to achieve 90% removal at Slade Heath would be very significant (~4 kWh/m^3). For this reason, UV alone or UV as an advanced oxidation process is not considered to be an economic option for this application.

Ozone alone (aqueous ozone) or ozone in combination with hydrogen peroxide, as an advanced oxidation process, did not achieve useful degradation of chlorthal.

The ion-exchange process at the Pipe Hill site, used for nitrate removal, is the most promising process for application at Slade Heath. Even for a site with a relatively small output, it is clear that implementing ion-exchange would result in a significant increase in operating costs, but for a site where nitrate removal is not required the process could be optimised for chlorthal removal.

v Recommendations

Pilot scale testing of Purolite A520-E and alternative resins at a range of contact times would provide confirmation of suitability for Slade Heath, and allow some optimisation of contact time to inform full scale process design.

vi Résumé of Contents

This report describes a series of laboratory and pilot scale tests carried out at WRc to evaluate a series of treatment options for removal of the compound chlorthal, which has been found to contaminate groundwater at some sites within South Staffordshire Water.

1. Introduction

South Staffordshire Water have identified an issue with the compound chlorthal in some ground water supplies. Chlorthal is a persistent metabolite of the herbicide chlorthal-dimethyl (no longer approved for use in the EU). Monitoring by South Staffordshire Water indicates that there is a persistent background concentration of approximately 1.8 to 2.0 µg/l chlorthal at one site (Slade Heath, 4 Ml/d) and somewhat lower at a second site (Pipe Hill, 12 Ml/d).

The metabolite has been found to be effectively removed at Pipe Hill by the existing ion-exchange process for nitrate removal, however if this were implemented at the Slade Heath site, which has only an iron and manganese removal process and disinfection, there would be a significant impact on operating costs.

South Staffordshire Water require an assessment of a range of different treatment options for removal of chlorthal from groundwater, to include treatment by granular activated carbon (GAC), Ultraviolet (UV) light irradiation, UV with hydrogen peroxide, ozonation, and ozonation with hydrogen peroxide.

A Rapid Column Test (RCT) was used to assess the performance of GAC for the Slade Hill source, simulating full scale conditions with a small scale, continuous flow test. By reduction of the GAC particle size, a period of 2 years operation can be simulated within 3 weeks.

A pilot scale UV treatment plant was used to assess the effectiveness of UV alone and UV in combination with hydrogen peroxide for both water sources.

Batch tests were used to assess the effectiveness of ozonation alone and in combination with hydrogen peroxide for both water sources.

2. Chlorthal

2.1 Formation of chlorthal

Chlorthal (2,3,5,6-tetrachloroterephthalic acid) is one of the two main environmental degradation products of chlorthal-dimethyl (dimethyl tetrachloroterephthalate), a pre-emergence herbicide which is no longer approved for use in the EU.

Chlorthal-dimethyl has low solubility in water, and is moderately persistent in soil with 90% degradation within periods of up to 328 days in field studies (University of Hertfordshire).

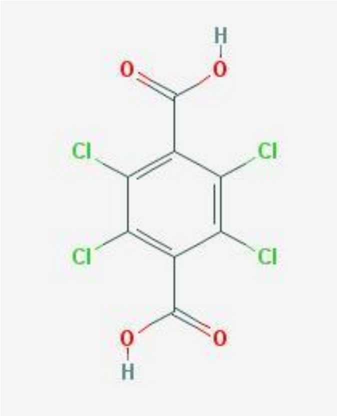
The greater fraction (~95%) of degradation products is expected to be chlorthal which is also known as TPA; the remainder is expected to be largely monomethyl tetrachloroterephthalic acid (MTP).

Within the EU, chlorthal is regarded as a ‘relevant metabolite’ with respect to the potential to contaminate groundwater sources, which means that as for the parent compound, the concentration of chlorthal should not exceed 0.1 µg/l in drinking water.

2.2 Structure and properties

Chlorthal has the chemical formula $C_8H_2Cl_4O_4$. The chemical structure is shown in Figure 2.1.

Figure 2.1 Structure of chlorthal



Chlorthal is highly soluble in water (~5.8g/l at 20 °C) and is regarded as persistent in soil, with 90% degradation within 333 to 4270 days in laboratory studies, however no information is available on the persistence of chlorthal in water.

3. Removal of chlorthal by GAC

3.1 Test design

Small scale rapid column tests (RCTs), with continuous flow, can provide an estimate of GAC bed life at full scale much more quickly than pilot plant trials and more reliably than batch isotherm tests.

RCTs use GAC ground to a smaller particle size than used in full-scale plant, to allow a faster rate of adsorption. The rate of adsorption is inversely proportional to the square of the particle size, as indicated in the following equation:

$$\mathrm{EBCT}_{\mathrm{RCT}} / \mathrm{EBCT}_{\mathrm{FS}} = (\mathrm{D}_{\mathrm{RCT}}/\mathrm{D}_{\mathrm{FS}})^2$$

where: EBCT = empty bed contact time

D = GAC particle diameter

RCT = rapid column test

FS = full scale

A sample of virgin Norit 12x40 grade GAC (mean size 0.98 mm) was used for the RCT, ground and sieved to 150-180 µm.

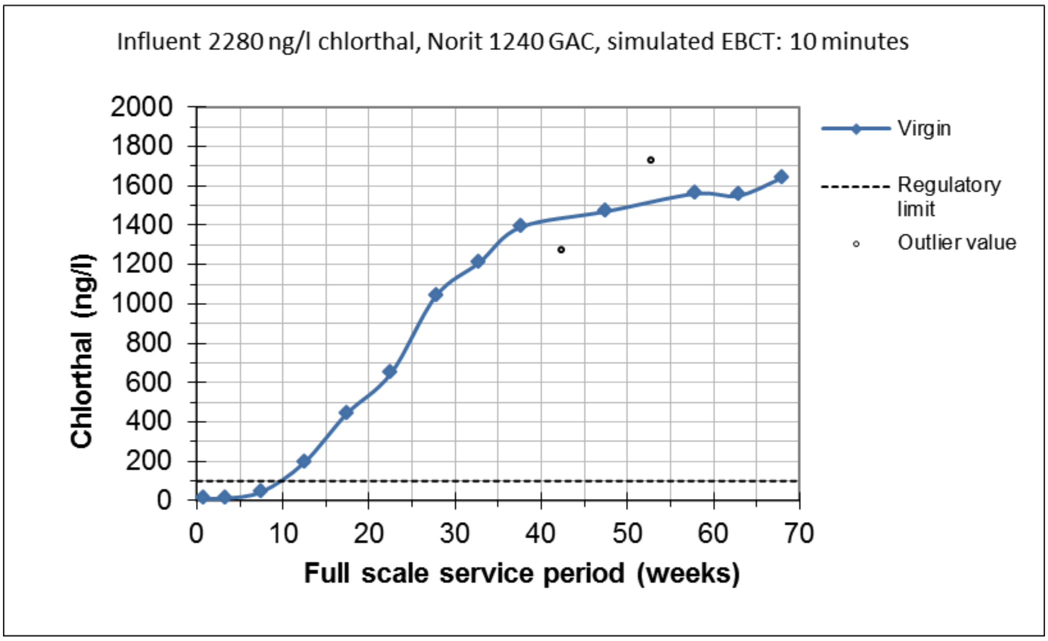
The feed water for the tests was obtained as untreated water from Slade Heath borehole, delivered to WRc in 10 litre containers and stored in the dark at 5 °C until required. No spiking was required as the water was expected to have sufficient background concentration of chlorthal for the adsorption test. The flow rate through the column was adjusted to simulate a relatively short empty bed contact time (EBCT) of 10 minutes at full scale.

The test was operated for 15 days, simulating operation for 1 year 15 weeks at full scale. Samples of feed water were taken at weekly intervals. Samples of treated water from the RCT column were collected daily. All of the samples were analysed for chlorthal by Eurofins Environmental Testing UK Ltd.

3.2 RCT results

The results are shown in Figure 3.1 as a breakthrough curve for chlorthal plotted against the equivalent operational period at full scale.

Figure 3.1 Breakthrough of chlorthal (Norit 1240 GAC)



The concentration of chlorthal exceeded the regulatory limit of 0.1 µg/l after an equivalent of approximately 10 weeks operation at full scale, indicating relatively poor removal by GAC. This result is consistent with a compound which has high aqueous solubility.

4. Degradation of chlorthal by ultraviolet light

4.1 Test design

Tests were carried out to establish whether water contaminated with chlorthal could be treated effectively by photolysis, using ultraviolet (UV) light.

4.1.1 UV reactor

Tests were carried out with a pilot scale rig, utilising a Xylem Wedeco UV reactor with a cylindrical reactor vessel containing a single low pressure UV-C lamp. The UV lamp had a nominal power requirement of 80 Watts, and was enclosed in a quartz sleeve. The reactor was equipped with a UV intensity sensor, selective to 254 nm, and the measurement from this was displayed on the system control unit as W/m^2.

4.1.2 Feed water and flow rate

Tests were initially carried out with a bulk sample of water from Slade Heath, collected and delivered on 09/09/2014. This water had been obtained downstream of the iron and manganese removal stage. No spiking was carried out, based on the assumption that chlorthal was expected to be present at sufficient background concentration to assess treatment efficacy.

Subsequently, further selective tests were carried out with a bulk sample of water from Pipe Hill borehole, collected and delivered on 24/09/2014. This water had been obtained upstream of the nitrate removal (ion-exchange) process at site (i.e. raw water). No chlorthal spiking was carried out.

4.1.3 Test conditions

The UV system was operated with water running to waste, until the measured UV intensity stabilised, indicating that the UV lamp had reached normal operating condition. Continuous flow tests were then carried out at 3 different flow rates (100, 200 and 300 l/h) to provide different degrees of UV exposure.

Following each change of flow rate, a period equivalent to 5 hydraulic residence times was allowed before collecting 2 consecutive samples of treated water for analysis. In addition, samples of the rig feed water were taken to enable calculation of chlorthal removal.

Once tests with UV alone were completed, further tests were carried out with the addition of hydrogen peroxide, as an advanced oxidation process (AOP). Exposure of hydrogen peroxide to UV produces hydroxyl radicals, which act as strong chemical oxidants. The dose of hydrogen peroxide needs to be sufficient to provide an excess of hydroxyl radicals, after the hydroxyl scavenging effects of dissolved minerals which constitute alkalinity. Excessive hydrogen peroxide can ultimately reduce the efficiency of the oxidative process. For these tests, hydrogen peroxide was applied at dose rates of 2.5, 7.5 and 15 mgH_2O_2/l, covering a typical range for this process.

Water quality will influence the efficiency of a UV system. The UV transmittance (UVT) of the feed water was measured with a spectrophotometer at 254 nm. The UVT for Slade Heath was high (99.9%), however the UVT for Pipe Hill was markedly lower (70.8%). Further information on the water quality for both sources tested is shown in Appendix A.

4.2 Results of UV treatment tests

The efficiency of treatment using UV or UV with hydrogen peroxide in achieving photolysis of chlorthal is measured by reference to the calculated energy requirement to result in a 1 log (90%) reduction in the contaminant concentration. This measure is known as the Electrical Energy Used per Order of Magnitude Reduction (EEO), calculated as shown in Equation 1.

Equation 1: Calculation of Electrical Energy Used per Order of Magnitude Reduction

$$EEO = \frac{\mathrm{P \; x \; T}}{\mathrm{60 \; x \; V \; x} \log \frac{Co}{Ce}}$$

Where

EEO = Electrical Energy Used per Order of Magnitude Reduction (kWh/m^3)
P = Power (kWh)
T = Irradiation time (minutes)
60 = minutes per hour
V = Reactor volume (m^3)
C_o = Initial concentration of contaminant
C_e = Concentration of contaminant in treated water

A summary of the results for the Slade Heath source is shown in Table 4.1.

Table 4.1 Results of UV treatment tests (Slade Heath source)

Influent concentration of chlorthal (µg/l)*	Flow rate (l/h)	UV intensity (mW/cm^2)	Hydrogen peroxide dose (mgH_2O_2/l)	Chlorthal removal (%)*	EEO (kWh/m^3)**
0.947	100	100.3	-	7.8	22.8
	200	100.6	-	7.2	12.4
	300	100.8	-	0.5	128.9
	100	98.9	2.5	7.3	24.2
	200	98.6	2.5	12.5	6.9
	300	98.4	2.5	0.4	145.1
	100	96.7	7.5	20.3	8.1
	200	96.1	7.5	9.3	9.4
	300	96.2	7.5	4.6	12.9
	100	97.3	15.0	24.8	6.5
	200	92.9	15.0	20.8	3.9
	300	92.9	15.0	13.2	4.3

*Based on mean measured concentration of chlorthal for 2 samples.

**Electrical energy used per order of magnitude reduction, based on UV power requirement of 80 Watts.

It can be seen that removal of chlorthal by UV alone at Slade Heath would be highly energy intensive, requiring 12.4 kWh/m^3 to achieve 90% removal, at best. A combination of hydrogen peroxide with UV achieves greater efficiency; at 2.5 mgH_2O_2/l the energy requirement for 90% removal remains high with an EEO of 6.9 kWh/m^3 at least. A combination of UV and 15.0 mgH_2O_2 achieved the best energy efficiency, with an EEO of 3.9 – 6.5 kWh/m^3.

The results of the tests with water from the Pipe Hill source are shown in Table 4.2.

Table 4.2 Results of UV treatment tests (Pipe Hill source)

Influent concentration of chlorthal (µg/l)*	Flow rate (l/h)	UV intensity (mW/cm^2)	Hydrogen peroxide dose (mgH_2O_2/l)	Chlorthal removal (%)*	EEO (kWh/m^3)**
0.218	100	91.1	-	1.2	159.3
	100	89.0	7.5	6.2	28.7
	100	88.9	15.0	10.1	17.3

*Based on mean measured concentration of chlorthal for 2 samples.

**Electrical energy used per order of magnitude reduction, based on UV power requirement of 80 Watts.

The concentration of chlorthal in the water from the Pipe Hill source (218 ng/l) was markedly lower than that for the Slade Heath source (947 ng/l). The relatively low UVT for the water from Pipe Hill, compared to Slade Heath, is the most likely reason for the greater EEO requirement.

5. Degradation of chlorthal by ozone

5.1 Test design

Batch tests were carried out to establish whether water contaminated with chlorthal could be treated effectively by oxidation, either directly using aqueous ozone or indirectly by the perozone process.

All tests were carried out in duplicate and the mean percentage removal of chlorthal calculated by comparison with the measured concentration in the untreated water.

5.1.1 Ozonation

A Xylem Wedeco pilot scale ozone generator was used to produce ozone gas from pure oxygen, and the gas bubbled through 1.5 litre batches of water from the Slade Heath and Pipe Hill sites.

Ozone production was set at a constant rate and concentration by setting the gas flow and electrical power applied. The exact dose could not be calculated because the gas to liquid transfer efficiency is unknown. The dose was estimated by ozonation of distilled water for the same time period as for the test water, and immediate measurement of the resultant ozone residual.

Ozonation periods of 10 and 15 seconds were found appropriate, resulting in an ozone residual of 0.2 and 0.4 mg/l respectively, after 10 minutes contact time following ozonation. For both ozonation periods, batches with post-ozonation contact times of 30 seconds, 5 minutes and 10 minutes were tested. At the end of the contact period, a sub-sample was immediately added to a sample bottle containing sodium thiosulphate, to quench the residual ozone. A further sub-sample was used to measure the residual ozone concentration.

5.1.2 Perozonation

Application of hydrogen peroxide in combination with ozone, accelerates decomposition of ozone and therefore generates an increased concentration of hydroxyl radicals, which act as strong chemical oxidants. The ratio of hydrogen peroxide to ozone is typically in the range 0.5:1 and 1:1 by mass, although beneficial use of a greater proportion of hydrogen peroxide (e.g. 4:1 ratio) may be used specifically for degradation of organic compounds rather than combining with a disinfectant function. As with ozone alone, pH and bicarbonate alkalinity play an important role in peroxone effectiveness due to competition for hydroxyl radicals at high alkalinity and/or high pH. Excessive peroxide can also limit the formation of the hydroxyl radical and reduce the effectiveness of peroxone.

In these tests, hydrogen peroxide was added to each 1.5 litre batch of water immediately before starting ozonation. Different ratios of hydrogen peroxide:ozone were tested, ranging from approximately 0.5:1 to 2.5:1 for the first series of tests with water from Slade Heath, and approximately 4:1 for the second series of tests with water from both Slade Heath and Pipe Hill.

5.2 Results of ozonation tests

5.2.1 Aqueous ozone

Tests with aqueous ozone were only carried out for the first series of tests with water from the Slade Heath site. The test results are summarised in Table 5.1.

Table 5.1 Results of batch tests with aqueous ozone (Slade Heath)

Ozonation period (seconds)	Post ozonation contact time (minutes)	Ozone residual (mgO_3/l)		Chlorthal concentration (µg/l)		
		Test 1	Test 2	Test 1	Test 2	Mean
0 (Control)	10	-	-	0.695	0.626	0.661
10	0.5	0.35	0.33	0.742	0.654	0.698
10	5	0.15	0.18	0.752	0.691	0.722
10	10	0.22	0.18	0.821	0.755	0.788
15	0.5	0.48	0.47	0.780	0.693	0.737
15	5	0.47	0.47	0.707	0.760	0.734
15	10	0.35	0.42	0.680	0.748	0.714

There was an unexpectedly high degree of variation in the measured concentration of chlorthal for duplicate tests, including a variation of 69 ng/l between the duplicate samples of untreated water (control).

The mean values for chlorthal in duplicated tests show no removal compared to the starting concentration; indeed they indicate an *increase* in chlorthal concentration. As the only known route to generating chlorthal would be degradation of chlorthal-dimethyl, samples of the untreated water from Slade Heath were analysed in duplicate for chlorthal-dimethyl, however the results showed that this compound was below the limit of detection (2 ng/l), and therefore it must be assumed that the apparent increase in chlorthal is due to unknown analytical issues, probably causing a decrease in analytical repeatability.

5.2.2 Perozone

Tests with perozone (ozone in combination with hydrogen peroxide) were carried out for the first series of tests with water from the Slade Heath site. Further tests were carried out at a greater ratio of hydrogen peroxide to ozone in a second series of tests with water from both sites. The test results are summarised in Table 5.2 and Table 5.3.

Table 5.2 Results of batch tests with perozone (Series 1)

Hydrogen peroxide dose (mgH_2O_2/l)	Ozonation period (seconds)	Approximate ratio of $H_2O_2 : O_3$	Post ozonation contact time (minutes)	Ozone residual (mgO_3/l)		Chlorthal concentration (µg/l)		
				Test 1	Test 2	Test 1	Test 2	Mean
0	(Control)	-	10	-	-	0.695	0.626	0.661
0.5	10	1.3 : 1	0.5	0.08	0.23	0.726	0.694	0.710
0.5	10	1.3 : 1	5	0.07	0.04	0.712	0.736	0.724
1	10	2.5 : 1	0.5	0.16	0.15	0.738	0.724	0.731
1	10	2.5 : 1	5	0.06	0.05	0.690	0.792	0.713
1	15	1.7 : 1	5	0.04	0.04	0.679	0.754	0.717

Note 1 Water temperature 14.5 – 16.9 °C

Table 5.3 Results of batch tests with perozone (Series 2)

Source	Hydrogen peroxide dose (mgH_2O_2/l)	Ozonation period (seconds)	Post ozonation contact time (minutes)	Ozone residual (mgO_3/l)		Chlorthal concentration (µg/l)		
				Test 1	Test 2	Test 1	Test 2	Mean
Slade Heath	-	(Control)	10	-	-	0.666	0.666	0.666
	2.4	15	10	0.05	0.05	0.666	0.601	0.634
Pipe Hill	-	(Control)	-	-	-	0.202	0.201	0.202
	2.4	15	10	0.10	0.08	0.213	0.219	0.216

Note 1 For all tests in Series 2, the approximate ratio of $H_2O_2 : O_3$ was 4:1

Note 2 Water temperature 10.0 °C

As for the tests with aqueous ozone, the analytical results for chlorthal showed an apparent *increase* in concentration following treatment. This appears to be an artefact of analysis but it must be assumed that perozone was ineffective in degrading chlorthal.

The second series of perozone tests showed improved repeatability for the duplicate untreated water samples. The greater ratio of hydrogen peroxide to ozone, compared to the first series of tests, achieved approximately 5% removal of chlorthal for the Slade Heath sample and no removal (slight increase) for the Pipe Hill sample.

The lower ozone residual for the perozone tests compared to tests with aqueous ozone is to be expected, due to the hydrogen peroxide causing an increased rate of ozone decomposition by formation of hydroxyl radicals.

6. Discussion

6.1 GAC adsorption

Unlike chlorthal-dimethyl, chlorthal has high aqueous solubility, which hinders effective removal by GAC. The RCT clearly showed that whilst GAC is capable of removing chlorthal, it has very limited capacity. The test carried out with Norit 12x40 GAC at a relatively short contact time (simulated EBCT 10 minutes), showed that GAC would need to be regenerated every 10 weeks for the concentration of chlorthal at Slade Heath, in order to comply with the regulatory limit of 0.1 µg/l.

A longer EBCT would reduce regeneration frequency. Alternative types of GAC may also offer some further benefit.

Addition of powdered activated carbon (PAC) could be considered, however an additional downstream process would be needed to remove the PAC after a suitable contact time. At these groundwater sites, the only likely feasible option for PAC removal would be a suitable ultrafiltration membrane process. The implications for operating costs are likely to make use of PAC inappropriate.

6.2 Ultraviolet irradiation

The pilot scale tests have shown that UV irradiation, in combination with hydrogen peroxide, can achieve useful degradation of chlorthal. Small scale UV reactors such as the one used in these tests tend to over-estimate the energy requirements, compared to a full scale system, however it is clear that the cost of the energy to achieve 90% removal of chlorthal at Slade Heath would increase the cost of water production unacceptably (£0.39/m^3 assuming £0.10/kWh).

6.3 Ozonation

Due to the poor repeatability of the duplicate tests and analyses, there is some degree of uncertainty in drawing conclusions from the batch tests with ozone and perozone. Despite this, neither ozonation nor perozonation appear to achieve useful degradation of chlorthal.

6.4 Further treatment options

Based on the test results from this study, a combination of advanced oxidation processes, comprising UV with hydrogen peroxide, together with ozone is unlikely to offer a significant benefit over UV with hydrogen peroxide.

The nitrate removal process (ion-exchange) at Pipe Hill is understood to be effective in removing chlorthal at this site. The concentration of chlorthal is lower, relative to Slade Heath, but in principle this process could be the best option for Slade Heath. The ion-exchange system installed at Pipe Hill uses Purolite A520-E, a macroporous strong base anion resin (styrene-divinylbenzene). Further investigation by pilot scale column tests could be useful in confirming that the resin would provide satisfactory performance for the greater concentration of chlorthal which is found at Slade Heath, as well as exploring the effects of contact time. Since nitrate removal is presumably not necessary at Slade Heath, the process could be optimised for removal of chlorthal. It would also be useful to confirm that the resin capacity for chlorthal is recovered satisfactorily following regeneration. It should also be noted that the regenerant waste will contain a relatively high concentration of chlorthal, possibly influencing the waste disposal route.

7. Conclusions

7.1 GAC

Whilst GAC is capable of removing chlorthal, it would have a small capacity to breakthrough at the regulatory limit of 0.1 µg/l, requiring frequent regeneration. For this reason, GAC is not considered a practicable option for this application.

Although not considered within the scope of this report, use of a high activity PAC in combination with ultrafiltration could be an alternative option if sufficient PAC contact time could be provided. However, operating costs would be expected to be very significant and an additional process downstream would be required, such as membrane ultrafiltration to remove the PAC.

7.2 Ultraviolet light irradiation

UV irradiation is only capable of significant removal of chlorthal by degradation when used in combination with a relatively high dose of hydrogen peroxide. The estimated energy requirement to achieve 90% removal at Slade Heath would be very significant (~4 kWh/m^3). For this reason, UV alone or UV as an advanced oxidation process is not considered to be an economic option for this application.

7.3 Ozonation

Ozone alone or ozone in combination with hydrogen peroxide, as an advanced oxidation process, did not achieve useful degradation of chlorthal.

7.4 Ion-Exchange

The ion-exchange process at the Pipe Hill site, used for nitrate removal, is the most promising process for application at Slade Heath. Even for a site with a relatively small output, it is clear that implementing ion-exchange would result in a significant increase in operating costs, but for a site where nitrate removal is not required the process could be optimised for chlorthal removal.

8. Recommendations

8.1 Ion-exchange

Pilot scale testing of Purolite A520-E and alternative resins at a range of contact times would provide confirmation of suitability for Slade Heath, and allow some optimisation of contact time to inform full scale process design.

Appendix A Water Quality

Table A.1 Water quality for samples used in laboratory and pilot scale tests

Source	Date obtained from site	Removal test	Parameter	Value
Slade Heath	18/07/2014	GAC (RCT)	TOC	1.1 mg/l
			Chlorthal	2.25 µg/l
	09/09/2014	UV, Ozone, AOP's	UVT	99.9%
			Turbidity	0.14 NTU
			Total alkalinity	195 $mgCaCO_3/l$
			Total hardness	368 $mgCaCO_3/l$
			pH	7.90
			Chlorthal	0.95 µg/l
Pipe Hill	24/09/2014	UV, Ozone, AOP's	UVT	70.8%
			Turbidity	Not measured
			Total alkalinity	182 $mgCaCO_3/l$
			Total hardness	335 $mgCaCO_3/l$
			pH	7.70
			Chlorthal	0.22 µg/l