

Water quality assessment for indirect potable reuse: a new methodology for controlling trace organic compounds at the West Basin Water Recycling Plant (California, USA)

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Abstract The West Basin Water Recycling plant (California: USA) was built to increase the region's water resource availability. The plant influent is produced at Los Angeles Hyperion wastewater treatment plant and is treated through two parallel treatment processes depending on the end use : (1) Title 22 water for industrial and urban use, and (2) barrier treatment for groundwater recharge. A new methodology was applied to monitor the fate of base neutral compounds in the water barrier treatment train. The methodology included large sample volumes coupled with integrated chromatographic analysis (ICA). Data indicated a 25% increase in concentration of base neutral compounds after RO pretreatment, followed by a 70% removal efficiency after RO. The increase in concentration after RO pretreatment appears to be linked to the use of lime clarification.

Keywords Indirect potable reuse; reverse osmosis; trace organics; lime clarification , base neutral compounds

Introduction

The presence of organic micropollutants in water reuse effluents has been of growing concern among public health officials especially with the development of indirect potable reuse projects and the fear of contaminating existing natural resources. The monitoring strategy usually adopted for U.S. water reuse studies has been to follow drinking water standards, including priority pollutant identification (so-called "target compounds"). However, many other "non-target" compounds are detected while analyzing for such compounds but are ignored.

In the 1970s, the Whittier Narrows project performed a thorough water quality study. The only fraction for which the study could not guaranty high removal was for the organic fraction, due to the difficulty in identifying more than a few percentages of all organic chemicals present (McEwen *et al.*, 1996). From the the Montebello Forebay groundwater recharge project only less than detectable amounts of 2,4-D, 2,3,5-TP and Methoxyclor were reported (U.S. EPA, 1992). However this did not mean that no other organic compounds were observed, but just that the listed target compounds were not identified.

Finally, among the more recent studies to have focused on organic contamination in reuse effluents are the San Diego risk assessment study (Western Consortium for Public Health, 1996) and the Lake Arrowhead wastewater reuse pilot plant (Levine, 1999). Both projects used large volume water samples to analyze for organic micropollutants in water reuse effluents, enabling to lower the detection limit of more standard liquid-liquid extraction methods thus providing additional information on this organic fraction. More importantly, the Lake Arrowhead data were analyzed using Integrated Chromatographic Analysis (ICA), which uses all of the chromatographic information, expanding data analysis beyond target compounds (Levine, 1999). ICA has shown to be successful in high-

lighting changes in base neutral composition after ozonation, biological activated carbon filtration and membrane processes (Levine *et al.*, 2000).

The purpose of this paper is to monitor, at the West Basin Water Recycling plant (California, USA), for the fate of base neutral organic across one of two treatment trains used to replenish the local aquifer.

Materials and methods

West Basin Water Recycling Plant

The West Basin Water Recycling Plant is located South of Los Angeles. It treats water from the Los Angeles Hyperion wastewater treatment plant through two parallel treatment trains : (1) a Title 22 train for industrial and irrigation use and (2) a barrier plant for ground water recharge to protect the aquifer against sea water intrusion (Figure 1). The second train is formed from two parallel processes with a total capacity of 7.5 MGD. The present paper will focus on the first set of processes which includes decarbonation of the plant influent followed by lime clarification, filtration and reverse osmosis (RO). The second train is a two step process with a microfiltration units followed by reverse osmosis and disinfection. Background data on the combined water barrier effluent quality will be provided as annual averages for both general parameters and specific organic micropollutant data as required by the Los Angeles Water Quality Control Board.

Base neutral analysis

Large sample volumes were extracted with dichloromethane (DCM) with a continuous liquid extractor (CLLE) (Baker *et al.*, 1987; Levine, 2000). The average extracted volume was 17 L of water. Two sets of samples were analyzed. The first were drawn in November 1997 and the second in May 1998. For conservation purposes, the samples were stored at 4°C, in the dark, after the pH of each sample had been adjusted to 3.0 with the addition of concentrated phosphoric acid. Before CLLE was performed sodium sulfate was added to the sample at a concentration of 50 ppm. The CLLE extraction rate was adjusted to 3 L/hr at a 10:1, water: solvent ratio. The overall estimated average extraction efficiency is 45% (Levine *et al.*, 2000).

For quality assurance and quality control purposes, surrogate standards were injected in the sample prior to extraction. After extraction, the samples were dried on a sodium sulfate column, concentrated to 1 ml by Kuderna Danish evaporation, and analyzed on a Finigan-4000 automated gas chromatograph/mass spectrometer (GC/MS). The GC column used was a DB-5MS, 30 m × 0.25 mm. The temperature program was 4 min at 30°C, 6°C/min to 300°C and 30 min hold. For analytical purposes, six internal standards (IS) were injected at 40 µg/L in the sample prior to GC/MS analysis. MS analysis was performed for both target and non-target compounds. Non-target compounds were tentatively identified based on an MS library search and mass spectra evaluation, while target compounds were confirmed and quantified by known standards and standard curves.

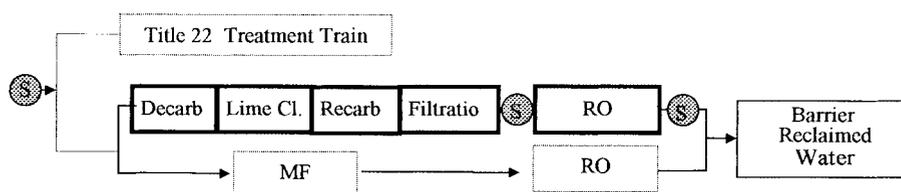


Figure 1 Flow chart of the West Basin Water Recycling Facility.

(S: sampling points; MF: microfiltration; RO: reverse osmosis; Cl: Clarification)

BNA interpretation. The original chromatograms obtained after GC/MS analysis were normalized on the average GC/MS scan index numbers of the six IS injected prior to analysis. Data analysis was performed by comparing influents and effluents of a same process for the same sampling date. Direct chromatographic comparison provides an indication of the changes that are occurring in the water matrix composition based on GC profile analysis as described by Suffet and Glaser (1980). ICA was performed based on the protocol described by Levine (1999) which includes chi-square analysis to compare chromatograms, semi-quantitative analysis and finally, lists of tentatively identified compounds. The objective is to highlight the change in the chemical composition of the base neutral organic fraction.

Results

General plant data

Table 1 presents the average annual data for the influent water and the final effluent for both Title 22 and barrier treatment trains. The data clearly show the differences in water quality after each treatment train. The low TDS, TOC and turbidity values are indicative of the high treatment efficiency of RO in enhancing water quality.

Table 2 presents the annual concentrations for those trace organic compounds that were quantified. It is interesting to note that the compounds that are detected by the conventional base neutral analysis protocol (EPA Method 625) and volatile organic analysis (EPA Method 524.2) are primarily, disinfection byproducts, such as chloroform and bromoform. Similar observations were made at the Lake Arrowhead wastewater reclamation site by Levine *et al.* (2000).

Table 1 1997 annual monitoring data at the West Basin Water Reclamation plant (adapted from West Basin Municipal Water District, 1998, 1998)

Constituents	Units	Influent		Title 22 Water		Barrier Water	
		Permit	Ann avg	Permit	Ann avg	Permit	Ann avg
Turbidity	NTU	–	5.4	2	1.8	2	0.39
pH	pH units	–	6.7	–	6.9	–	8
TOC	mg/L	20	10.6	–	–	2	0.7
TSS	mg/L	30	14	20	1.2	–	0.1
BOD	mg/L	30	27	–	1	–	–
TDS	mg/L	–	–	1000	789	–	124

Table 2 West Basin 1997 annual average concentration in µg/L for specific organic compounds (adapted from West Basin Municipal Water District, 1998)

Constituent	Method	Detection Limit	Title 22 Water	Barrier Water
<i>VOLATILE ORGANICS (µg/L)</i>				
Trichloromethane	524.2	0.2	6.9	2.2
Dibromochloromethane	524.2	0.3	2.6	1.2
Bromodichloromethane	524.2	0.3	3.4	1.2
Tribromomethane	524.2	0.3	1.7	1.1
1,4-Dichlorobenzene	524.2	0.2	1.3	0.4
Tetrachloroethylene	524.2	0.3	1.6	0.26
<i>ACID & BASE/NEUTRAL Extractable (µg/L)</i>				
N-Nitrosodimethylamine	625	1	1	ND
Bis (2-Ethylhexyl) phthalate	525.1	0.4	0.8	ND
Phenol	625	1	0.3	ND

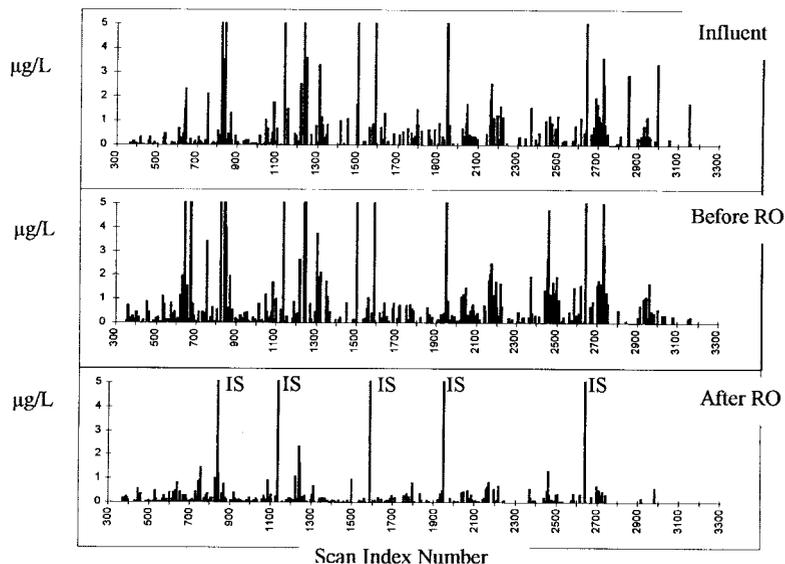


Figure 2 Base neutral computer re-constructed chromatograms at the West Basin Recycling Plant on the barrier treatment train (17 L composite sample drawn on May 5, 1998; analyzed by GC/MS; GC conditions: 4 min at 30°C, 6°C/min to 300°C, hold 30 min; column: DB-5MS, 30 m × 0.25 mm)

Base neutral data

Figure 2 presents computer reconstructed chromatograms across the barrier treatment train. The visual comparison of the different computer reconstructed chromatograms indicate that the base neutral content of the West Basin influent water is low and that the peaks are evenly distributed across the spectrum. This pattern is similar to those described in France at the Evry sewage treatment plant (Levine, 1999) and in Sweden at the Göteborg sewage treatment plant (Paxéus and Schröder, 1996), which were both activated sludge secondary effluents.

Figure 2 shows little modification in the peak distribution across the chromatogram until the RO unit. After RO treatment, the overall peak height decreased indicating the efficient removal of these micropollutants from the influent water. The highest peaks on the chromatograms were the IS used for quantification. In addition, the chromatograms indicated fewer later eluting compounds, indicating that the higher molecular weight and lower volatility compounds were preferentially removed from the water.

Discussion

Three new tools were used to evaluate quantitatively the data presented above: (1) a statistical test which compares similarities between chromatograms, (2) a semi quantitative analyses of the base neutral content and (3) a compound specific quantitative analysis.

Chi-square analysis

The chi-square statistical analysis was performed to compare chromatograms based on two parameters: the peak's elution time and the peak chromatographic area or estimated concentration. Table 3 summarizes the results for both November and May data comparing the influent and the effluent within the barrier treatment train for a same sampling period evaluating the spatial impact and for a same sample point studying the temporal effect on the profile.

The chi-square analysis statistically confirmed the observations visually observed and

Table 3 Chi-square statistics summary table of the West Basin Data sampled in November, 1997 and May, 1998. The tests were performed as a function of the treatment chain for a given time period (*Inf.* Influent water; *RO. Inf.* Reverse Osmosis Influent water; *RO. Eff.* Reverse osmosis effluent water) or as a function of time for a given sample point. The conclusion of the test is expressed as *SD* (significantly different) or *inc* (inconclusive)

Sampling period Comparison	November		May		November/ May	
	Inf./Final Eff.	Inf. / RO Inf.	RO Inf/RO Eff	Inf	Final RO	
Calculated Value			106,37	22.18	31.46	
Degree of Freedom	30	30	30	30	30	
Theoretical Value	43.7	43.7	43.7	43.7	43.7	
Conclusion	SD	Inc	SD	inc	inc	

extend it to a different sample period. First, no significant differences were observed between the May and November data sets for each specific site. Second, the lime clarification treatment chain did not significantly impact the profile of the chromatogram, suggesting that the chromatograms before and after lime treatment are not significantly different. Obviously, this does not mean that they are identical. Finally, chi-square analysis shows that the only significant step to impact the peak distribution of the chromatogram is RO treatment for which both analyses (November and May) showed significant differences before and after treatment. This conclusion confirms, the importance of RO in the treatment of organic micropollutants on an industrial scale. The next step will allow to quantify the differences and also highlight the possible lack of sensitivity of the statistical test to reveal subtle changes of the organic micropollution composition.

Figure 3 represents the average estimated concentration values at West Basin based on the different chromatograms obtained for each sample point. All data have been normalized and are directly comparable. Each chromatogram was broken down into three fractions : before scan index number 1132, between 1134 and 1945, and beyond scan index number 1947. Each axis represents the estimated average concentration in $\mu\text{g/L}$ of base neutral organic compounds for each fraction.

Figure 3 also presents the data from the barrier reclaimed water treatment train. After the first stage (i.e. Pre-decarbonation/lime clarification/recarbonation/filtration) the total base neutral organic concentration increased from 130 $\mu\text{g/L}$ to 188 $\mu\text{g/L}$. This increase in

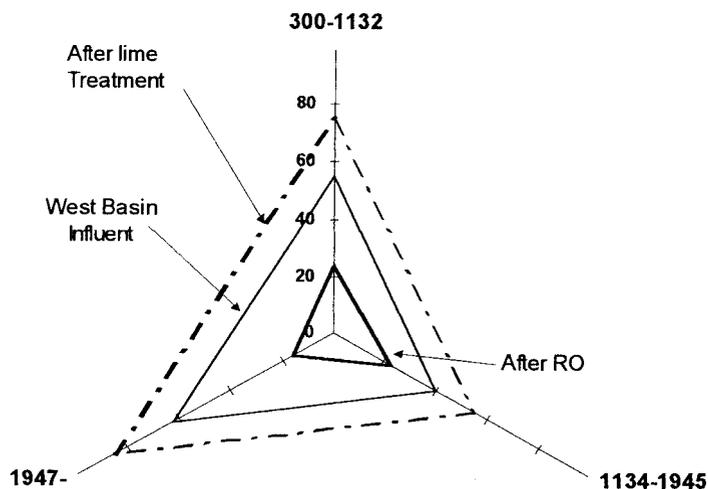


Figure 3 Triangular representation of the average base neutral concentration (in $\mu\text{g/L}$) as a function of three chromatographic elution fractions ($x < 1133$; $1133 < x < 1946$; $1946 < x$) at West Basin

concentration was evenly spread across the three fractions of the chromatogram. This result was unexpected, especially since the purpose of this preliminary stage is (1) to disinfect and (2) to protect the RO membrane. The disinfection capacity of this first stage being guaranteed by the high pH values in the lime clarifier, approximately 11.3. The RO pre-treatment is performed through direct precipitation of magnesium in the lime clarifier due to the high pH values, and the removal of suspended solids by flocculation with the addition of ferric chloride and filtration.

The increase in base neutral organic concentration noticed in Figure 3 after lime clarification can be the result of the high pH values after lime addition. Two mechanisms are hypothesized to be involved in the increase of base neutral concentration (1) desorption of base neutral compounds from the dissolved organic carbon (DOC) present in the influent, and (2) base hydrolysis of DOC and base neutral compounds.

The most accepted models generally proposed to describe the association between non-polar organic compounds and DOC suggest that humic substances bind organic compounds by a process of incorporation into the DOC gel structure (Caron and Suffet, 1989). Consequently, the presence of DOC in the water results in the increase of the apparent solubility of non-polar compounds in the water (Kile and Chiou, 1989). The observed solute-solubility enhancement results in a partition-like interaction between the solute and the DOC, which can be quantified when estimating its partition coefficient (K_{oc}). Previous work has shown that this partition coefficient varies with pH. Means and Wijayarathne (1989) measured decreasing K_{oc} values for benzidine, toluidine and azobenzene for pH values varying from 5 to 9. Similar observations were made by Gao *et al.* (1998) for pesticides and by Sabbah et Rebhun (1997) and Mollah and Robinson (1996) for phenolic compounds. In the first example, the authors showed that increasing the pH from 2 to 10 caused an increase in pesticide desorption resulting in an increase in free pesticide concentration in the solution. In the case of phenolic compounds (e.g. 2,4,5-trichlorophenol or pentachlorophenol), it was the overall adsorption coefficient of the compound which decreased with increasing pH values.

The desorption phenomena observed after increasing the pH can be explained partly through the nature of the compound. For example, 2,4,5-trichlorophenol (TCP) undergoes a basic hydrolysis and is transformed in to TCP⁻ (Schwarzenbach *et al.*, 1993) decreasing the compounds polarity (Jafvert *et al.*, 1990). Meanwhile, the excess of negatively charged ions can lead to changes on surface of particles. According to Schwarzenbach *et al.* (1993), at higher pH the surface charge buildup includes increasingly negative charges which can destabilize the original configuration of the DOC. This change could lead to the release of sorbed compounds particularly since electrostatic forces are much more important forces than van De Waals forces.

In addition to destabilizing the partition-like interaction of organic solutes with DOC, the high pH values present in the lime clarifier can also favor chemical degradation of dissolved organic carbon fraction by alkaline hydrolysis. Sonnenberg *et al.* (1989) identify benzenes, phenols and acids as major reaction products after base hydrolysis of natural humic material. According to Sonnenberg *et al.* (1989), these substructures were thought to be joined to DOC through carbon-carbon bonds. Examples of base catalyzed hydrolysis reactions are given by Schwarzenbach *et al.* (1993). Among the most important bonds within the humic structure are (1) base hydrolysis of carboxylic acid esters; (2) base elimination reactions and (3) base hydrolysis of carboxylic acid amides (Figure 4).

After the lime clarification, the water is recarbonated. This reduces the pH to approximately 7. According to Schwarzenbach *et al.* (1993) the desorption phenomena initially described is reversible. Yet due to base hydrolysis, the micropollutant concentration has increased, the type of organic micropollutants has changed, and finally due to the addition

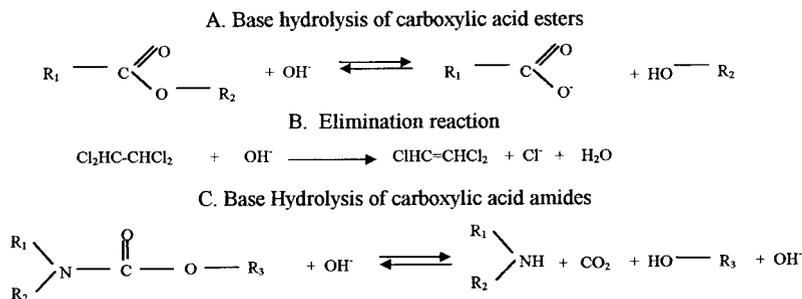


Figure 4 Organic reactions which could take place in an alkaline environment (adapted from Scherwartzbach *et al.*, 1993)

of ferric chloride in the water, the DOC content decreased. The result of these different mechanism is an increase in concentration of base neutral organics as it is shown in Figure 3. Further investigation of the specific phenomena is needed to better understand the mechanisms involved.

This is not the first time such a phenomena has been observed. At Evry (France), Levine (1999) measured high base neutral concentrations after the return flow, from sludge dewatering, before primary treatment. At the time, the authors suggested that base neutral compounds were in fact sorbed on to the floc in the activated sludge unit, which was then removed by gravity with the floc, and then released from the floc during sludge treatment. No additional explanation was given. Another suggestion is that the use of lime to stabilize the sludge could have resulted in the release of the base neutral compounds sorbed on to the floc. Hence, both phenomena would have for origin the use of lime leading to pH values greater than 11.

The main difference, between Evry's and West Basin's effluents is the impact to the downstream process. At the Evry wastewater treatment plant, the base neutral compounds tended to concentrate on site because of successive recycling events. On the other hand, at West Basin, the reverse osmosis unit efficiently removes large amounts of the base neutral compounds from the water. Yet the amount of base neutral compounds remaining is in the range of that measured after biological activated carbon filtration at the Lake Arrowhead reclamation plant (Levine *et al.*, 2000).

The final step is the analysis of base neutral compounds tentatively identified by GC/MS analysis after each sample point and their estimated concentrations. Table 4 lists these compounds and confirms the trend highlighted in Figure 3. Based on West Basin's RO pretreatment, four categories of compounds can be identified based on their removal rate (1) compounds which at least doubled in concentration before RO; (2) compounds that increased by at least 20% but less than 100%; (3) compounds that were apparently unaffected by the RO pretreatment, and (4) compounds that were partially or totally removed from the aqueous phase.

Among the first two categories are trihalomethanes and a large number of more polar oxygenated compounds such as hydroxy ethers. These compounds fall in the category of compounds identified by Sonnenberg *et al.* (1989) in the frame work of alkaline hydrolysis of humic material. The last categories of compounds group ester acidic compounds and phenols, which are compounds that could be subject to base hydrolysis reactions.

After RO all base neutral compounds were removed in part or totally from the water phase with the exception of 2-propanol-1-propoxy a polar oxygenated hydroxy ether which was significantly increased after RO. Also, a few other polar compounds were detected after RO and were not detected before RO, such as heptanoic acid. RO showed poor

Table 4 List of base neutral compounds tentatively identified by GC/MS analysis in µg/L for each sample point

retention time	Compounds	Influent µg/l	Lime treatment µg/l	After RO µg/l
THM/ Halogenated				
444	Chloroform	0.2	0.9	0.5
528	Dibromochloromethane	0.3	1.1	0.5
585	1,2,2 Trichloro-1,1-difluoroethane	0.5	0.1	
682	Bromoform	0.1	0.8	0.3
726	1,1,2,2-Tetrachloroethane			
956	Bromocyclopentane		0.2	< 0.1
Benzene				
460	Toluene	0.5	0.4	0.4
608	Chlorobenzene		0.1	<0.1
778	Benzaldehyde	0.1	0.6	0.3
Acid				
1040	Tetraethyl Ester Diphosphoric acid	1.1	1.2	0.2
1121	Heptanoic acid			0.2
1267	3,3-Dimethyl butanoic acid	0.5	0.8	0.3
1794	Phosphoric acid, tributyl ester	1.6	2.7	0.8
2031	1-Phenanthrene Carboxylic acid	0.4	1.1	< 0.1
Nitrogen compounds				
714	2,5-Dimethyl Pyrazine	0.2	0.2	0.1
1684	2,3,5,6-Tetrafluoro-1,4-Benzenediamine		0.8	0.1
1710	N,N-Diethyl-3-Methyl Benzamide	0.5	0.7	0.1
Ketones				
687	Cyclohexanone	0.2	0.3	0.1
844	2-Methyl 2,5-Cyclohexadiene-1,4-Dione		4	1.2
1217	4-(1,1 Dimethylethyl)-Cyclohexanone	2.7	2.7	1.1
1779	Benzophenone	0.3	0.6	0.3
Alcohols				
606	2-Propanol-1-Propoxy	0.7	0.2	0.4
638	2-Butoxy Ethanol	1.6	1.4	0.3
824	1-(2-Methoxy-1-Methylethoxy)-2-Propanol	5.9	7.2	0.2
1243	2-(2-Hydroxypropoxy)-1-Propanol	3.9	5.8	1.6
1635	2-(2-Phenoxyethoxy) Ethanol	1.4	0.8	0.1

removal for the earlier polar eluting compounds such as chloroform, toluene or dichloroacetonitrile ; while later eluting compounds were removed more efficiently with an average removal of 70.8%. These observations are consistent to those made by Levine *et al.* (1999) at the Lake Arrowhead wastewater reclamation plant.

Conclusions

This was the first time CLLE coupled with ICA was used to study the fate of micropollutants at an industrial wastewater reclamation plant. The analysis of the chromatograms is able to confirm conclusions that were made previously at the Lake Arrowhead water reclamation plant, such as the significance of RO as a treatment process removing base neutral compounds from the effluent.

More importantly, the data were able to highlight a possible release of base neutral compounds after lime clarification due to the high pH values achieved after the addition of lime to the water. This result was unexpected based on conventional water quality parameters, but has shown to be coherent with the present understanding of sorption theory to DOC. This result is of particular interest in areas where lime is used to achieve high pH levels such as sludge stabilization or drinking water treatment. Therefore additional work needs to be performed in this area.

Finally, this work has shown the practicality of using broad-spectrum analysis as a

monitoring tool for base neutral compounds. In the future such an approach could contribute to permitting policy, enhance risk assessment strategies, or used as a basis for resource evaluation.

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