# High Resolution-mass Spectrometry Analysis of Procured Polyethylene Extractions

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**Abstract**

This research uses mass spectrometry to examine the additive levels in unused plastic with the intention of impacting global polyethylene consumption and disposal methods. The inspiration for this project is attributed to high levels of microplastic found in our oceans and likelihood of chemical pollution (Ter Halle et al. 2017). Additives were expunged from four polyethylene plastics using three different methods: room temperature extraction, soxhlet extraction, and $CO_2$ supercritical fluid extraction. The resulting liquids were then evaporated and analyzed using gas chromatography. Five different additives were identified and analysed and the results can be used for future environmental chemistry research.

**Introduction**

Polyethylene is the most produced synthetic polymer in the world (Gilbert 2016). It is most commonly used in packaging materials, all of which inevitably make their way to landfills, forests, and waterways (Hermabessiere et al. 2017). It is known that as plastic breaks down, it releases toxins into its environment (Plastics in oceans decompose, release...). Although plastics are considered biochemically inert (Rocha-Santos et al. 2015), additives meant to improve their properties such as, plasticizers, colorants, and flame retardants (Fox 2008 ), concern environmental scientists. Additives known to disrupt the endocrine system are expelled from the polymer (Meeker et al. 2009). Previous environmental chemistry research has discovered traces of bisphenol A (BPA) and polystyrene in urban water samples from oceans surrounding the United States, Europe, and Asia (Corrales et al. 2015). These materials negatively affect our marine and terrestrial habitats because of physical buildups and expulsion of chemicals (Hermabessiere et al. 2017). Microplastics are often ingested by wildlife which can cause choking and starvation (da Costa et al. 2016). Plastic debris have also created a new habitat for microorganisms (da Costa et al. 2017), which is especially dangerous because of their ability to harbor diseases pathogens (Goedknegt et al. 2016). Algae, barnacles, mollusks, and small reptiles have been observed rafting on large plastic pieces, leaving environments susceptible to harmful invasive species (Li et al. 2016; Dias et al. 2016). Large plastics cause a loss of environmental aesthetic value which yields economic

repercussions for marine tourism reliant industries. These findings may be of interest to other scientists because of its interdisciplinary applications such as public health research, as well as water and waste management.

**Experimental**

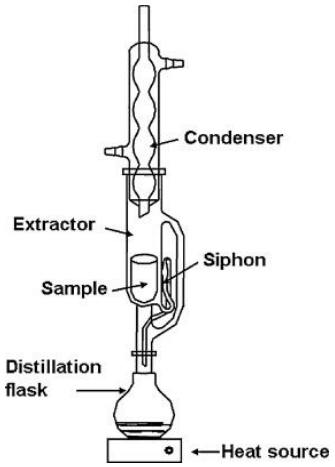
Procured Plastics: 15.0 g of collected plastic were cut into pieces no larger than 0.25 square inches and placed in crucibles. The crucibles were then filled with liquid nitrogen to preserve plastic before grinding. Machine speed was set to 14,000 rotations per minute (rpm) and plastic pieces were then inserted slowly using tweezers, grinded to 500 µm. The remaining plastic pieces were kept immersed in liquid nitrogen at all times. After completion, machine was stopped and dusted of all plastic particles, which were then stored in 2 mL glass containers.

Plastic pellets: 15.0 g of plastic pellets were placed in crucibles. The crucibles were then filled with liquid nitrogen to preserve plastic before grinding. Machine speed was set to 14,000 rotations per minute (rpm) and plastic pieces were inserted slowly using tweezers, grinded to 500 µm. The remaining plastic pieces were kept immersed in liquid nitrogen at all times. After completion, machine was stopped and dusted of all plastic particles, which were then stored in 2 mL glass containers. Freeze grinding samples preserves the plastic´s integrity and expands the surface area that is exposed to the supercritical fluid, increasing the efficiency of the extraction (Vandenburg et al. 1998). To obtain representative samples from inhomogeneous sample materials such as polymer compounds, particle reduction is used in order to have an increase in additive recovery.

Room temperature extraction: 1.0 g of grinded plastic was added to a small capped container and introduced to 10 mL of dichloromethane solvent. The container was then placed into the reciprocal shaker with specifications of 120 mot for 9 days. The solution was then filtered using 30 µm mesh nylon fabric and placed into a covered 100 mL container as preparation for evaporation.

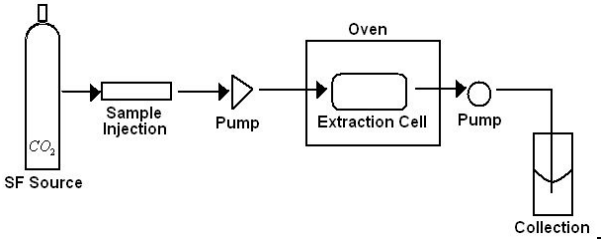
Soxhlet Extraction: 50 mL of dichloromethane was added to both the distillation flask and solvent extractor. A magnetic stirrer was then added to the distillation flask, and a cotton thimble with 1.0 g of plastic was placed into the solvent extractor and allowed to sink completely. An oil bath served as the heat source and was heated to 77$^{o}$ C while the magnetic stirrer was set to 250 rpm. The water tube, connected to the condenser, was turned on lightly to condensate the vapor solvent. The soxhlet was allowed to cycle

for 8 hours, then the liquid from the distillation flask and solvent extractor was pour into a 250 mL sealable jar and covered with foil as preparation for evaporation.




Soxhlet extraction is one of the most widely used techniques, but it is non selective and has the potential for for toxic emissions during extraction (Naudé et al. 1998).

Supercritical fluid extraction: The extraction cell was filled with glass beads and 1.00 g of desired ground plastic below the internal line. After inserting the extraction cell into the oven, chromoscope software was calibrated to the initial temperature and pressure specifications as shown below:




| Collector Vessel | Oven Temperature (°C) | Cosolvent Flow | Pressure | Time/Cycles |
|---|---|---|---|---|
| 1 | 40 | 0.50 mL/min | 200 bar | 30 min/2 |
| 2 | 40 | 1.0 mL/min | 200 bar | 30 min/2 |
| 3 | 50 | 0.50 mL/min | 200 bar | 30 min/2 |
| 4 | 50 | 1.0 mL/min | 200 bar | 30 min/2 |
| 5 | 60 | 1.0 mL/min | 200 bar | 30 min/2 |
| 6 | 60 | 0.50 mL/min | 200 bar | 30 min/2 |

The system was then allowed to run for desired time, and extraction liquids were capped in their original containers as preparation for evaporation.

This extraction method uses a supercritical fluid to separate soluble material from solids (Sapkale and Patil ) . It is an effective process that generates concentrated yields without degradation. Carbon dioxide polarity was adjusted with the co-solvent ethanol to increase extraction levels (Azevedo et al. 2008), and was chosen as the supercritical fluid primarily because it is environmentally benign (Lundin et al. 2015).

Mass Spectrometry: The 1.5 mL containers that held sample and let most of the remaining liquid evaporate under the hood. The container was then filled with 100 microliters of heptane. The new combination was syringed into new 1.5 mL containers than have pre installed plastic pieces. Prior to sample analysis hexane blanks were injected to ensure there was no residual contamination

Many of the additives in plastics are phthalates, man-made chemical compounds primarily used in the manufacture of plastics. We come in contact with phthalates through ingestion, inhalation, skin absorption, and intravenous injection. By looking at the peaks on the mass spectroscopy scans, common plastic additives were easily identified. The area of the peaks is directly related to the intensity and amount of additives in the plastic, the taller and wider the peak, the more of that additive is in the plastic in comparison to another plastic.

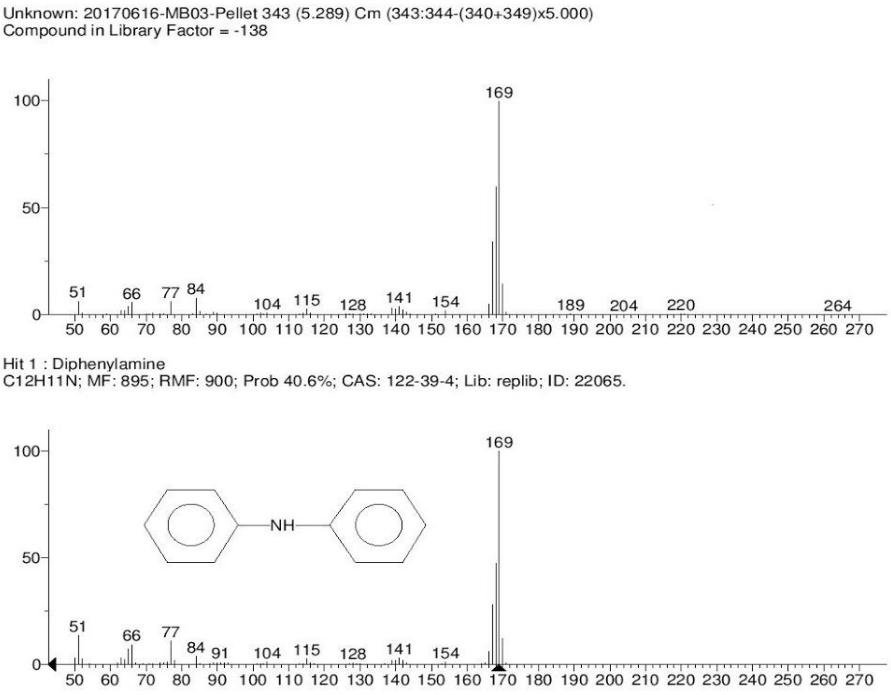
**Results and Discussion**

Phthalates are a diverse group of chemicals. Widespread mammal and marine research studies has concluded all phthalates have some disruptive effect the endocrine system (Rodgers et al. 2014). A comparison of five different phthalates was done across all procured plastics and their respective blank samples to insure this was not a contamination. Blank samples improve the reliability of chemical measurements and confidence in the results (Vitha et al. 2005)**.**

**Diphenylamine - Minute 5.3**

Diphenylamine ($C_{12}H_{11}N$), most commonly known as DPA, is an stabilizer most commonly added to polyethylene products. It is also registered as a pesticide with the United States Environmental Protection Agency (EPA) ([PDF]US EPA - Pesticides - Fact Sheet...). Stabilizers are used to help prevent plastic degradation caused by exposure to heat and light (Guo and Horsey 1998).

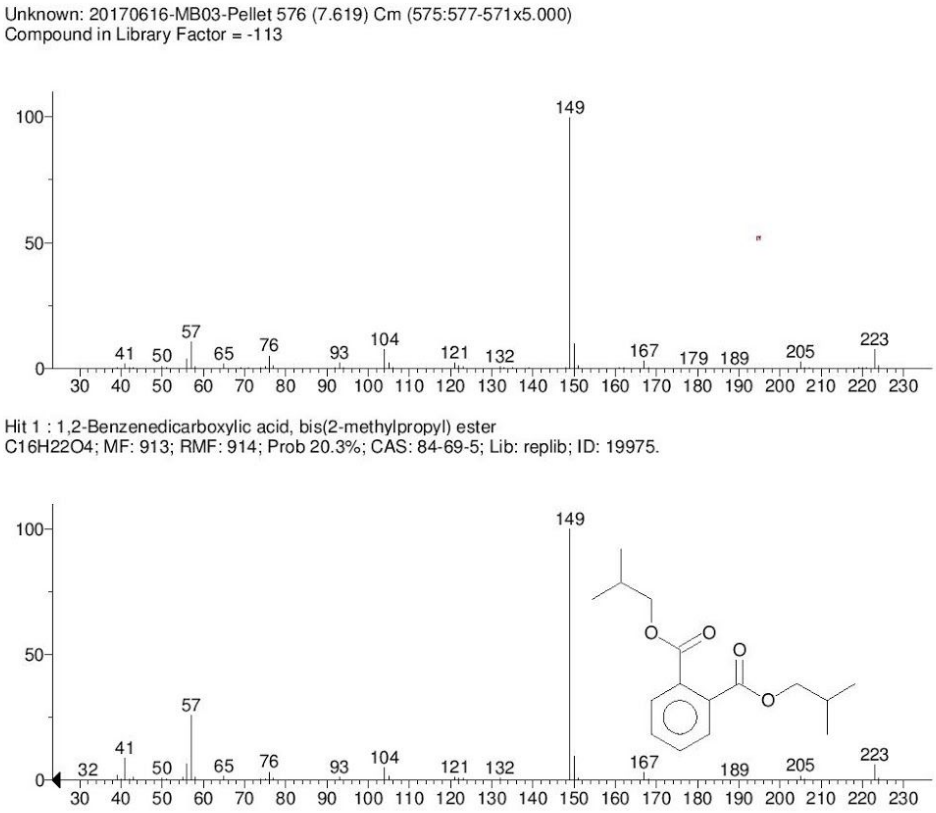
| **Sample name** | **Extraction form** | **Mass Spectrometry Integration** |
|---|---|---|
| Blanc Heptane | Heptane Sample | 0 |
| 20170616 MB03-Blanc | Room Temperature Extraction | 0 |
| 20170616 MB03-Pellet | Room Temperature Extraction | 1726 |
| 20170705 MB06-Nesquik | Room Temperature Extraction | 0 |
| 20170705 MB06-Trans | Room Temperature Extraction | 0 |
| 20170616 MB03-Folgers | Room Temperature Extraction | 1225 |
| 20170706 MB04-Blanc | Soxhlet Extraction | 529 |
| 20170706 MB04-Pellet | Soxhlet Extraction | 3015 |
| 20170616 MB04-Folgers | Soxhlet Extraction | 865 |



**Dibutyl Phthalate- Minute 7.6**

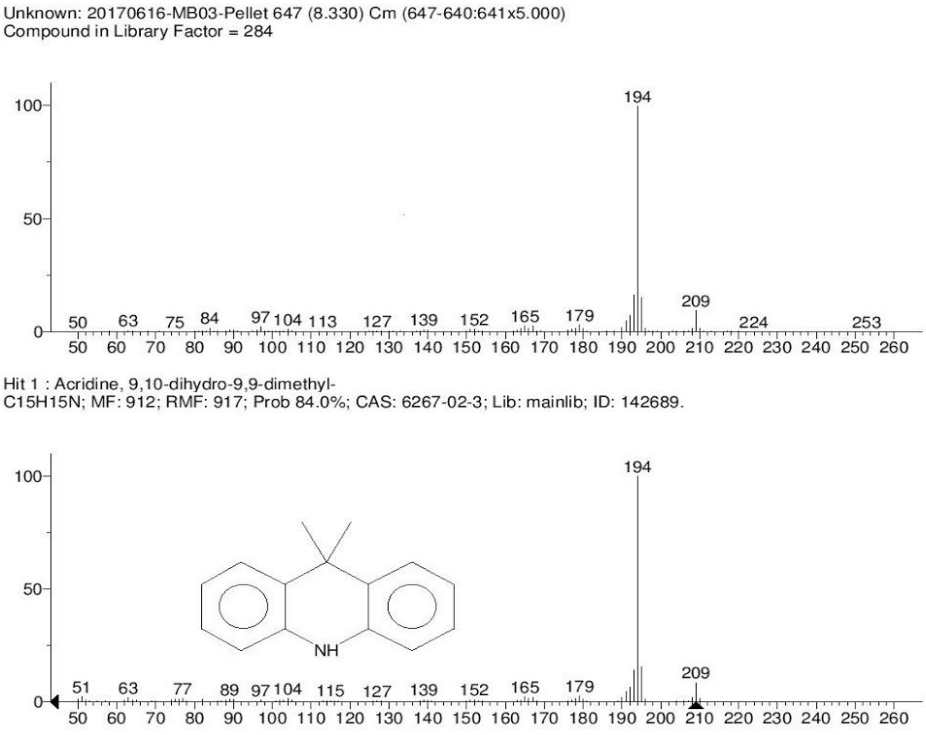
Dibutyl phthalates are plasticizers used to help make a number plastics, primarily PVC, soft and flexible ([PDF]Dibutyl Phthalate - United State...). They have been extensively researched for toxicology concerns because of their widespread use. In general, plasticizers are colorless and odorless with extremely low water solubility (Cadogan 1991).

| Sample name | Extraction form | Mass Spectrometry Integration |
|---|---|---|
| 20170615 Blanc Heptane | Heptane Sample | 0 |
| 20170610 MB03-Pellet | Room Temperature Extraction | 2600 |
| 20170616 MB03-Folger | Room Temperature Extraction | 360 |
| 20170616 MB03-Nesquik | Room Temperature Extraction | 120 |
| 20170610 MB04- Nesquik | Soxhlet Extraction | 850 |
| 20170706 MB04-Blank | Soxhlet Extraction | 380 |




**Acridine- Minute 8.3**

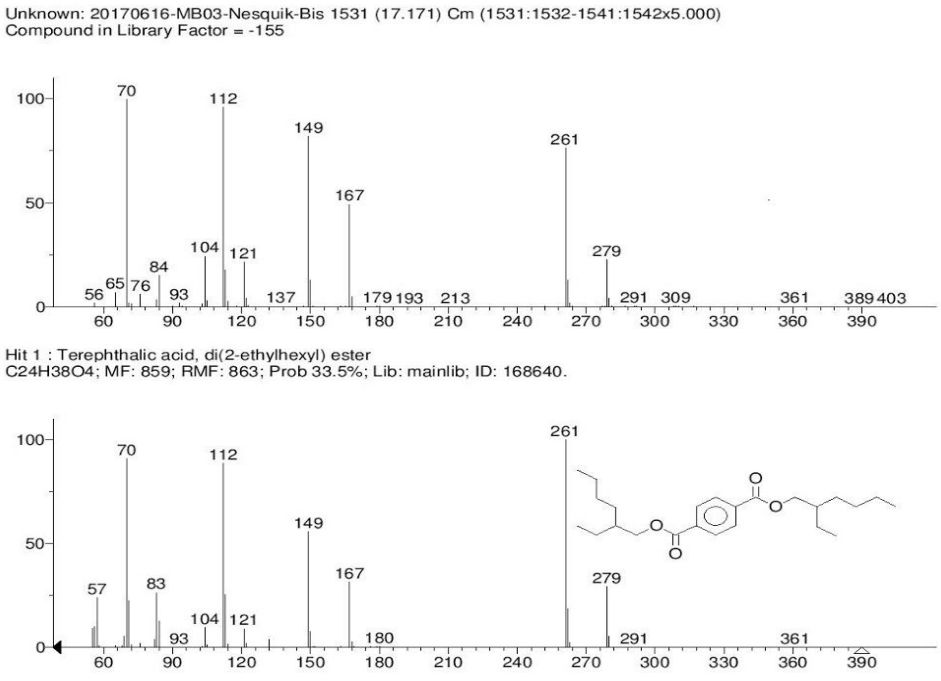
| Sample name | Extraction form | Mass Spectrometry Integration |
|---|---|---|
| 20170615 Blanc Heptane | Heptane Sample | 0 |
| 20170616 MB03-Blanc | Room Temperature Extraction | 452 |
| 20170705 MB06-Blanc | Room Temperature Extraction | 342 |
| 20170616 MB03-Pellet | Room Temperature Extraction | 3825 |
| 20170616 MB03-Nesquik | Room Temperature Extraction | 390 |
| 20170616 MB03-Folgers | Room Temperature Extraction | 2315 |
| 20170616 MB04-Blanc | Soxhlet Extraction | 976 |
| 20170610 MB04- Folgers | Soxhlet Extraction | 1656 |
| 20170706 MB04-Nesquik | Soxhlet Extraction | 2073 |




**Terephthalic Acid- Minute 17.8**

Terephthalic acid ($C_8H_6O_4$) is used in the production of polyethylene food and beverage storage containers.Terephthalic acid is toxic and known as an endocrine disruptor (Lin et al. 2016).Studies have suggested that it has some of the same effects on the body as estrogen, but no harmful effects on the reproductive system were found (Terephthalic Acid 2005).

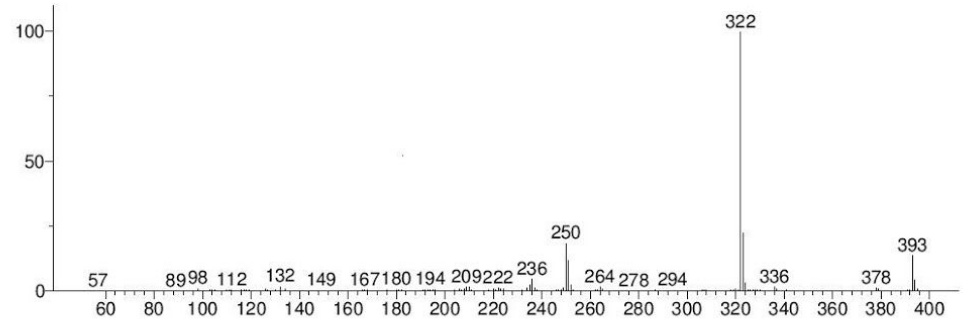
| Sample name | Extraction form | Mass Spectrometry Integration |
|---|---|---|
| 20170615 Blanc Heptane | Heptane Sample | 0 |
| 20170616 MB03-Blanc | Room Temperature Extraction | 0 |
| 20170705 MB06-Blanc | Room Temperature Extraction | 0 |
| 20170616 MB03-Pellet | Room Temperature Extraction | 3610 |
| 20170616 MB03-Nesquik | Room Temperature Extraction | 3569 |
| 20170616 MB03-Folgers | Room Temperature Extraction | 1616 |
| 20170705 MB06-Trans | Room Temperature Extraction | 0 |
| 20170616 MB04-Blanc | Soxhlet Extraction | 726 |
| 20170616 MB04-Nesquik | Soxhlet Extraction | 3569 |
| 20170616 MB04-Folgers | Soxhlet Extraction | 1681 |
| 20170616 MB04-Pellet | Soxhlet Extraction | 5449 |



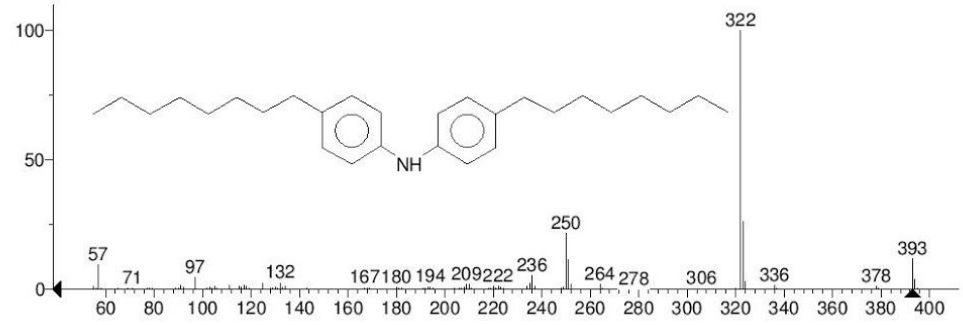

**Benzenamine- Minute 20.13**

| Sample name | Extraction form | Mass Spectrometry Integration |
|---|---|---|
| 20170615 Blanc Heptane | Heptane Sample | 0 |
| 20170616 MB03-Blanc | Room Temperature Extraction | 0 |
| 20170705 MB06-Blanc | Room Temperature Extraction | 0 |
| 20170616 MB03-Pellet | Room Temperature Extraction | 0 |
| 20170616 MB03-Nesquik | Room Temperature Extraction | 0 |
| 20170616 MB03-Folgers | Room Temperature Extraction | 10507 |
| 20170705 MB06-Trans | Room Temperature Extraction | 0 |
| 20170616 MB04-Blanc | Soxhlet Extraction | 0 |
| 20170616 MB04-Nesquik | Soxhlet Extraction | 0 |
| 20170616 MB04-Folgers | Soxhlet Extraction | 0 |
| 20170616 MB04-Pellet | Soxhlet Extraction | 0 |

Unknown: 20170616-MB03-Folgers 1827 (20.132) Cm (1827-1820:1821x5.000)
Compound in Library Factor = 625




Hit 1 : Benzenamine, 4-octyl-N-(4-octylphenyl)-
C28H43N; MF: 910; RMF: 914; Prob 98.0%; CAS: 101-67-7; Lib: mainlib; ID: 181518.



**Conclusion and Future Work**

At room temperature, the blank "extraction" had no significant traces of the respective compound, which allows us to make conscientious analyses of these plastic extractions (MB03/MB06). The respective compounds were found in the soxhlet blanc sample (MB04), making this extraction form unable to be accurately analyzed. The supercritical fluid extraction (MB05/MB07) had no traces of any related additive compound so it is also unable to be analyzed. From this data it is assumed that room temperature is the best extraction method for procured polyethylene plastics. The mass spectrometry analysis images compare researchers results (top image) to previously identified compounds, allowing a comparison between them for more accurate results.

These results can be used for future work in environmental chemistry. One possibility is to compare the additive levels and times to a controlled degradation of the same plastics to detect how much of the additives were transferred to the water. Additional research can be done to determine the longevity of these phthalates in humans.

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**Cultural Impact**

My time in Europe was very enriching. I enjoyed working in my laboratory and feel as if I got a graduate school experience. I worked alongside some very friendly people who helped me practice my French language skills. Alongside my research I was able to learn about the different cultures and lifestyles of many different people across the globe. I had the opportunity to travel to 8 countries and 3 different locations in France, most of which I visited by myself. The countries I visited were Morocco, Italy, England, Spain, Germany, Belgium, Poland, and Sweden as well as the French cities of Paris, Bordeaux, and Beziers. I had to use some detailed planning and quick thinking to successfully travel to so many places. Thank you for selecting me for this opportunity, it is one I will never forget.

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