

## **Abstract**

Sue D'Antonio, Joan Stevens, & Lynne Marshall,

In this experiment we took water samples from the bay near the battleship Texas to determine the presence of contaminants in the water. We employed an Agilent Time of Flight mass spectrometer to identify these contaminants utilizing accurate

## Introduction

The Battleship Texas was launched in 1912. It was the last of the Dreadnaught class of ships. The Texas served in both World Wars I and II. She is now located at the San Joaquin battle site memorial. She has been moored in this location since 1980.



Accurate Mass provides positive identification for compounds of interest by determining the mass to the 4<sup>th</sup> decimal place rather than just unit mass. This level of mass accuracy is obtained because of the high resolution of the instrument.

To prepare the water samples taken directly from the bay, we used Agilent QuEChERS. Not only does this type of extraction help to keep the instrument clean, it more importantly helps to eliminate ion suppression.

The Agilent 1290 UHPLC was used to introduce the samples into the Time of Flight mass spectrometer. Chromatographic separation with the 1290 facilitates fast, high resolution using Agilent sub 2 micron packing material.

The TOF data give us the ability to search databases to find compounds matches. If a compound is not in a database, we are able to use the ring and double bond information, plus the formula to propose identification such as we did with the Benzaldehyde.

Agilent Bond Elut EN extraction salts p/n 5982-5650 Agilent Bond Elut EN d-SPE p/n 5982-5056 Agilent Ceramic Homogenizers p/n 5982-9313 Accurately weigh 10 g silt bed sample (±0.05 g) into a 50 mL centrifuge tube, Add 10 mL of ACN and vortex, add Agilent Bond Elut QuEChERS EN extraction salt packet Cap and shake vigorously 1 min Centrifuge @ 5000 rpm 5 min Transfer 6 mL of upper ACN layer to Bond Elut QuEChERS EN dispersive-SPE tube Vortex 1min, centrifuge @ 5000 rpm for 5 min Transfer 200 µL extract to LC vial, add 10 µL of 1% FA, dilute with 800 µL aqueous solution Sample are ready for LC-TOF analysis



## **HPLC Parameters**

QuEChERS EN sample preparation procedures flow chart

Mobile Phase A H2O + 0.1% Formic Acid Mobile Phase B MeOH + ).1% Formic Acid Flow 0.5 ml/min Gradient 5% B to 95% B over 10 minutes Injection volume 1 ul with a 60 sec ACN/IPA/H20 wash Column temperature 40 degrees C Diode Array Detector 254nm with a bandwidth off 4 and Reference off Flow cell 10mmpath 1.8 ul volume collecting all spectra

Column Zorbax Eclipse Plus 2.1 x 100mm 1.8 ul particle size

Agilent 1290 UHPLC system

## Time of Flight Parameters

Positive mode Agilent Jet Stream Gas temperature 350 degrees C Drying gas 10 I/min Nebulizer 60 degrees C Sheath gas temperature 350 degrees Sheath gas flow 11 I/min Mass range 110 to 1100 Reference Masses 121.050873 & 922.009798

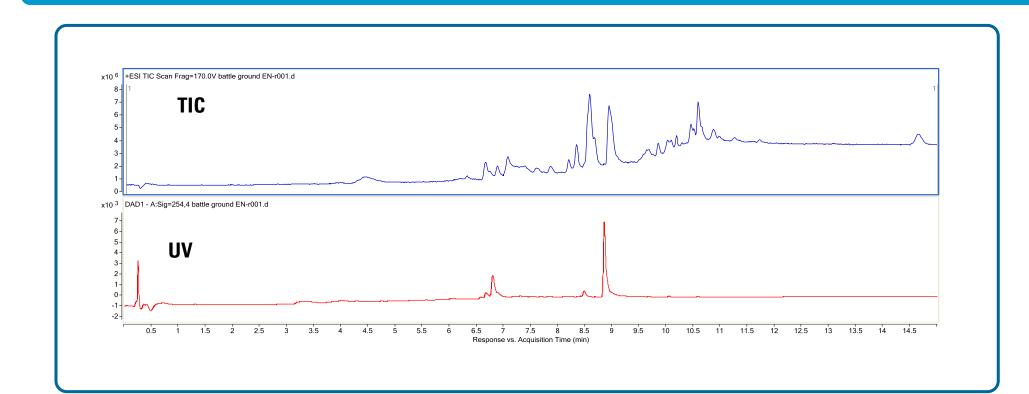
# <u>Software</u>

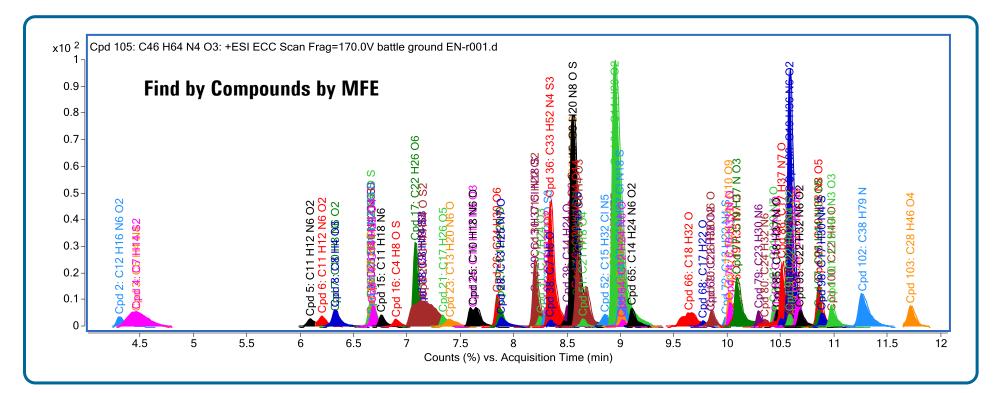
Mass Hunter Data Acquisition Mass Hunter Qualitative Analysis Molecular Feature Extractor Personal Compound Data Library

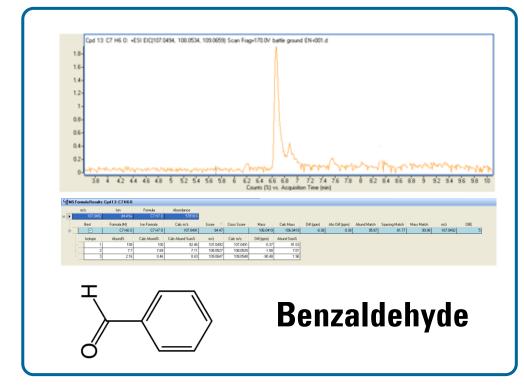


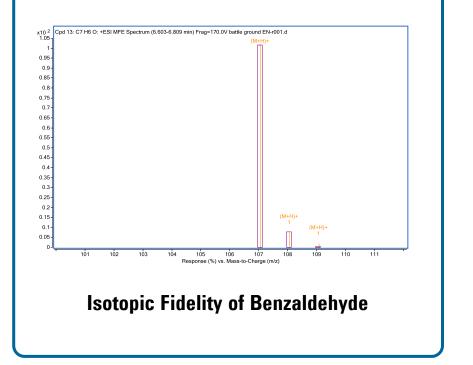
G6230AA Accurate Mass TOF LC/MS Agilent Jet Stream

# **Experimental**



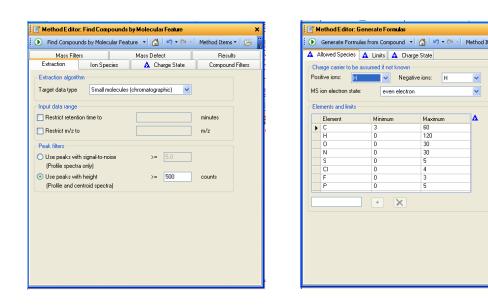






# **Results and Discussion**

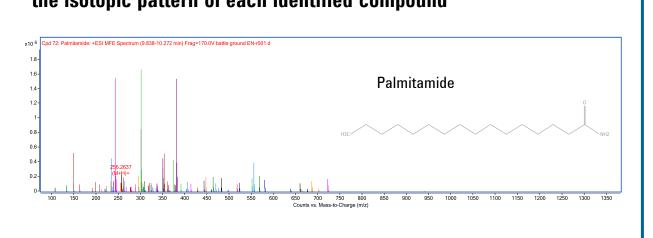
You choose your possible elements in Molecular Formula Generator



(a)C	a Compound List												
III Automatically Show Columns III II I													
	Show/Hide	Cpd ▽	Label 7	Name ⊽⊽	Formula $ abla$	Score ♥	Mass ▽	Avg Ma. 🗤	Mass (DB) ▼	Base Peak ▼	m/z		
<b>∄</b> ▶	<b>∀</b>	54	Cpd 54: Paroxypropione	Paroxypropione	C9 H10 O2	99.81	150.0682	150.1929	150.0681	151.0755	151.075		
₽	₹	72	Cpd 72: Palmitamide	Palmitamide	C16 H33 N O	99.41	255.2565	255.4251	255.2562	256.2637	256.263		
₽	<b>?</b>	9	Cpd 9: Motapizone	Motapizone	C12 H12 N4 O S	99	260.0728	260.2484	260.0732	261.0801	261.080		
∄	<b>V</b>	29	Cpd 29: Menthyl salicylate	Menthyl salicylate	C17 H24 O3	99.68	276.1727	276.3809	276.1725	299.1622	299.162		
∄ _	<b>?</b>	42	Cpd 42: Imazamethabenz	lmazamethabenz	C16 H20 N2 O3	95.98	288.1481	288.3359	288.1474	289.1554	289.155		
₽	<b>?</b>	12	Cpd 12: Formylsulfisomidine	Formylsulfisomidine	C13 H14 N4 O3 S	99.9	306.0785	306.3021	306.0787	307.0858	307.085		
±	<b>V</b>	25	Cpd 25: Estriol triacetate	Estriol triacetate	C24 H30 O6	99.89	414.2044	414.5261	414.2042	437.1937	437.193		
₽ _	<b>V</b>	64	Cpd 64: Dodemorph	Dodemorph	C18 H35 N O	99.69	281.2721	281.4329	281.2719	282.2794	282.279		
±	V	57	Cpd 57: Carbofuran (-C2H3NO)	Carbofuran (-C2H3NO)	C10 H12 O2	99.96	164.0837	164.2364	164.0837	165.091	165.09		
⊕	<b>V</b>	60	Cpd 60: Butibufen	Butibufen	C14 H20 O2	99.57	220.1465	220.3162	220.1463	243.1358	243.135		
±	V	81	Cpd 81: Aldimorph	Aldimorph	C18 H37 N O	99.55	283,2878	283,498	283.2875	284.2952	284.295		

Then running Molecular Formula Generator the compound Formulas and the match score will be displayed

## The results from a database search. This shows the structure and the isotopic pattern of each identified compound



### Compounds found by searches of METLIN, Agilent Tox and Forensic databases

Palmitamid		C16 H33 N O	98.92	255.2565
Motapizon		C12 H12 N4 O S	94.26	260.0728
Formylsulfisomidine	Э	C13 H14 N4 O3 S	95.49	306.0785
Butibufe	n	C14 H20 O2	98.68	220.1466
Saccharopine	C11 H20 N2 O6		81.19	276.1327
Purine	C5 H4 N4		52.56	120.0436
Oleamide	C18 H35 N O		99.09	281.2721
N-Hexadecyl-L-				
hydroxyproline	C21 H41 N O3		85.13	355.3083
N-(3-Indolylacetyl)-L-				
isoleucine	C16 H20 N2 O3		79.15	288.148
methyl 8-[2-(2-formyl-				
vinyl)-3-hydroxy-5-oxo-				
cyclopentyl]-octanoate			91.27	310.1782
Met Trp Phe	C25 H30 N4 O4 S		92.93	482.1983
Met His Lys	C17 H30 N6 O4 S		90.38	414.2045
Lys Cys His	C15 H26 N6 O4 S		90.94	386.1733
hydrocinnamic acid	C9 H10 O2		95.91	150.0682
GPA(10:0/10:0)	C23 H45 O8 P		83.37	480.2842
cyclandelate	C17 H24 O3		84.93	276.173
5,8-tetradecadienal	C14 H24 O		99.32	208.1827
4,7,10,13-				
hexadecatetraenoic				
acid	C16 H24 O2		86.33	248.1777
3E,13Z-octadecadien-1-				
ol	C18 H34 O		98.82	266.2608
2-Phenylbutyric acid	C10 H12 O2		47.6	164.0837
20-oxo-heneicosanoic				
acid	C21 H40 O3		84.15	340.2972
10,10-dimethyl-				
5Z,8Z,11Z-eicosatrienoi				
acid	C22 H38 O2		95.48	334.2872

# **Conclusion**

Utilizing Agilent's Time of Flight accurate mass, high resolution, superior isotopic fidelity and the unique features of Mass Hunter software, such as Molecular Feature Extractor and Molecular Formula Generator, we were able to identify over 100 compounds found in the samples.