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Determination of the identity and quantity of leachables and extractables from Medical Grade Polymer Sheets

Sponsor: Valued Customer
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Released by:

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Identification of Test Articles

Test Articles

1. *Medical Grade Polymer Sheets*

Objective

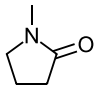
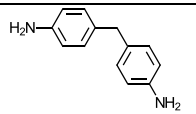
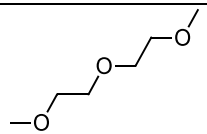
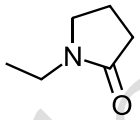
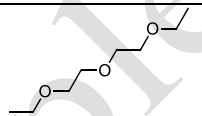
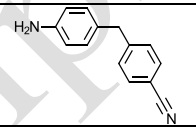
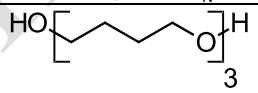
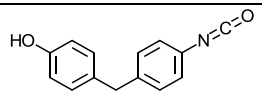
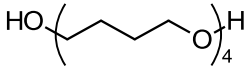
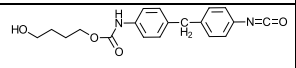
The goal of this analysis was to identify the chemical composition of extractables and leachables from three medical grade polymer films. The three polymers included a Thermoplastic polyurethane (TPU), polycarbonate (PC) and a silicone based, room temperature cured, biomedical grade elastomer (Silicone).

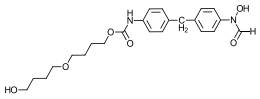
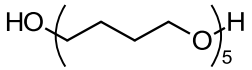
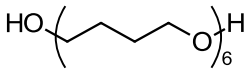
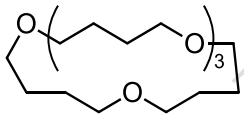
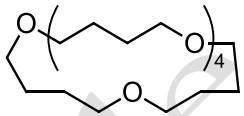
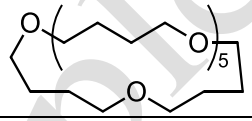
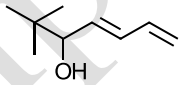
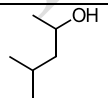
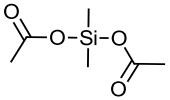
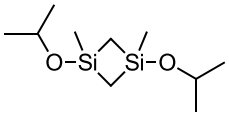
Summary of Results

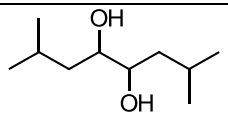
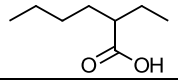
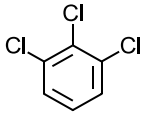
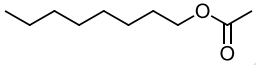
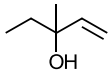
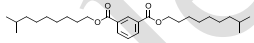
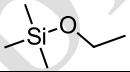
The polymer films were subjected to extractions with water, ethanol and hexane (extractables) and saline solutions (leachables). The resulting extracts were analyzed by QTOF-LCMS, QTOF-GCMS, HGCMS, and ICP-MS. **Tables 3-6** include a summary of the extractables and leachables found in the extracts.

Table 1: Alphabetical List of Acronyms	
CAD	Charged Aerosol Detection
CAS	Chemical Abstracts Service Registry Number
HGCMS	Headspace Gas Chromatography Mass Spectroscopy
ICP-MS	Inductively Coupled Plasma Mass Spectroscopy
N.A	Not Applicable
N.D.	Not Detected
N.D.L.	Not Detected by LCMS
N.D.H.	Not Detected by HPLC
N.F.	Not Found
QQQ	Triple Quadrupole Mass Spectrometer
QTOF-GCMS	Quadrupole Time of Flight Gas Chromatography Mass Spectroscopy
QTOF-LCMS	Quadrupole Time of Flight Liquid Chromatography Mass Spectroscopy
UHPLC	Ultrahigh Performance Liquid Chromatography
UV	Ultraviolet
FTIR	Fourier Transform Infrared Spectroscopy
Semi-Quant	Semi-Quantitative Analysis

Table 2 Analysis Methods						
Extract	Saline	Water	Ethanol	Ethanol Precipitate	Hexane	Purpose
Non-volatile Residue	X	X	X		X	Screen for Non-volatile Extractables
QTOF-GCMS	X	X	X		X	Volatile, Semi-volatiles Compound Identification
QTOF-LCMS	X	X	X		X	Non-volatile, Ionizable Compound Identification
QQQ	X					Quantitation of 4,4-methylenedianiline
HGCMS	X	X				Volatile Compound Identification
ICP-MS	X	X				Metals analysis
GCMS-Semi-Quant	X	X	X		X	Semi-quant of Volatile, Semi-volatile Compounds
UHPLC-CAD-UV	X	X	X		X	Semi-quant of Non-volatile, Ionizable Compounds
FTIR				X		Precipitate Identification

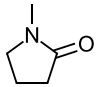
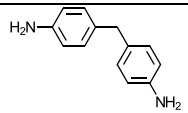
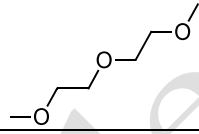
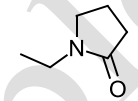
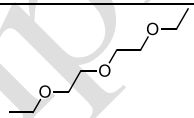
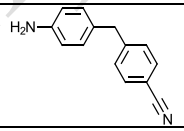
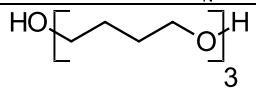
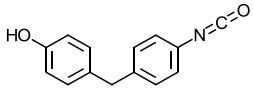
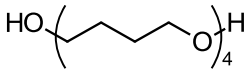
Table 3 Summary of Compounds Identified in Saline Extract (Leachables)							
Proposed Identification	CAS	Chemical Formula	Structure	Identification Confidence	Mass per Test Article (µg)	Detected by	Quantification Method
2-Pyrrolidinone, 1-methyl-	872-50-4	$C_5 H_9 N O$		*Confident*	220.79	LCMS	CAD
4,4′-methylenedianiline	101-77-9	$C_{13} H_{14} N_2$		*Confirmed*	15.28	LCMS	QQQ
2-Methoxy-1-(2-methoxyethoxy)ethane	111-96-6	$C_6 H_{14} O_3$		*Confident*	345.84	LCMS	CAD
2-Pyrrolidinone, 1-ethyl-	2687-91-4	$C_6 H_{11} N O$		*Confident*	701.45	LCMS	CAD
2-Ethoxy-1-(2-ethoxyethoxy)ethane	112-36-7	$C_8 H_{18} O_3$		*Confident*	318.48	LCMS	CAD
p-[(*p*-Aminophenyl)methyl] benzonitrile	748104-31-6	$C_{14} H_{12} N_2$		*Tentative*	265.73	LCMS	UV
Poly butylene glycol	*N.A.*	$C_{12} H_{26} O_4$		*Confident*	345.84	LCMS	CAD
p-[(p-Isocyanatophenyl)methyl]phenol	*N.A.*	$C_{14} H_{11} N O_2$		*Tentative*	141.66	LCMS	UV
Poly butylene glycol	*N.A.*	$C_{16} H_{34} O_5$		*Confident*	550.02	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{19}H_{20}N_2O_4$		*Tentative*	340.95	LCMS	UV

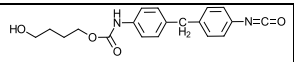
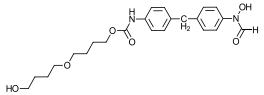
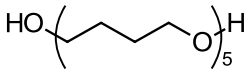
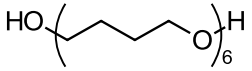
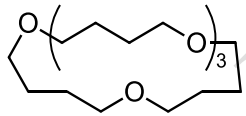
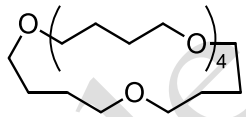
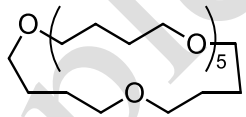
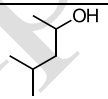
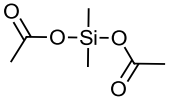
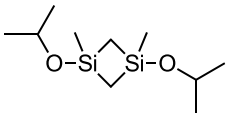
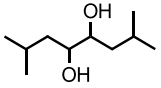
p-({p-[N-Formyl(hydroxyamino)]phenyl}methyl)phenylamino 5-(4-hydroxybutoxy)valerate	*N.A.*	$C_{23}H_{30}N_2O_6$		*Tentative*	1044.36	LCMS	UV
Poly butylene glycol	*N.A.*	$C_{20}H_{42}O_6$		*Confident*	240.33	LCMS	CAD
Poly butylene glycol	*N.A.*	$C_{24}H_{50}O_7$		*Confident*	2519.54	LCMS	CAD
Cyclic poly butylene glycol	*N.A.*	$C_{20}H_{40}O_5$		*Confident*	927.12	LCMS	CAD
Cyclic poly butylene glycol	*N.A.*	$C_{24}H_{48}O_6$		*Confident*	5492.39	LCMS	CAD
Cyclic poly butylene glycol	*N.A.*	$C_{28}H_{56}O_7$		*Confident*	1322.79	LCMS	CAD
6,6-Dimethyl-1,3-heptadien-5-ol	81912-03-0	$C_9H_{16}O$		*Tentative*	378.30	GCMS	GCMS
2-Pentanol, 4-methyl-	108-11-2	$C_6H_{14}O$		*Tentative*	1326.81	GCMS	GCMS
Silanediol, dimethyl-, diacetate	2182-66-3	$C_6H_{12}O_4Si$		*Tentative*	1308.99	GCMS	GCMS
1,3-Diisopropoxy-1,3-dimethyl-1,3-disilacyclobutane	198066-66-9	$C_{10}H_{24}O_2Si_2$		*Tentative*	220.22	GCMS	GCMS

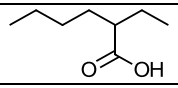
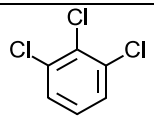
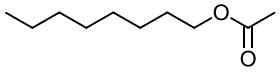
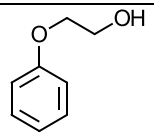
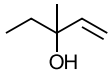
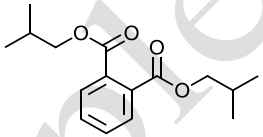
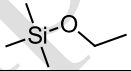
4,5-Octanediol, 2,7-dimethyl-	*N.A.*	$C_{10}H_{22}O_2$		*Tentative*	209.93	GCMS	GCMS
Siloxanes	*N.A.*	*N.A.*	*N.A.*	*Tentative*	29391.05 (GC)	GCMS	GCMS
Hexanoic acid, 2-ethyl-	149-57-5	$C_8H_{16}O_2$		*Tentative*	23730.97	GCMS	GCMS
Benzene, 1,2,3-trichloro-	87-61-6	$C_6H_3C_{l3}$		*Confident*	4447.15	GCMS	GCMS
Acetic acid, octyl ester	112-14-1	$C_{10}H_{20}O_2$		*Tentative*	291.96	GCMS	GCMS
Hydrocarbons	*N.A.*	*N.A.*	*N.A.*	*Tentative*	1140.26	GCMS	GCMS
1-Penten-3-ol, 3-methyl- and isomers	918-85-4	$C_6H_{12}O$		*Confident*	1250.80	GCMS	GCMS
1,2-Benzenedicarboxylic acid, bis(8-methylnonyl) ester	89-16-7	$C_{28}H_{46}O_4$		*Tentative*	448.87	GCMS	GCMS
Ethoxytrimethylsilane	1825-62-3	$C_5H_{14}OSi$		*Confident*	1657.14 (HGCMS)	HGCMS	HGCMS
Li	N.A.	N.A.	N.A.	N.A.	0.55	ICP-MS	ICP-MS
Mg	N.A.	N.A.	N.A.	N.A.	89.86	ICP-MS	ICP-MS
Si	N.A.	N.A.	N.A.	N.A.	2867.36	ICP-MS	ICP-MS
Cr	N.A.	N.A.	N.A.	N.A.	0.04	ICP-MS	ICP-MS
Co	N.A.	N.A.	N.A.	N.A.	0.13	ICP-MS	ICP-MS
Ni	N.A.	N.A.	N.A.	N.A.	2.16	ICP-MS	ICP-MS
Cu	N.A.	N.A.	N.A.	N.A.	0.83	ICP-MS	ICP-MS
As	N.A.	N.A.	N.A.	N.A.	0.07	ICP-MS	ICP-MS
Mo	N.A.	N.A.	N.A.	N.A.	0.10	ICP-MS	ICP-MS
Ag	N.A.	N.A.	N.A.	N.A.	0.38	ICP-MS	ICP-MS
Cd	N.A.	N.A.	N.A.	N.A.	0.44	ICP-MS	ICP-MS
Gd	N.A.	N.A.	N.A.	N.A.	1.25	ICP-MS	ICP-MS

Pt	N.A.	N.A.	N.A.	N.A.	0.29	ICP-MS	ICP-MS

Table 4
Summary of Compounds Identified in Water Extract (Extractables)

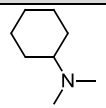
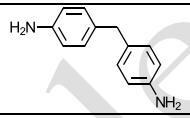
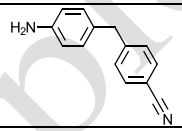
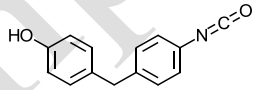
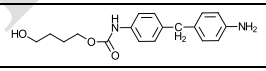
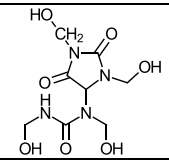
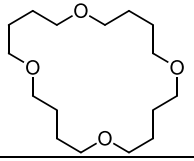
Proposed Identification	CAS	Chemical Formula	Structure	Identification Confidence	Mass per Test Article (µg)	Detected by	Quantification Method
2-Pyrrolidinone, 1-methyl-	872-50-4	$C_5 H_9 N O$		*Confident*	411.14 (GCMS), 208.05 (CAD)	GCMS, LCMS	GCMS, CAD
4,4′-methylenedianiline	101-77-9	$C_{13} H_{14} N_2$		*Confirmed*	79.43	LCMS	UV
2-Methoxy-1-(2-methoxyethoxy)ethane	111-96-6	$C_6 H_{14} O_3$		*Confident*	28.47	LCMS	CAD
2-Pyrrolidinone, 1-ethyl-	2687-91-4	$C_6 H_{11} N O$		*Confident*	820.98	LCMS	CAD
2-Ethoxy-1-(2-ethoxyethoxy)ethane	112-36-7	$C_8 H_{18} O_3$		*Confident*	63.44	LCMS	CAD
p-[(*p*-Aminophenyl)methyl]benzonitrile	748104-31-6	$C_{14} H_{12} N_2$		*Tentative*	125.13	LCMS	UV
Poly butylene glycol	*N.A.*	$C_{12} H_{26} O_4$		*Confident*	368.90	LCMS	CAD
p-[(p-Isocyanatophenyl)methyl]phenol	*N.A.*	$C_{14} H_{11} N O_2$		*Tentative*	150.11	LCMS	UV
Poly butylene glycol	*N.A.*	$C_{16} H_{34} O_5$		*Confident*	179.33	LCMS	CAD

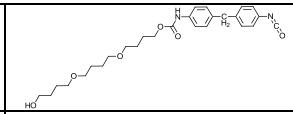
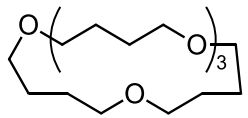
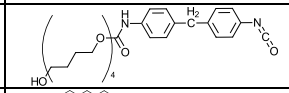
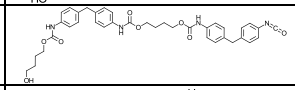
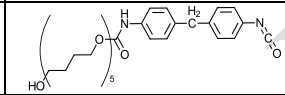
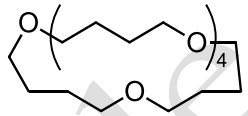
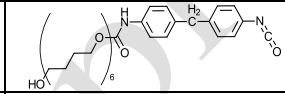
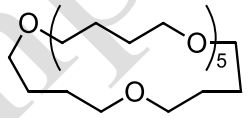
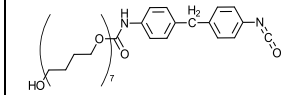
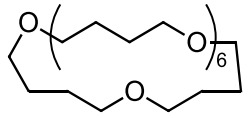
Poly butylene glycol urethane derivatives	*N.A.*	$C_{19}H_{20}N_2O_4$		*Tentative*	85.67	LCMS	UV
p-({p-[N-Formyl(hydroxyamino)] phenyl}methyl)phenylamino 5-(4-hydroxybutoxy)valerate	*N.A.*	$C_{23}H_{30}N_2O_6$		*Tentative*	20.73	LCMS	UV
Poly butylene glycol	*N.A.*	$C_{20}H_{42}O_6$		*Confident*	283.48	LCMS	CAD
Poly butylene glycol	*N.A.*	$C_{24}H_{50}O_7$		*Confident*	53.95	LCMS	CAD
Cyclic poly butylene glycol	*N.A*	$C_{20}H_{40}O_5$		*Confident*	1040.77	LCMS	CAD
Cyclic poly butylene glycol	*N.A*	$C_{24}H_{48}O_6$		*Confident*	39.71	LCMS	CAD
Cyclic poly butylene glycol	*N.A*	$C_{28}H_{56}O_7$		*Confident*	1710.89	LCMS	CAD
2-Pentanol, 4-methyl-	108-11-2	$C_6H_{14}O$		*Tentative*	1104.88	GCMS	GCMS
Silanediol, dimethyl-, diacetate	2182-66-3	$C_6H_{12}O_4Si$		*Tentative*	1065.89	GCMS	GCMS
1,3-Diisopropoxy-1,3-dimethyl-1,3-disilacyclobutane	198066-66-9	$C_{10}H_{24}O_2Si_2$		*Tentative*	608.54	GCMS	GCMS
4,5-Octanediol, 2,7-dimethyl-	*N.A*	$C_{10}H_{22}O_2$		*Tentative*	194.44	GCMS	GCMS
Siloxanes	*N.A*	*N.A.*	*N/A*	*Confident*	35001.66 (GCMS)	GCMS	GCMS

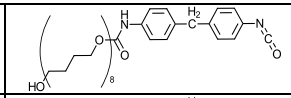
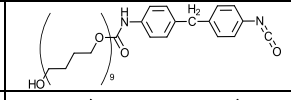
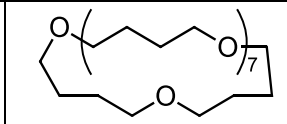
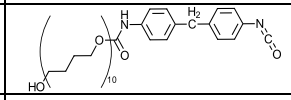
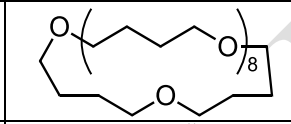
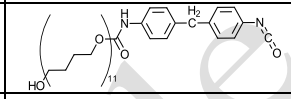
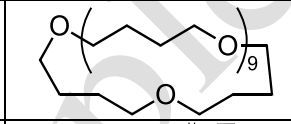
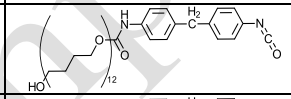
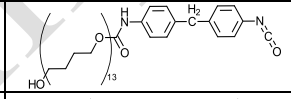
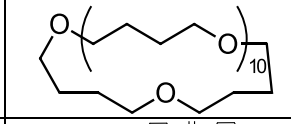
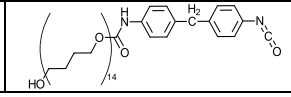
Hexanoic acid, 2-ethyl-	149-57-5	$C_8 H_{16} O_2$		*Tentative*	52045.36	GCMS	GCMS
Benzene, 1,2,3-trichloro-	87-61-6	$C_6 H_3 Cl_3$		*Confident*	4275.01	GCMS	GCMS
Acetic acid, octyl ester	112-14-1	$C_{10} H_{20} O_2$		*Tentative*	290.51	GCMS	GCMS
Ethanol, 2-phenoxy-	122-99-6	$C_8 H_{10} O_2$		*Tentative*	1077.02	GCMS	GCMS
Hydrocarbons	*N.A*	*N.A.*		*Tentative*	1676.43	GCMS	GCMS
1-Penten-3-ol, 3-methyl- and isomers	918-85-4	$C_6 H_{12} O$		*Confident*	1866.18	GCMS	GCMS
1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester	84-69-5	$C_{16} H_{22} O_4$		*Tentative*	130.36	GCMS	GCMS
Ethoxytrimethylsilane	1825-62-3	$C_5H_{14}OSi$		*Confident*	1367.10	HGCMS	HGCMS
Li	*N.A.*	*N.A.*	*N.A.*	*N.A.*	0.61	ICP-MS	ICP-MS
B	*N.A.*	*N.A.*	*N.A.*	*N.A.*	16.42	ICP-MS	ICP-MS
Na	*N.A.*	*N.A.*	*N.A.*	*N.A.*	265.78	ICP-MS	ICP-MS
Mg	*N.A.*	*N.A.*	*N.A.*	*N.A.*	195.27	ICP-MS	ICP-MS
Si	*N.A.*	*N.A.*	*N.A.*	*N.A.*	14205.75	ICP-MS	ICP-MS
K	*N.A.*	*N.A.*	*N.A.*	*N.A.*	69.87	ICP-MS	ICP-MS
Ca	*N.A.*	*N.A.*	*N.A.*	*N.A.*	717.60	ICP-MS	ICP-MS
Co	*N.A.*	*N.A.*	*N.A.*	*N.A.*	0.15	ICP-MS	ICP-MS
Ni	*N.A.*	*N.A.*	*N.A.*	*N.A.*	1.29	ICP-MS	ICP-MS
Cu	*N.A.*	*N.A.*	*N.A.*	*N.A.*	0.22	ICP-MS	ICP-MS
Rb	*N.A.*	*N.A.*	*N.A.*	*N.A.*	0.02	ICP-MS	ICP-MS

Sr	*N.A.*	*N.A.*	*N.A.*	*N.A.*	1.11	ICP-MS	ICP-MS
Mo	*N.A.*	*N.A.*	*N.A.*	*N.A.*	0.80	ICP-MS	ICP-MS
Cd	*N.A.*	*N.A.*	*N.A.*	*N.A.*	0.04	ICP-MS	ICP-MS
W	*N.A.*	*N.A.*	*N.A.*	*N.A.*	0.02	ICP-MS	ICP-MS
Ir	*N.A.*	*N.A.*	*N.A.*	*N.A.*	0.74	ICP-MS	ICP-MS
Pt	*N.A.*	*N.A.*	*N.A.*	*N.A.*	1.26	ICP-MS	ICP-MS

Table 5
Summary of Compounds Identified in Ethanol Extract (Extractables)

Proposed Identification	CAS	Chemical Formula	Structure	Identification Confidence	Mass per Test Article (µg)	Detected by	Quantification Method
N,N-Dimethylcyclohexanamine	98-94-2	$C_8 H_{17} N$		*Tentative*	1625.15	LCMS	CAD
4,4′-methylenedianiline	101-77-9	$C_{13} H_{14} N_2$		*Confirmed*	330.54	LCMS	UV
p-[(*p*-Aminophenyl)methyl] benzonitrile	*N.A.*	$C_{14} H_{12} N_2$		*Tentative*	1487.42	LCMS	UV
p-[(p-Isocyanatophenyl)methyl]phenol	*N.A.*	$C_{14} H_{11} N O_2$		*Tentative*	4407.18	LCMS	UV
Poly butylene glycol urethane derivatives	*N.A.*	$C_{18} H_{22} N_2 O_3$		*Tentative*	1845.51	LCMS	UV
Diazolidinyl urea	78491-02-8	$C_8 H_{14} N_4 O_7$		*Tentative*	1570.06	LCMS	CAD
Cyclic poly butylene glycol	*N.A.*	$C_{16} H_{32} O_4$		*Confident*	2671.85	LCMS	CAD

Poly butylene glycol urethane derivatives	*N.A.*	$C_{27}H_{36}N_2O_6$		*Tentative*	936.53	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{20}H_{40}O_5$		*Confident*	16471.84	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{31}H_{44}N_2O_7$		*Tentative*	5398.80	LCMS	UV
Poly butylene glycol urethane derivatives	*N.A.*	$C_{38}H_{40}N_4O_8$		*Tentative*	40243.08	LCMS	UV
Poly butylene glycol urethane derivatives	*N.A.*	$C_{35}H_{52}N_2O_8$		*Tentative*	1680.24	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{24}H_{48}O_6$		*Confident*	16444.30	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{39}H_{60}N_2O_9$		*Tentative*	2451.50	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{28}H_{56}O_7$		*Confident*	3773.65	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{43}H_{68}N_2O_{10}$		*Tentative*	8070.65	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{32}H_{64}O_8$		*Confident*	2589.22	LCMS	CAD

Poly butylene glycol urethane derivatives	*N.A.*	$C_{47}H_{76}N_2O_{11}$		*Tentative*	10742.51	LCMS	UV
Poly butylene glycol urethane derivatives	*N.A.*	$C_{51}H_{84}N_2O_{12}$		*Tentative*	523.35	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{36}H_{72}O_9$		*Confident*	11376.04	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{55}H_{92}N_2O_{13}$		*Tentative*	3443.11	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{40}H_{80}O_{10}$		*Confident*	5040.71	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{59}H_{100}N_2O_{14}$		*Tentative*	2341.32	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{44}H_{88}O_{11}$		*Confident*	936.53	LCMS	CAD
poly butylene glycol urethane derivatives	*N.A.*	$C_{63}H_{108}N_2O_{15}$		*Tentative*	7602.39	LCMS	UV
Poly butylene glycol urethane derivatives	*N.A.*	$C_{67}H_{116}N_2O_{16}$		*Tentative*	5508.98	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{48}H_{96}O_{12}$		*Confident*	1377.24	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{71}H_{124}N_2O_{17}$		*Tentative*	2093.41	LCMS	UV

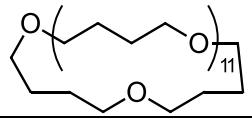
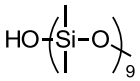
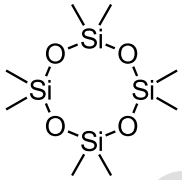
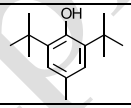
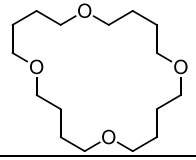
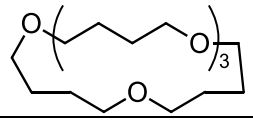
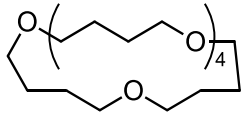
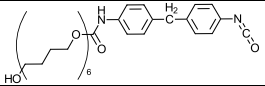
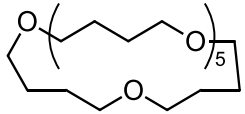
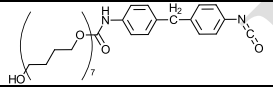
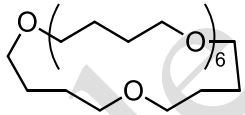
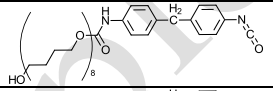
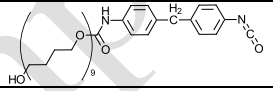
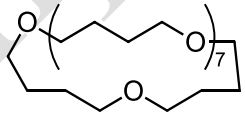
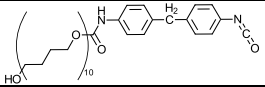
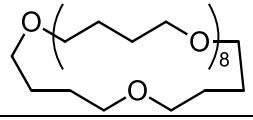
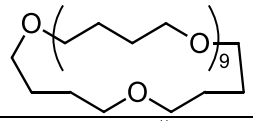
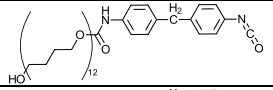
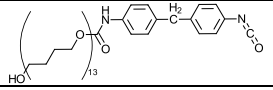
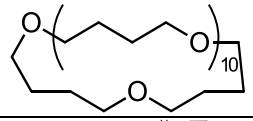
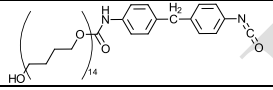
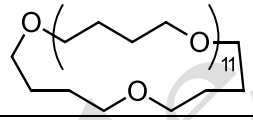
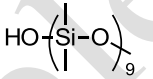
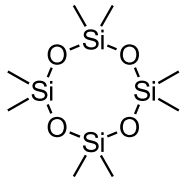
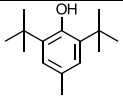
Cyclic poly butylene glycol	*N.A.*	$C_{52}H_{104}O_{13}$		*Confident*	2644.31	LCMS	CAD
Siloxane	N.A.	$C_{18}H_{56}O_{10}Si_9$		*Tentative*	2341.32	LCMS	CAD
Dicyclic Siloxane	*N.A.*	$C_{14}H_{42}O_9Si_8$	*N.A.*	*Tentative*	54098.16	LCMS	CAD
Siloxane	*N.A.*	$C_{16}H_{42}O_{12}Si_6$	*N.A.*	*Tentative*	1707.78	LCMS	CAD
Siloxane	*N.A.*	$C_{20}H_{48}O_{18}Si_6$	*N.A.*	*Tentative*	5123.35	LCMS	CAD
Siloxane	*N.A.*	$C_{23}H_{56}O_{20}Si_8$	*N.A.*	*Tentative*	605.99	LCMS	CAD
Cyclic Siloxanes	*N.A.*	$C_{2n}H_{6n}O_nSi_n$	*N.A.*	*Tentative*	4269.46	LCMS	CAD
Cyclotetrasiloxane, octamethyl-	556-67-2	$C_8H_{24}O_4Si_4$		*Confident*	38.70	GCMS	GCMS
Cyclic Siloxanes other than D4	*N.A.*	*N.A.*	*N.A.*	*Confident*	29281.48	GCMS	GCMS
Linear Siloxanes	*N.A.*	*N.A.*	*N.A.*	*Confident*	146407.42	GCMS	GCMS
Butylated Hydroxytoluene	128-37-0	$C_{15}H_{24}O$		*Confident*	1577.12	GCMS	GCMS

Table 6
Summary of Compounds Identified in Hexane Extract (Extractables)

Proposed Identification	CAS	Chemical Formula	Structure	Identification Confidence	Mass per Test Article (µg)	Detected by	Quantification Method
Cyclic poly butylene glycol	*N.A.*	$C_{16}H_{32}O_4$		*Confident*	3830.36	LCMS	CAD

Cyclic poly butylene glycol	*N.A.*	$C_{20}H_{40}O_5$		*Confident*	2747.02	LCMS	CAD
Cyclic poly butylene glycol	*N.A.*	$C_{24}H_{48}O_6$		*Confident*	889.88	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{39}H_{60}N_2O_9$		*Tentative*	3133.93	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{28}H_{56}O_7$		*Confident*	5068.45	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{43}H_{68}N_2O_{10}$		*Tentative*	18029.76	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{32}H_{64}O_8$		*Confident*	309.52	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{47}H_{76}N_2O_{11}$		*Tentative*	348.21	LCMS	UV
Poly butylene glycol urethane derivatives	*N.A.*	$C_{51}H_{84}N_2O_{12}$		*Tentative*	1586.31	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{36}H_{72}O_9$		*Confident*	12497.02	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{55}H_{92}N_2O_{13}$		*Tentative*	2437.50	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{40}H_{80}O_{10}$		*Confident*	928.57	LCMS	CAD

Cyclic poly butylene glycol	*N.A.*	$C_{44}H_{88}O_{11}$		*Confident*	1160.71	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{63}H_{108}N_2O_{15}$		*Tentative*	967.26	LCMS	UV
Poly butylene glycol urethane derivatives	*N.A.*	$C_{67}H_{116}N_2O_{16}$		*Tentative*	1315.48	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{48}H_{96}O_{12}$		*Confident*	657.74	LCMS	CAD
Poly butylene glycol urethane derivatives	*N.A.*	$C_{71}H_{124}N_2O_{17}$		*Tentative*	2979.17	LCMS	UV
Cyclic poly butylene glycol	*N.A.*	$C_{52}H_{104}O_{13}$		*Confident*	619.05	LCMS	CAD
Siloxane	*N.A.*	$C_{18}H_{56}O_{10}Si_9$		*Confident*	1005.95	LCMS	CAD
Dicyclic Siloxane	*N.A.*	$C_{14}H_{42}O_9Si_8$	*N.A.*	*Confident*	1470.24	LCMS	CAD
Siloxane	*N.A.*	$C_{16}H_{42}O_{12}Si_6$	*N.A.*	*Confident*	72931.55	LCMS	CAD
Siloxane	*N.A.*	$C_{20}H_{48}O_{18}Si_6$	*N.A.*	*Confident*	4139.88	LCMS	CAD
Siloxane	*N.A.*	$C_{23}H_{56}O_{20}Si_8$	*N.A.*	*Confident*	1160.71	LCMS	CAD
Cyclic Siloxanes	*N.A.*	$C_{2n}H_{6n}O_nSi_n$	*N.A.*	*Confident*	45151.79	LCMS	CAD
Cyclotetrasiloxane, octamethyl-	556-67-2	$C_8H_{24}O_4Si_4$		*Confident*	51.46	GCMS	GCMS
Cyclic Siloxanes other than D4	*N.A.*	*N.A.*	*N.A.*	*Confident*	54553.79	GCMS	GCMS
Linear Siloxanes	*N.A.*	*N.A.*	*N.A.*	*Confident*	280178.18	GCMS	GCMS
Butylated Hydroxytoluene	128-37-0	$C_{15}H_{24}O$		*Confident*	1071	GCMS	GCMS

Sample Preparation & Gravimetric Analysis

Materials	CAS	Manufacturer	Lot (Expiration)
Ethanol (EtOH)	64-17-5	Pharmco	Lot C15F09002-00RE200-FB; Batch WO103588
Water (H_2O)	7732-18-5	Jordi Labs (ID 917)	
Hexane	110-54-3	Pharmco	C15A22DRM-000HX95
Sodium Chloride (NaCl)	7647-14-5	Sigma	Lot MKBF1522V

Polymer sheets of PDMS, TPU and PC were prepared for each polymer with a thickness of not less than 0.5 mm, and then were cut into approximately 8 cm × 8 cm pieces. Several pieces of each polymer sheet was used to achieve a total surface area of approximately 500 cm^2 for each polymer, and approximately 500 mL of extraction solvent was added to the mixture of three polymers, to achieve an extraction ratio of 3 cm^2/mL:

$$\frac{500\ cm^2\ (of\ PDMS) + 500\ cm^2\ (of\ TPU) + 500\ cm^2 (of\ PC)}{500\ mL\ (solvent)} = 3\ cm^2/mL$$

The extraction vessels were closed and placed in ovens. Control extracts were prepared using the same lot and approximate volume of solvent used for the respective extractions. The water and saline extractions were performed at 70 °C. The ethanol and hexane extractions were performed at 50 °C. All extractions were allowed to continue for 72±2 hours with shaking at 50 rpm. Following cooling of the extractions, an aliquot (50 mL for hexane extract, 100 mL for ethanol, water and saline extracts) from each extract was removed. Precipitation was noted in the ethanol extract upon cooling. The particulates were filtered out of the solution, before the ethanol extract was concentrated by rotary evaporation. No particulates were observed in the remaining extracts. The concentrated samples were quantitatively transferred to a pre-massed scintillation vial and dried to a constant mass under a gentle stream of nitrogen with heating at approximately 70 ºC. Once constant mass was achieved, samples were subjected to gravimetric analysis. The gravimetric results are summarized in **Table 7**. The remainder of the extract was retained for LCMS, GCMS and HGCMS analysis.

Table 7 Gravimetric Analysis						
Solvent	Sample	Total Extract Volume (mL)	Volume Dried (mL)	Residue Mass (mg)	Total Residue in Extract (mg)[1]	Corrected Total Residue in Extract (mg)[2]
Ethanol	Polymer Mixture	540	100	113.65	613.71	611.28
	Blank	540	100	0.45	2.43	N.A.
Hexane	Polymer Mixture	520	50	83.34	866.73	866.37
	Blank	520	100	0.07	0.364	N.A.
Water	Polymer Mixture	532	100	2.21	11.76	7.13
	Blank	532	100	0.87	4.63	N.A.
Saline	Polymer Mixture	517	100	863.06	4462.04	N.A. [3]
	Blank	500	100	889.06	4445.28	N.A.

[1] $Total\ Residue\ in\ Extract = \frac{Residue\ Mass}{Volume\ Dried / Total\ Extract\ Volume}$

[2] Corrected Total Residue in Extract = Total Residue in Extract (Polymer Mixture)- Total Residue in Extract (Blank)

[3] Gravimetric analysis cannot be used to determine the mass of non-volatile extractables in this sample due to the high content of salt

Particulate Identification

Particulates were observed in the ethanol extracts. The particulates were recovered by filtration and dried under vacuum for 48 hours. 19.16 mg of insoluble particulate material was obtained from filtering the ethanol extraction solution of the polymer mixtures, and was then analyzed by FTIR.

Results

An FTIR micrograph of the insoluble particulates from the ethanol extract can be seen in **Figure 1**. It was found that the particulates were consistent with a mixture of polyurethane and polyamide. It was noted that two C=O stretches consistent with polyurethane were noted, while one C=O stretch consistent with polyamide was noted. In the region between 1570-1500 cm^{-1}, three overlapping absorbances are also observed. This likely indicates that the particulates are composed of two types of urethane and one type of polyamide. Specific absorbance assignments for the particulates are provided in **Table 8.** Further identification of the particulates including monomer type could be provided with pyrolysis mass spectroscopy (PYMS). The FTIR spectrum of the particulates can be seen in **Figure 2**.

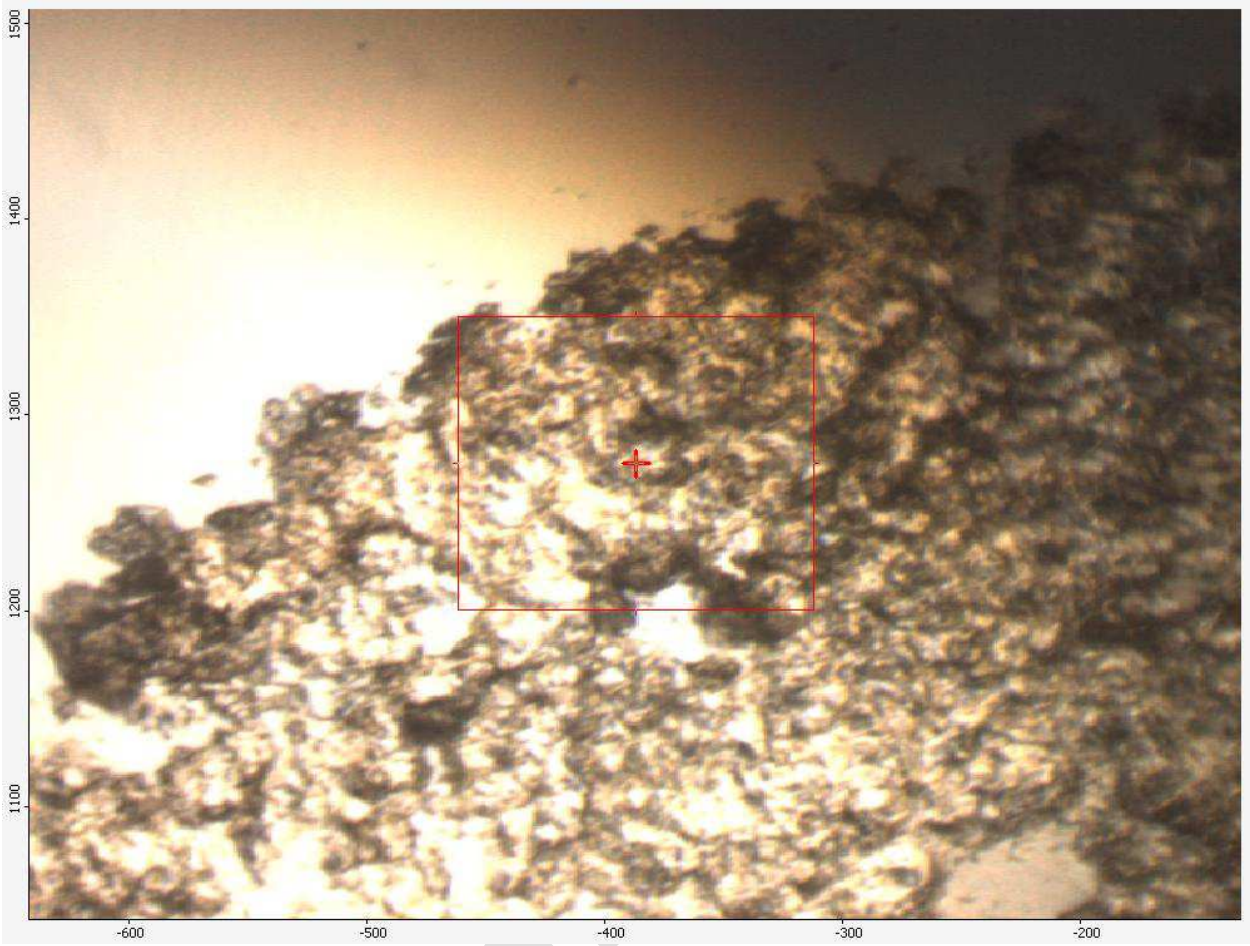


Figure 1 FTIR Micrograph of the insolubles

Table 8 *FTIR Results*		
IR Frequency (cm^{-1})	**Functional Group**	**Possible source**
3302, 3084	NH stretch	Polyurethane, polyamide
2955, 2918, 2850	CH stretch	Polyurethane, polyamide
1731, 1702	C=O stretch	Polyurethane
1636	C=O stretch (Amide I)	Polyamide
1564, 1549, 1536	N-H bend , N-C-O stretch (Amide II)	Polyurethane, polyamide
1471, 1463, 1448, 1414	CH_2 bend	Polyurethane
1377	CH_3 bend	Polyurethane
1311	C-O stretch	Polyurethane, polyamide
1249	C-N stretch	Polyamide
1224	N-(C=O)-O stretch, asymmetric	Polyurethane
1110	C-O-C stretch	Polyurethane
1019, 944	N-(C=O)-O stretch, symmetric	Polyurethane
817	NH rock	Polyurethane, polyamide
742, 728, 720	CH_2 rock	Polyurethane

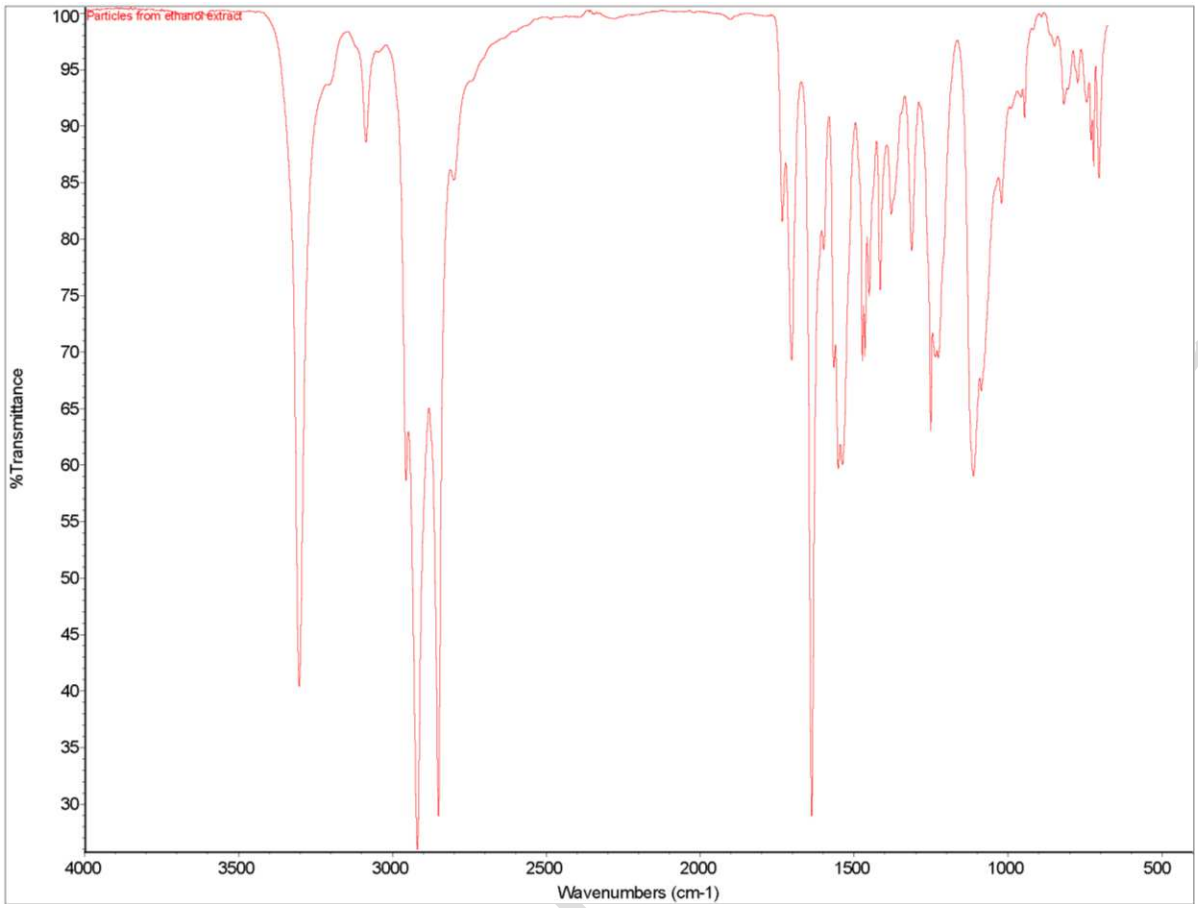


Figure 2. FTIR spectrum of the particulates from ethanol extract

MS Data Interpretation

Mass spectral identifications are based on comparison with the NIST spectral library of over 796,613 compounds as well as Jordi proprietary databases. Manual data review has also been conducted to confirm the database identifications. This includes a review of the predominate ions in the mass spectrum of each unknown followed by confirmation that these ions are also observed in the database spectra. Ion intensity ratios are also considered when evaluating the match quality as appropriate.

If high mass accuracy data is available (QTOF-GCMS or QTOF-LCMS), molecular formula generation (MFG) can be conducted to determine the best matching elemental composition for the individual ions. MSMS spectra are also examined to aid in identification.

A rating of confirmed, confident, tentative or unknown has been assigned (based on guidance provided by USP 1663) to each identification to provide an indication of the confidence level associated with a given identification. As stated in USP 1663, "Given the number and chemical diversity of organic extractables, it is unreasonable to expect that authentic reference compounds will be available (or can be made available) to confirm every identification. It is therefore necessary that levels of identification confidence be established and appropriately utilized. Data

typically available from GC/MS and LC/MS analyses (see A through E below) are used to designate individual extractables identifications in the categories of Confirmed, Confident, or Tentative:"

a) Mass spectrometric fragmentation behavior (MSMS)
b) Confirmation of molecular weight
c) Confirmation of elemental composition
d) Mass spectrum matches automated library or literature spectrum
e) Mass spectrum and chromatographic retention index match authentic reference compound

A Confirmed identification means that A, B (or C), and D (or E) have been fulfilled. A confident identification means that a combination of D with any of A, B, or C can be used. A Tentative identification means that data have been obtained that are consistent with a class of molecule only. An Unknown identification means that the class of molecule cannot be identified based on the data obtained.

QTOF LCMS

Background: QTOF-LCMS combines high mass accuracy time of flight mass spectroscopy with the power of a liquid chromatography separation to provide detailed information about the elemental composition of unknowns.

The presence of an additional quadrupole mass spectrometer (Q) provides the added capability to perform fragmentation experiments. This increases the confidence of unknown identification. It is preferable that a standard of the suspected unknown be analyzed under identical conditions as the sample. If the fragmentation patterns, high accuracy mass data, isotope patterns and LC retention times match for the unknown and standard then there is a very high probability that the identification is correct. It is possible to gain significant information about the structure of an unknown, even in cases in which standards are not available by using the molecular formula generation (MFG) algorithms contained in the Mass Hunter qualitative software.

LCMS requires that the molecule of interest be ionized. Thus, data is typically plotted in positive and negative modes indicating the charge on the ions. Ion formation is accomplished through the formation of a molecular adduct using a charge carrying species. Typical charge carriers in positive ion mode include H^+, Na^+, K^+, NH_4^+ etc. Thus the observed mass is typically the mass of the compound plus the mass of the charge carrier.

The nature of the mobile phase and the ionization conditions determine the ions formed. In negative ion, the loss of hydrogen is generally observed which results in the loss of one mass unit (1.0078 amu). Other transformations are also possible including dehydration, dimer formation, etc.

A number of plots are used to aid in interpreting QTOF-LCMS data. This includes Base Peak Chromatograms (BPC), Extracted Ion Chromatograms (EIC), Extracted Compound Chromatogram (ECC), Mass spectra (MS) and Product Ion Spectra (MSMS). A BPC is formed

by plotting the most intense ion at a given retention time. This spectrum is particularly useful for identifying the retention time of unknowns. EICs are formed by plotting a single mass at all retention times. This could be considered a plot of peak intensity (~compound concentration) for a single compound (and its isomers) versus retention time. ECC's are the sum of all the ions determined to be related to a single compound.

MS spectra plot the observed masses and their intensities at a single retention time. MS/MS spectra show the fragmentation pattern for a single compound. Mass Spectra plot the mass to charge ratio (m/z) and not the mass of the compound.

All structures indicated represent best estimates based on the data observed. In most cases the MS/MS fragmentation spectra have been consulted briefly to aid in identification of possible structures.

Sample Preparation

The water and saline extracts and blanks were concentrated 10 times before being analyzed by QTOF-LCMS. The ethanol extract was analyzed as prepared and was filtered before being analyzed. The hexane extract was dried and reconstituted in an equal volume of 80/20 methanol/isopropanol (*v/v*) solvent prior to analysis. Hexane and ethanol extracts were not concentrated due to the mass of extractables observed in gravimetric analysis.

Results

Tables 9-12 provide a summary of the LCMS results for the sample extracts in ethanol, hexane, water, and saline, respectively. **Figure 3-10** provide overlays of the base peak chromatograms (BPCs) obtained in positive and negative ionization modes, respectively.

Table 9
Summary of LCMS Results
Ethanol Extracts

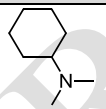
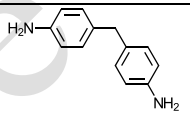
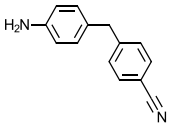
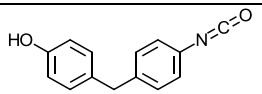
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
0.343	128.1431		127.1359	$C_8 H_{17} N$	87.3	1.71	 *N,N*-Dimethylcyclohexanamine	*Tentative*	0.12	3.01	1625.15	CAD
0.397	199.1229		198.1155	$C_{13} H_{14} N_2$	94.66	1.16	 4,4′-methylenedianiline	*Confirmed*	0.02	0.61	330.54	UV
2.065	209.1072		208.0999	$C_{14} H_{12} N_2$	84.45	0.48	 *p*-[(*p*-Aminophenyl)methyl]benzonitrile	*Tentative*	0.11	2.75	1487.42	UV
2.843	243.1128		225.0795	$C_{14} H_{11} N O_2$	98.55	-2.43	 p-[(p-Isocyanatophenyl)methyl]phenol	*Tentative*	0.32	8.16	4407.18	UV

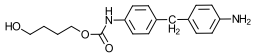
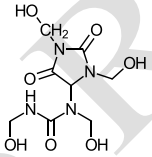
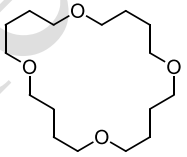
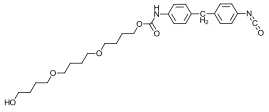
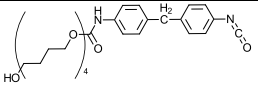
Table 9 Summary of LCMS Results Ethanol Extracts												
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
2.843	337.1524		314.1632	C_{18} H_{22} N_2 O_3	99.37	-0.37	 Poly butylene glycol urethane derivatives	*Tentative*	0.13	3.42	1845.51	UV
3.977	279.0934		278.0862	C_8 H_{14} N_4 O_7	84.78	0.2	 Diazolidinyl urea	*Tentative*	0.11	2.91	1570.06	CAD
4.154	311.2799 306.2641		288.2304	C_{16} H_{32} O_4	96.1	-1.02	 Cyclic poly butylene glycol	*Confident*	0.19	4.95	2671.85	CAD
4.342	507.2464 502.2911		484.2573	C_{27} H_{36} N_2 O_6	99.18	0.17	 Poly butylene glycol urethane derivatives	*Tentative*	0.07	1.73	936.53	UV
4.552	383.2760 378.3217		360.2878	C_{20} H_{40} O_5	98.06	-0.72	Cyclic poly butylene glycol	*Confident*	1.20	30.50	16471.84	CAD
4.596	574.3487 579.3041		556.3148	C_{31} H_{44} N_2 O_7	98.89	0.01	 Poly butylene glycol	*Tentative*	0.39	10.00	5398.80	UV

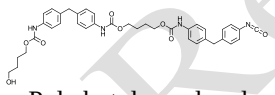
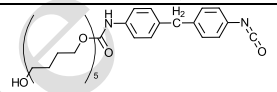
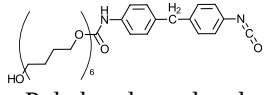
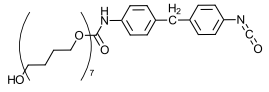
Table 9 Summary of LCMS Results Ethanol Extracts												
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
							urethane derivatives					
4.629	703.2733 698.3182		680.2844	C_{38} H_{40} N_4 O_8	99.1	0.32	 Poly butylene glycol urethane derivatives	*Tentative*	2.92	74.52	40243.08	UV
4.831	646.4066 629.3798 651.3621		628.3727	C_{35} H_{52} N_2 O_8	98.73	-0.53	 Poly butylene glycol urethane derivatives	*Tentative*	0.12	3.11	1680.24	UV
4.898	433.3523 455.3339, 450.3793		432.3449	C_{24} H_{48} O_6	97.68	0.35	Cyclic poly butylene glycol	*Confident*	1.19	30.45	16444.30	CAD
5.032	701.4374, 723.4197, 718.4643		700.4304	C_{39} H_{60} N_2 O_9	98.04	-0.7	 Poly butylene glycol urethane derivatives	*Tentative*	0.18	4.54	2451.50	UV
5.127	527.3912, 522.4366		504.4027	C_{28} H_{56} O_7	98.05	-0.25	Cyclic poly butylene glycol	*Confident*	0.27	6.99	3773.65	CAD
5.189	773.4950, 795.4768, 790.5214		772.4875	C_{43} H_{68} N_2 O_{10}	99.01	-0.14	 Poly butylene glycol urethane derivatives	*Tentative*	0.59	14.95	8070.65	UV

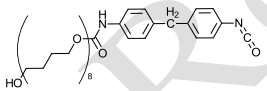
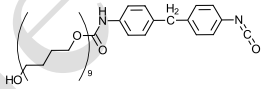
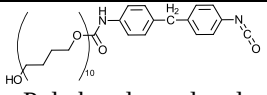
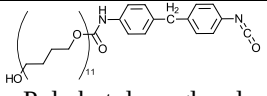
Table 9 Summary of LCMS Results Ethanol Extracts												
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
5.313	577.4669, 599.4489, 594.4940		576.4601	C_{32} H_{64} O_8	97.81	-0.04	Cyclic poly butylene glycol	*Confident*	0.19	4.79	2589.22	CAD
5.321	845.5526, 867.5347, 862.5789		844.5451	C_{47} H_{76} N_2 O_{11}	99.36	-0.23	 Poly butylene glycol urethane derivatives	*Tentative*	0.78	19.89	10742.51	UV
5.429	917.6094, 939.5916, 934.6364		916.6026	C_{51} H_{84} N_2 O_{12}	99.4	-0.22	 Poly butylene glycol urethane derivatives	*Tentative*	0.04	0.97	523.35	UV
5.454	649.5242, 671.5066, 666.5518		648.5179	C_{36} H_{72} O_9	98.49	-0.36	Cyclic poly butylene glycol	*Confident*	0.83	21.07	11376.04	CAD
5.532	989.6664, 1011.6478, 1006.6937		988.66	C_{55} H_{92} N_2 O_{13}	99.1	-0.06	 Poly butylene glycol urethane derivatives	*Tentative*	0.25	6.38	3443.11	UV
5.575	743.5643, 738.6089		720.5749	C_{40} H_{80} O_{10}	99.43	0.3	Cyclic poly butylene glycol	*Confident*	0.37	9.33	5040.71	CAD
5.618	1083.7051, 1078.7519		1060.7173	C_{59} H_{100} N_2 O_{14}	97.95	0.13	 Poly butylene glycol urethane derivatives	*Tentative*	0.17	4.34	2341.32	UV
5.679	815.6208,		792.6321	C_{44} H_{88} O_{11}	98.95	0.73	Cyclic poly butylene	*Confident*	0.07	1.73	936.53	CAD

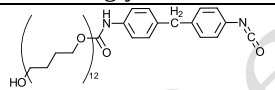
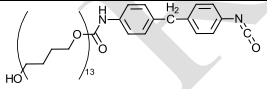
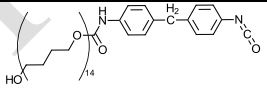
Table 9 Summary of LCMS Results Ethanol Extracts												
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
	810.6659						glycol					
5.695	1155.7610, 1150.8076		1132.7734	$C_{63}H_{108}N_2O_{15}$	98.07	1.39	 Poly butylene glycol urethane derivatives	*Tentative*	0.55	14.08	7602.39	UV
5.758	1222.8647, 620.4502		1204.8302	$C_{67}H_{116}N_2O_{16}$	94.09	1.91	 Poly butylene glycol urethane derivatives	*Tentative*	0.40	10.20	5508.98	UV
5.758	887.6781, 882.7245		864.6903	$C_{48}H_{96}O_{12}$	98.51	-0.13	Cyclic poly butylene glycol	*Confident*	0.10	2.55	1377.24	CAD
5.819	661.4329		1276.8897	$C_{71}H_{124}N_2O_{17}$	97.69	0.24	 Poly butylene glycol urethane derivatives	*Tentative*	0.15	3.88	2093.41	UV
5.836	959.7352, 954.7822		936.7479	$C_{52}H_{104}O_{13}$	98.74	-0.23	Cyclic poly butylene glycol	*Confident*	0.19	4.90	2644.31	CAD
6.222	707.1693, 723.1436, 702.2136		684.1802	$C_{18}H_{56}O_{10}Si_9$	75.64	-0.72	Siloxane	*Tentative*	0.17	4.34	2341.32	CAD
6.222	579.1058		578.0986	$C_{14}H_{42}O_9Si_8$	79.68	-0.44	Dicyclic Siloxane	*Tentative*	3.93	100.18	54098.16	CAD
6.215		593.1227	594.1298	$C_{16}H_{42}O_{12}Si_6$	81.56	-1.01	Siloxane	*Tentative*	0.12	3.16	1707.78	CAD

Table 9
Summary of LCMS Results
Ethanol Extracts

RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
6.411		743.1393	744.1467	$C_{20}H_{48}O_{18}Si_6$	74.95	-0.78	Siloxane	*Tentative*	0.37	9.49	5123.35	CAD
6.493		875.1452	876.1523	$C_{23}H_{56}O_{20}Si_8$	72.69	-0.43	Siloxane	*Tentative*	0.04	1.12	605.99	CAD
6.222-10.00	536.1659, 610.1846, 684.2040, 758.2234, 832.2427, 1500.4097, 1574.4291			$C_{2n}H_{6n}O_nSi_n$			Cyclic Siloxanes	*Tentative*	0.31	7.91	4269.46	CAD

[1] $\text{Estimated Conc. } (\mu g/mL) = \frac{\text{Peak Area (Sample)} \times \text{Standard Conc.(5.00 } \mu g/mL)}{\text{Peak Area (Standard)}}$

[2] $\text{Mass per device (ug)} = \frac{\text{Estimated Conc. } (\mu g/mL) \times \text{Total Extract Vol. (540 mL)}}{\text{Concentration Factor (10)} \times \text{Number of Devices (1)}}$

Table 10
Summary of LCMS Results
Hexane Extracts

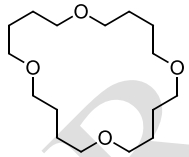
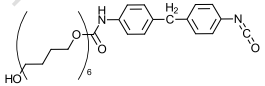
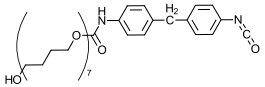
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
4.154	311.2799 306.2641		288.2304	C_{16} H_{32} O_4	96.1	-1.02	 Cyclic poly butylene glycol	*Confident*	0.17	7.37	3830.36	CAD
4.552	383.2760 378.3217		360.2878	C_{20} H_{40} O_5	98.06	-0.72	Cyclic poly butylene glycol	*Confident*	0.12	5.28	2747.02	CAD
4.898	433.3523 455.3339, 450.3793		432.3449	C_{24} H_{48} O_6	97.68	0.35	Cyclic poly butylene glycol	*Confident*	0.04	1.71	889.88	CAD
5.032	701.4374, 723.4197, 718.4643		700.4304	C_{39} H_{60} N_2 O_9	98.04	-0.7	 poly butylene glycol urethane derivatives	*Tentative*	0.14	6.03	3133.93	UV
5.127	527.3912, 522.4366		504.4027	C_{28} H_{56} O_7	98.05	-0.25	Cyclic poly butylene glycol	*Confident*	0.22	9.75	5068.45	CAD
5.189	773.4950, 795.4768, 790.5214		772.4875	C_{43} H_{68} N_2 O_{10}	99.01	-0.14	 Poly butylene glycol urethane derivatives	*Tentative*	0.78	34.67	18029.76	UV
5.313	577.4669, 599.4489, 594.4940		576.4601	C_{32} H_{64} O_8	97.81	-0.04	Cyclic poly butylene glycol	*Confident*	0.01	0.60	309.52	CAD

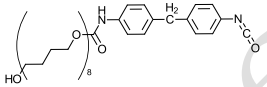
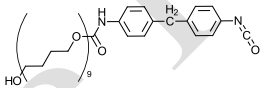
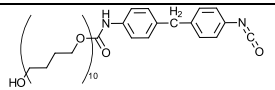
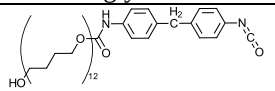
Table 10 Summary of LCMS Results Hexane Extracts												
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
5.321	845.5526, 867.5347, 862.5789		844.5451	$C_{47}H_{76}N_2O_{11}$	99.36	-0.23	 poly butylene glycol urethane derivatives	*Tentative*	0.02	0.67	348.21	UV
5.429	917.6094, 939.5916, 934.6364		916.6026	$C_{51}H_{84}N_2O_{12}$	99.4	-0.22	 poly butylene glycol urethane derivatives	*Tentative*	0.07	3.05	1586.31	UV
5.454	649.5242, 671.5066, 666.5518		648.5179	$C_{36}H_{72}O_9$	98.49	-0.36	Cyclic poly butylene glycol	*Confident*	0.54	24.03	12497.02	CAD
5.532	989.6664, 1011.6478, 1006.6937		988.66	$C_{55}H_{92}N_2O_{13}$	99.1	-0.06	 poly butylene glycol urethane derivatives	*Tentative*	0.11	4.69	2437.50	UV
5.575	743.5643, 738.6089		720.5749	$C_{40}H_{80}O_{10}$	99.43	0.3	Cyclic poly butylene glycol	*Confident*	0.04	1.79	928.57	CAD
5.679	815.6208, 810.6659		792.6321	$C_{44}H_{88}O_{11}$	98.95	0.73	Cyclic poly butylene glycol	*Confident*	0.05	2.23	1160.71	CAD
5.695	1155.7610, 1150.8076		1132.7734	$C_{63}H_{108}N_2O_{15}$	98.07	1.39	 poly butylene glycol urethane derivatives	*Tentative*	0.04	1.86	967.26	UV

Table 10
Summary of LCMS Results
Hexane Extracts

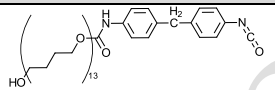
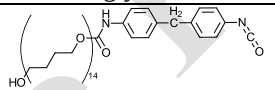
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
5.758	1222.8647, 620.4502		1204.8302	$C_{67}H_{116}N_2O_{16}$	94.09	1.91	 poly butylene glycol urethane derivatives	*Tentative*	0.06	2.53	1315.48	UV
5.758	887.6781, 882.7245		864.6903	$C_{48}H_{96}O_{12}$	98.51	-0.13	Cyclic poly butylene glycol	*Confident*	0.03	1.26	657.74	CAD
5.819	661.4329		1276.8897	$C_{71}H_{124}N_2O_{17}$	97.69	0.24	 poly butylene glycol urethane derivatives	*Tentative*	0.13	5.73	2979.17	UV
5.836	959.7352, 954.7822		936.7479	$C_{52}H_{104}O_{13}$	98.74	-0.23	Cyclic poly butylene glycol	*Confident*	0.03	1.19	619.05	CAD
6.222	707.1693, 723.1436, 702.2136		684.1802	$C_{18}H_{56}O_{10}Si_9$	75.64	-0.72	Siloxane	*Tentative*	0.04	1.93	1005.95	CAD
6.222	579.1058		578.0986	$C_{14}H_{42}O_9Si_8$	79.68	-0.44	Dicyclic Siloxane	*Tentative*	0.06	2.83	1470.24	CAD
6.215		593.1227	594.1298	$C_{16}H_{42}O_{12}Si_6$	81.56	-1.01	Siloxane	*Tentative*	3.14	140.25	72931.55	CAD
6.411		743.1393	744.1467	$C_{20}H_{48}O_{18}Si_6$	74.95	-0.78	Siloxane	*Tentative*	0.18	7.96	4139.88	CAD
6.493		875.1452	876.1523	$C_{23}H_{56}O_{20}Si_8$	72.69	-0.43	Siloxane	*Tentative*	0.05	2.23	1160.71	CAD
6.222-10.00	536.1659, 610.1846,			$C_{2n}H_{6n}O_nSi_n$			Cyclic Siloxanes	*Tentative*	1.95	86.83	45151.79	CAD

Table 10
Summary of LCMS Results
Hexane Extracts

RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
	684.2040, 758.2234, 832.2427, 1500.4097, 1574.4291											

[1] $\text{Estimated Conc. } (\mu g/mL) = \frac{\text{Peak Area (Sample)} \times \text{Standard Conc.(5.00 } \mu g/mL)}{\text{Peak Area (Standard)}}$

[2] $\text{Mass per device } (ug) = \frac{\text{Estimated Conc. } \left(\frac{\mu g}{mL}\right) \times \text{Total Extract Vol. (520 mL)}}{\text{Concentration Factor (10)} \times \text{Number of Devices (1)}}$

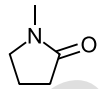
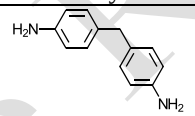
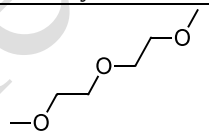
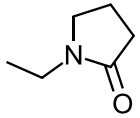
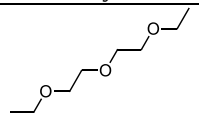
Table 11 Summary of LCMS Results Water Extracts												
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
0.37	100.0759 122.0576		99.0686	C_5 H_9 N O	99.4	-1.99	 2-Pyrrolidinone, 1-methyl-	*Confident*	0.17	3.91	208.05	CAD
0.397	199.1229		198.1155	C_{13} H_{14} N_2	94.66	1.16	 4,4′-methylenedianiline	*Confirmed*	0.06	1.49	79.43	UV
0.57	135.1017 157.0838 152.1286		134.0945	C_6 H_{14} O_3	99.35	-1.29	 2-Methoxy-1-(2-methoxyethoxy)ethane	*Confident*	0.02	0.54	28.47	CAD
0.73	114.0913		113.084	C_6 H_{11} N O	99.85	0.73	 2-Pyrrolidinone, 1-ethyl-	*Confident*	0.66	15.43	820.98	CAD
1.32	163.1329 181.155		162.1256	C_8 H_{18} O_3	98.39	-0.04	 2-Ethoxy-1-(2-ethoxyethoxy)ethane	*Confident*	0.05	1.19	63.44	CAD

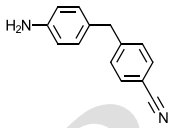
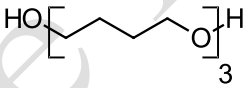
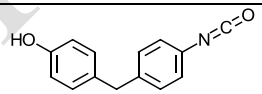
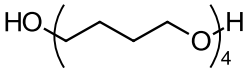
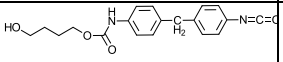
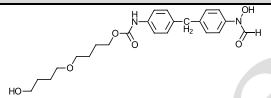
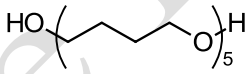
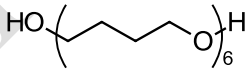
Table 11 Summary of LCMS Results Water Extracts												
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
2.065	209.1072		208.0999	$C_{14}H_{12}N_2$	84.45	0.48	 *p*-[(*p*-Aminophenyl)methyl]benzonitrile	*Tentative*	0.10	2.35	125.13	UV
2.722	235.1907		234.1834	$C_{12}H_{26}O_4$	98.21	-1.31	 Poly butylene glycol	*Confident*	0.30	6.93	368.90	CAD
2.843	243.1128		225.0795	$C_{14}H_{11}NO_2$	98.55	-2.43	 p-[(p-Isocyanatophenyl)methyl]phenol	*Tentative*	0.12	2.82	150.11	UV
3.325	307.2485		306.2413	$C_{16}H_{34}O_5$	96.86	-2.09	 Poly butylene glycol	*Confident*	0.14	3.37	179.33	CAD
3.561	341.1500 363.1321		340.1427	$C_{19}H_{20}N_2O_4$	99.23	-1.28	 Poly butylene glycol urethane derivatives	*Tentative*	0.07	1.61	85.67	UV

Table 11
Summary of LCMS Results
Water Extracts

RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
3.667	431.2179 453.2001 448.2442		430.2106	$C_{23}H_{30}N_2O_6$	99.51	-0.5	 p-({p-[N-Formyl(hydroxyamino)]phenyl}methyl)phenyl amino 5-(4-hydroxybutoxy)valerate	*Tentative*	0.02	0.39	20.73	UV
3.787	379.3062		378.299	$C_{20}H_{42}O_6$	96.67	-2.19	 Poly butylene glycol	*Confident*	0.23	5.33	283.48	CAD
4.132	451.3632		450.3559	$C_{24}H_{50}O_7$	97.7	-0.51	 Poly butylene glycol	*Confident*	0.04	1.01	53.95	CAD
4.552	383.2760 378.3217		360.2878	$C_{20}H_{40}O_5$	98.06	-0.72	Cyclic poly butylene glycol	*Confident*	0.83	19.56	1040.77	CAD
4.898	433.3523 455.3339, 450.3793		432.3449	$C_{24}H_{48}O_6$	97.68	0.35	Cyclic poly butylene glycol	*Confident*	0.03	0.75	39.71	CAD
5.127	527.3912, 522.4366		504.4027	$C_{28}H_{56}O_7$	98.05	-0.25	Cyclic poly butylene glycol	*Confident*	1.37	32.16	1710.89	CAD

[1] $\text{Estimated Conc. } (\mu g/mL) = \frac{\text{Peak Area (Sample)} \times \text{Standard Conc.(5.00 } \mu g/mL)}{\text{Peak Area (Standard)}}$

[2] $\text{Mass per device (ug)} = \frac{\text{Estimated Conc. } (\mu g/mL) \times \text{Total Extract Vol. (532 mL)}}{\text{Concentration Factor (10)} \times \text{Number of Devices (1)}}$

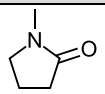
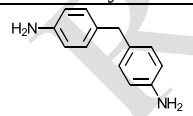
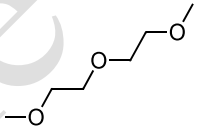
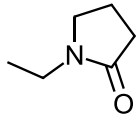
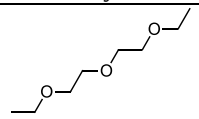
Table 12 Summary of LCMS Results Saline Extracts												
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
0.37	100.0759 122.0576		99.0686	C_5 H_9 N O	99.4	-1.99	 2-Pyrrolidinone, 1-methyl-	*Confident*	0.32	4.27	220.79	CAD
0.397	199.1229		198.1155	C_{13} H_{14} N_2	94.66	1.16	 4,4′-methylenedianiline	*Confirmed*	0.05	N.A.	N.A.	Formal Quantitation by QQQ
0.57	135.1017 157.0838 152.1286		134.0945	C_6 H_{14} O_3	99.35	-1.29	 2-Methoxy-1-(2-methoxyethoxy)ethane	*Confident*	0.51	6.69	345.84	CAD
0.73	114.0913		113.084	C_6 H_{11} N O	99.85	0.73	 2-Pyrrolidinone, 1-ethyl-	*Confident*	1.03	13.57	701.45	CAD
1.32	163.1329 181.155		162.1256	C_8 H_{18} O_3	98.39	-0.04	 2-Ethoxy-1-(2-ethoxyethoxy)ethane	*Confident*	0.47	6.16	318.48	CAD

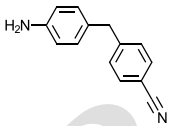
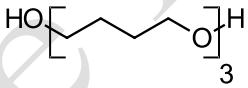
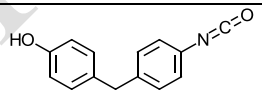
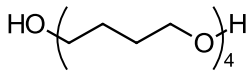
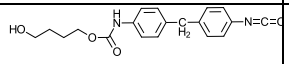
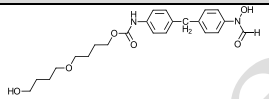
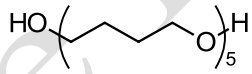
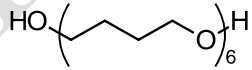
Table 12 Summary of LCMS Results Saline Extracts												
RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
2.065	209.1072		208.0999	$C_{14}H_{12}N_2$	84.45	0.48	 *p*-[(*p*-Aminophenyl)methyl]benzonitrile	*Tentative*	0.39	5.14	265.73	UV
2.722	235.1907		234.1834	$C_{12}H_{26}O_4$	98.21	-1.31	 Poly butylene glycol	*Confident*	0.51	6.69	345.84	CAD
2.843	243.1128		225.0795	$C_{14}H_{11}NO_2$	98.55	-2.43	 p-[(p-Isocyanatophenyl)methyl]phenol	*Tentative*	0.21	2.74	141.66	UV
3.325	307.2485		306.2413	$C_{16}H_{34}O_5$	96.86	-2.09	 Poly butylene glycol	*Confident*	0.80	10.64	550.02	CAD
3.561	341.1500 363.1321		340.1427	$C_{19}H_{20}N_2O_4$	99.23	-1.28	 Poly butylene glycol urethane derivatives	*Tentative*	0.50	6.59	340.95	UV

Table 12
Summary of LCMS Results
Saline Extracts

RT (min)	*Positive m/z*	*Negative m/z*	Mass	Best Match	Score	Diff.	Possible ID	Confident Level	Peak Area	Estimated Conc. (µg/mL)[1]	Mass per Device (µg)[2]	Quantification Method
3.667	431.2179 453.2001 448.2442		430.2106	$C_{23}H_{30}N_2O_6$	99.51	-0.5	 p-({p-[N-Formyl(hydroxyamino)]phenyl}methyl)phenyl amino 5-(4-hydroxybutoxy)valerate	*Tentative*	1.53	20.20	1044.36	UV
3.787	379.3062		378.299	$C_{20}H_{42}O_6$	96.67	-2.19	 Poly butylene glycol	*Confident*	0.35	4.65	240.33	CAD
4.132	451.3632		450.3559	$C_{24}H_{50}O_7$	97.7	-0.51	 Poly butylene glycol	*Confident*	3.68	48.73	2519.54	CAD
4.552	383.2760 378.3217		360.2878	$C_{20}H_{40}O_5$	98.06	-0.72	Cyclic poly butylene glycol	*Confident*	1.36	17.93	927.12	CAD
4.898	433.3523 455.3339, 450.3793		432.3449	$C_{24}H_{48}O_6$	97.68	0.35	Cyclic poly butylene glycol	*Confident*	8.03	106.24	5492.39	CAD
5.127	527.3912, 522.4366		504.4027	$C_{28}H_{56}O_7$	98.05	-0.25	Cyclic poly butylene glycol	*Confident*	1.93	25.59	1322.79	CAD

[1] $\text{Estimated Conc. } (\mu g/mL) = \frac{\text{Peak Area (Sample)} \times \text{Standard Conc.(5.00 } \mu g/mL)}{\text{Peak Area (Standard)}}$

[2] $\text{Mass per device } (ug) = \frac{\text{Estimated Conc. } (\mu g/mL) \times \text{Total Extract Vol. (517 mL)}}{\text{Concentration Factor (10)} \times \text{Number of Devices (1)}}$

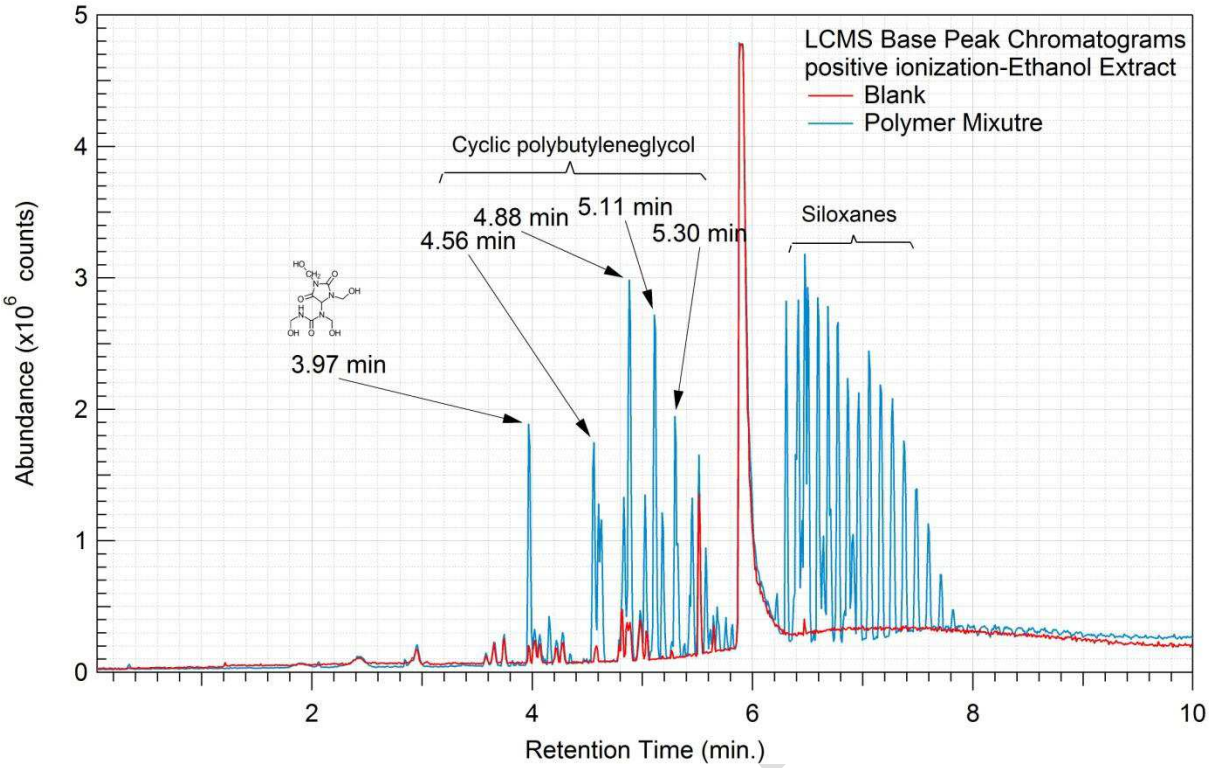


Figure 3- Overlay of LCMS base peak chromatograms of ethanol extract, positive ionization.

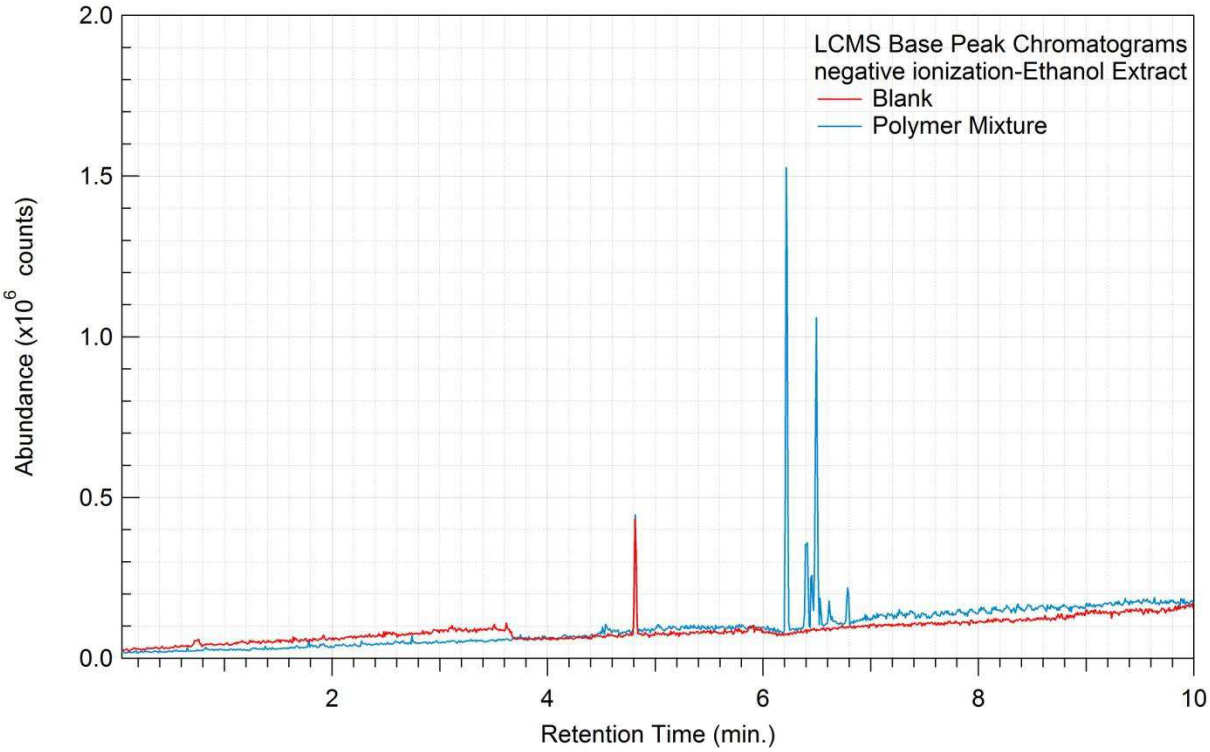


Figure 4 - Overlay of LCMS base peak chromatograms of ethanol extract, negative ionization.

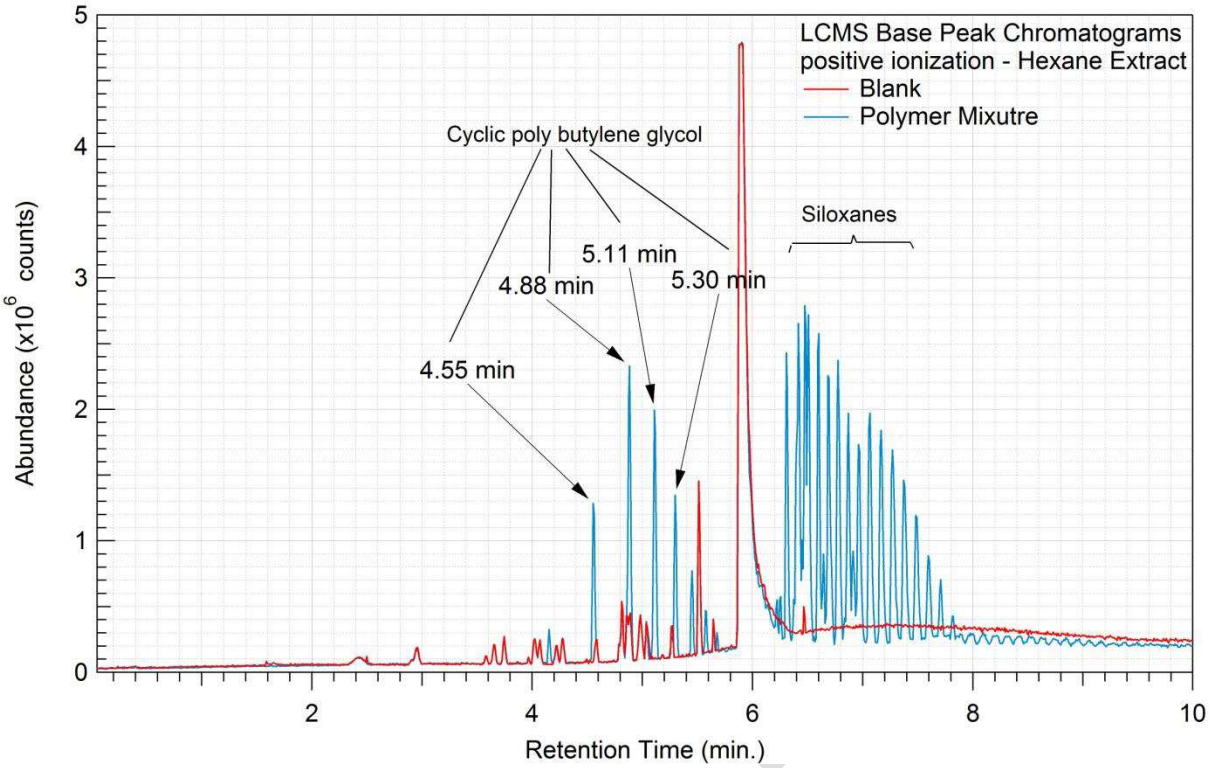


Figure 5- Overlay of LCMS base peak chromatograms of hexane extract, positive ionization.

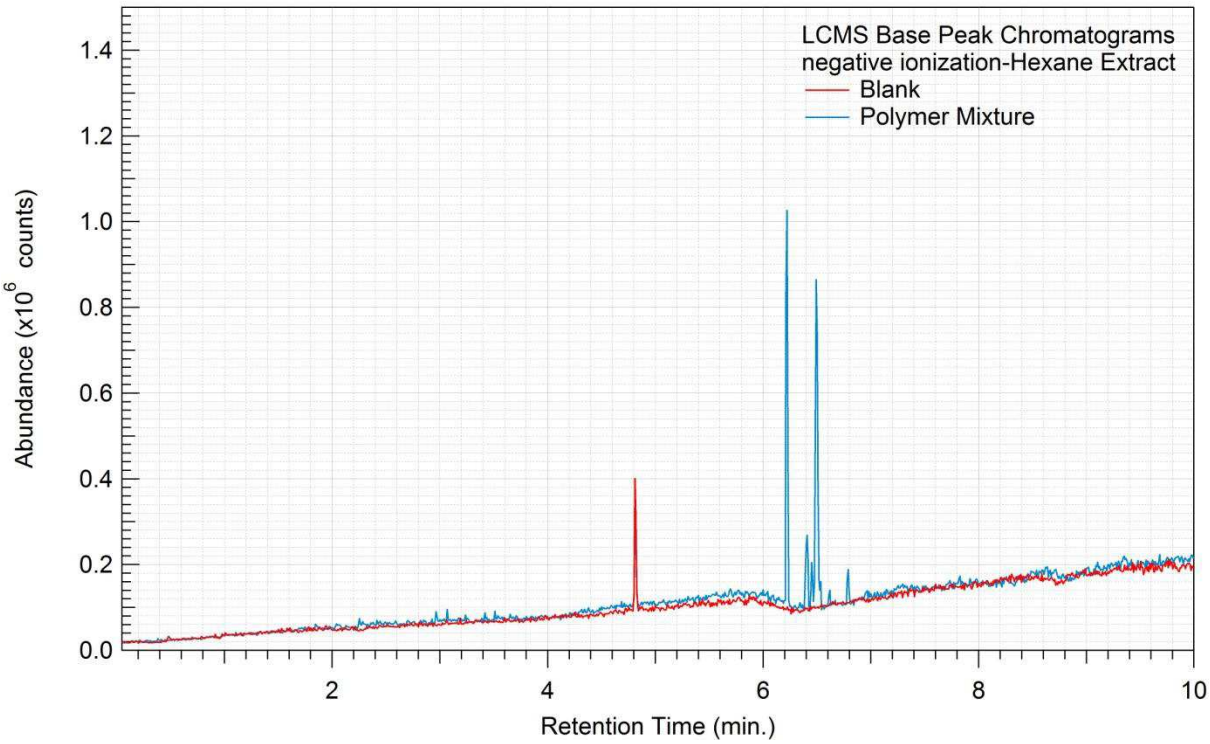


Figure 6 - Overlay of LCMS base peak chromatograms of hexane extract, negative ionization.

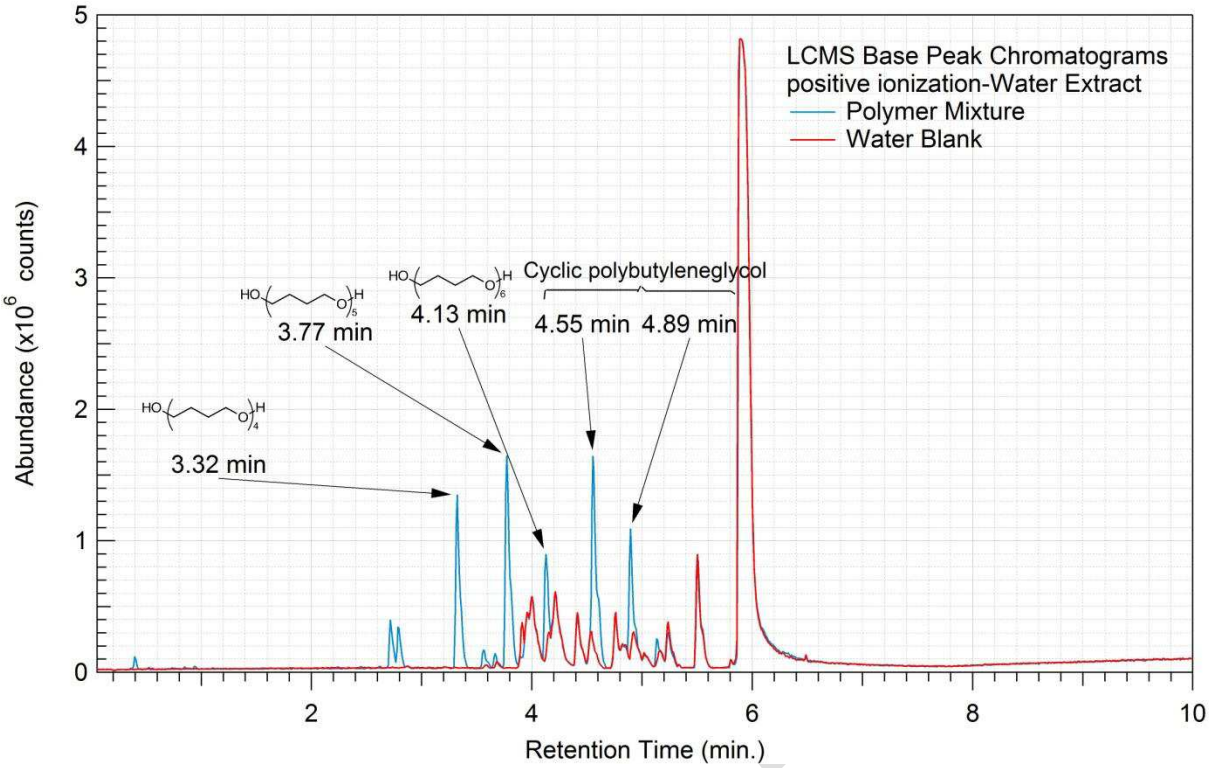


Figure 7- Overlay of LCMS base peak chromatograms of water extract, positive ionization.

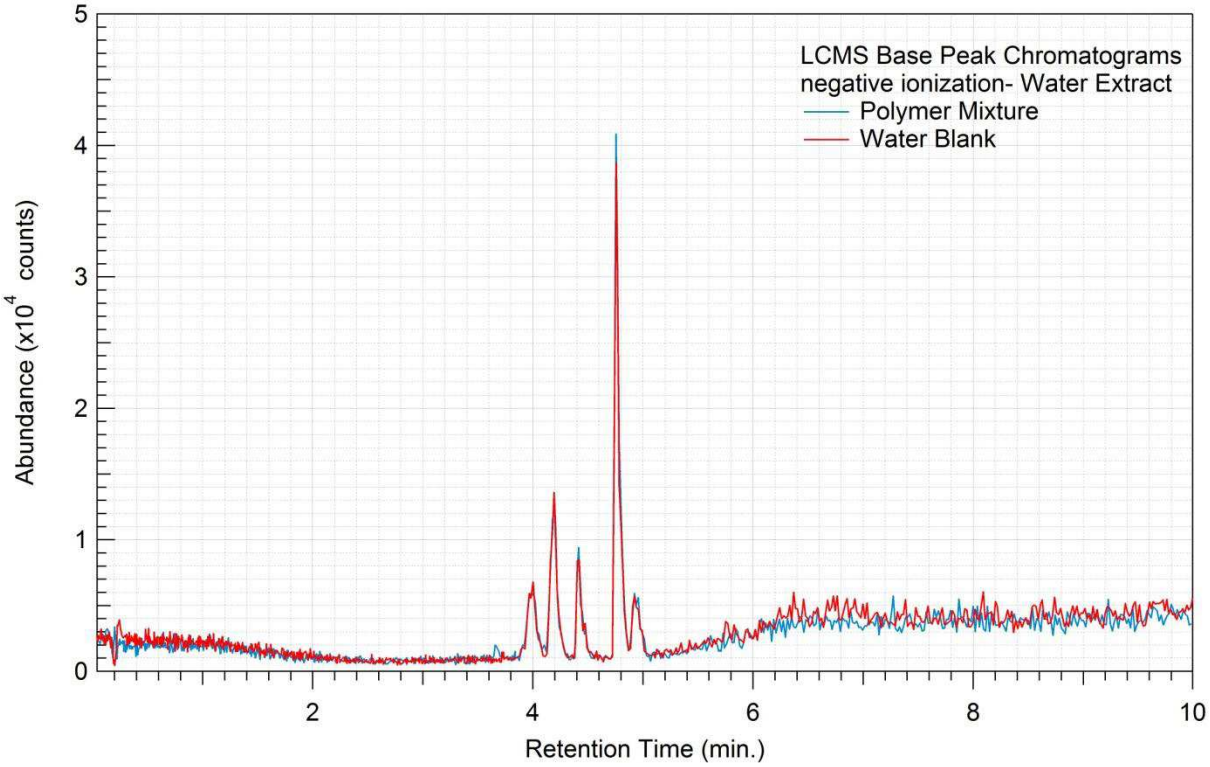


Figure 8 - Overlay of LCMS base peak chromatograms of water extract, negative ionization.

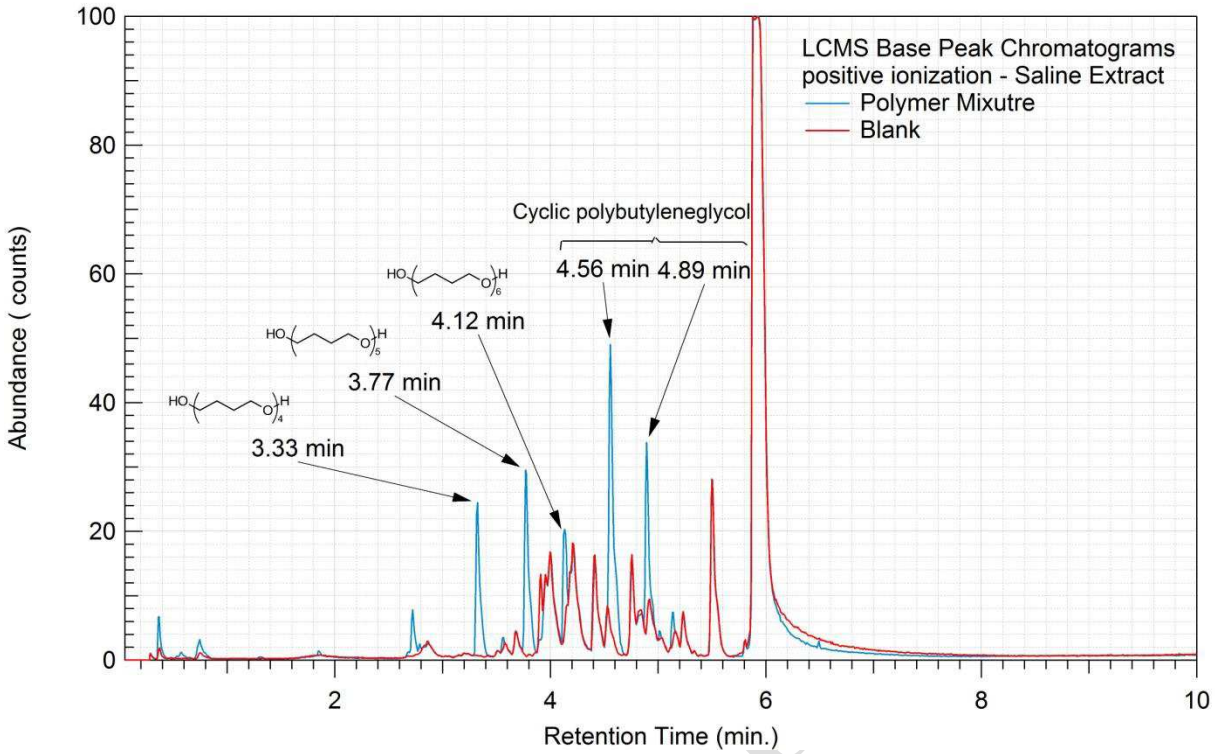


Figure 9- Overlay of LCMS base peak chromatograms of saline extract, positive ionization.

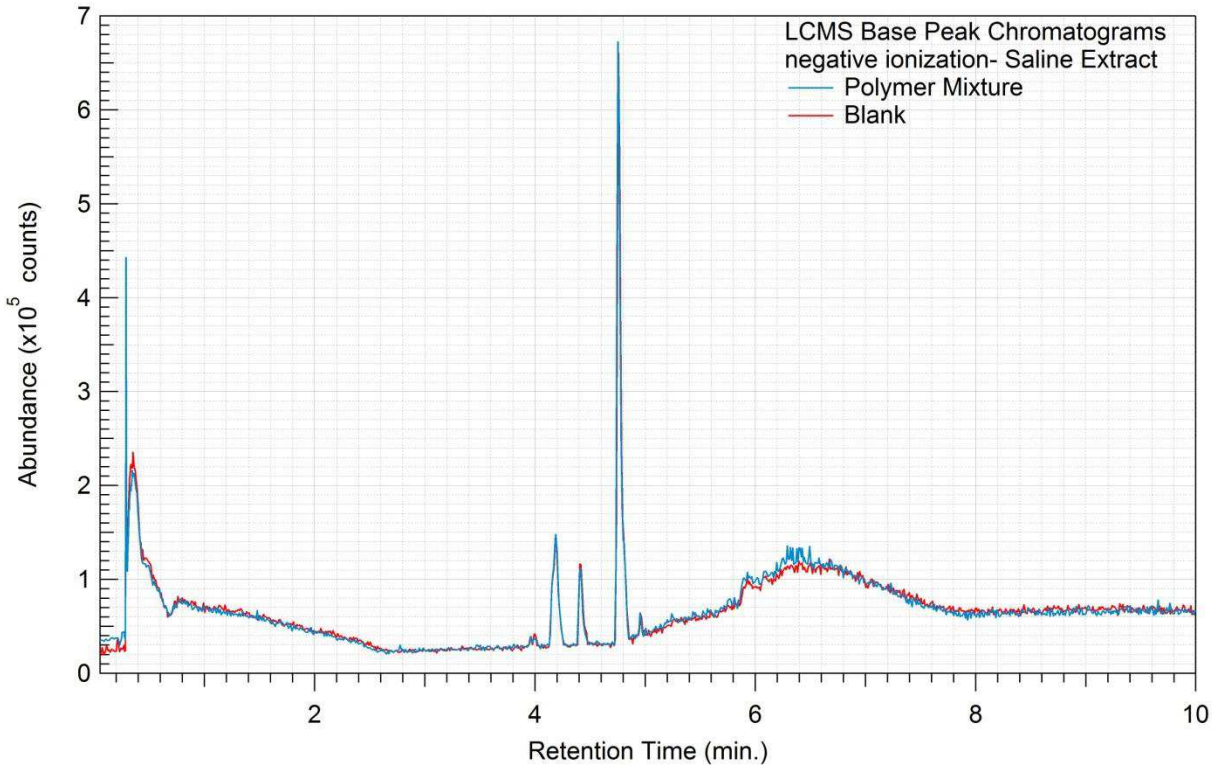


Figure 10- Overlay of LCMS base peak chromatograms of saline extract, negative ionization.

Formal Quantitation of 4,4'-methylenedianiline

The saline extract was analyzed by QQQ-LCMS to quantitate 4,4-methylenedianiline, and a five point calibration curve was prepared as shown in **Figure 11**. A 4,4-methylenedianiline standard was dissolved and diluted to known concentrations in methanol from 20 ng/mL to 500 ng/mL. Each standard was injected in duplicate prior to and following the samples, and the averages of all four injections were used in constructing the calibration curve. The resulting equation for the linear regression line and the R^2 value for the line are included in the figures. The LOQ for the method was found to be 20 ng/ml.

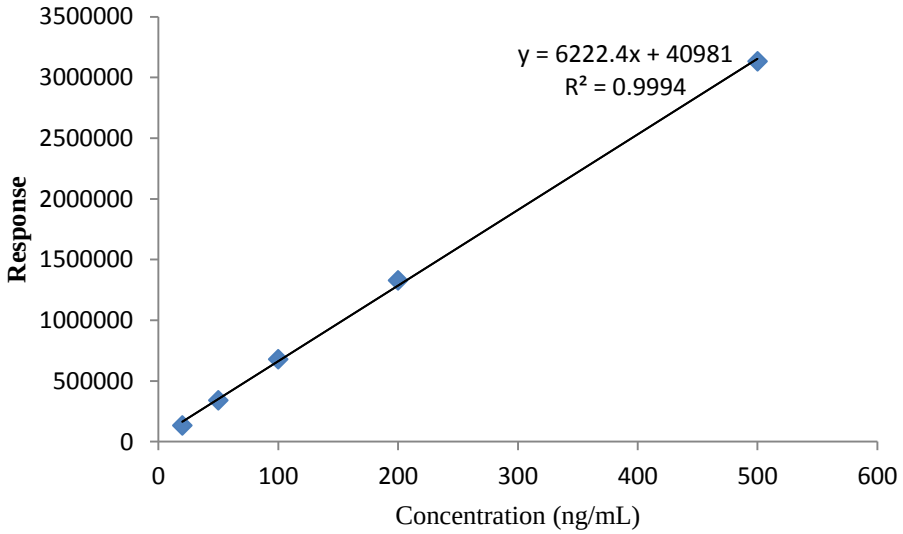


Figure 11. Calibration Curve for 4,4-methylenedianiline

The saline extract was prepared as detailed in the LCMS analysis section. For the spiked sample, 20 µL of 10 µg/mL 4,4-methylenedianiline standard was added to 1 mL of the extract. The samples were analyzed in duplicate**.** Results of the samples and the sample spike are shown in **Table 13**. The spike recovery was 97.24%.

Table 13
4,4-methylenedianiline Quantitation Results

Sample	Concentration (ng/mL)[1]		Average Concentration (ng/mL)	Spike Recovery[2]	Mass per Device (µg)[3]
Saline Extract	290.44	300.56	295.50	N.A.	15.28
Saline Extract Spike	491.80	498.16	494.98	99.74%	N.A.

[1] Concentration was calculated based on the calibration curved in **Figure 11**

[2] Spiking recovery = (Average Concentration (Spike Sample)- Average Concentration (Sample)) ÷ 200 ng/mL × 100%

[3]Mass per Device = Average Concentration × Total Volume Added (517 mL) ÷ Number of Devices (1) ÷ Concentration Factor (10) × $\frac{1\mu g}{1000\ ng}$

N.A. – Not Applicable

QTOF GCMS

GCMS analysis was performed in electron impact mode. The spectra collected using electron impact (EI) ionization can be compared to the NIST mass spectral database for identification. In addition fragments can be identified using the accurate mass data collected. This ionization mode is high energy and generally causes a large amount of analyte fragmentation. In many cases the EI mass spectra collected only contain fragment ions making definitive unknown identification impossible for compounds not present in the mass spectral database.

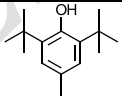
Sample Preparation

10 mL of the saline and water extracts were each extracted with 10 mL of DCM. The DCM solutions were concentrated to 1 mL under a gentle stream of nitrogen and then analyzed by QTOF-GCMS. A 100 µL aliquot of the ethanol and hexane extracts were diluted to 1 mL with the extraction solvent and analyzed by QTOF-GCMS.

Results

Tables 14-17 provide a summary of the GCMS results for the sample extracts in ethanol, hexane, water, and saline, respectively. **Figures 12-15** provide overlays of the base peak chromatograms (BPCs) obtained in positive and negative ionization modes, respectively.

Table 14
Summary of GCMS Results - Ethanol Extracts

RT (min)	Possible ID	*CAS*	*Confident Level*	Peak Area*	Estimated Conc.[1] (µg/mL)	Mass per Device[2] (µg)
12.34	Cyclotetrasiloxane, octamethyl-(D4)	556-67-2	*Confident*	28103.17	0.01	38.70
16.39-19.70	Cyclic Siloxanes other than D4	*N.A*	*Confident*	21260920.94	5.42	29281.48
10.89 15.61 19.95-29.63	Linear Siloxanes	*N.A*	*Tentative*	106304604.70	27.11	146407.42
15.39	 Butylated Hydroxytoluene	128-37-0	*Confident*	1145128.65	0.29	1577.12

Quantitation Standard Compound			
RT (min)	Possible Identification	Conc. (µg/mL)	Peak Area
12.18	Siloxane	5.00	19604363.36

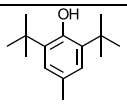
* Average of two injections

[1]Estimated Conc. $(\mu g/mL) = \frac{\text{Peak Area (Sample)} \times \text{Standard Conc.(5.00 µg/mL)}}{\text{Peak Area (Standard)}}$

[2]Mass per device $(ug) = \frac{\text{Estimated Conc. (µg/mL)} \times \text{Total Extract Vol. (540 mL)} \times \text{Dilution Factor (10)}}{\text{Number of Devices (1)}}$

N.A. – Not Applicable

Table 15
Summary of GCMS Results - Hexane Extracts

RT (min)	Possible ID	*CAS*	*Confident Level*	Peak Area*	Estimated Conc.[1] (µg/mL)	Mass per Device[2] (µg)
12.36	Cyclotetrasiloxane, octamethyl-(D4)	556-67-2	*Confident*	38805.24	0.01	51.46
16.45-19.72	Cyclic Siloxanes other than D4	*N.A*	*Confident*	41134322.20	10.49	54553.79
10.78 15.66 19.99-29.65	Linear Siloxanes	*N.A*	*Tentative*	211258262.30	53.88	280178.18
15.39	 Butylated Hydroxytoluene	128-37-0	*Confident*	807912.73	0.21	1071

Quantitation Standard Compound

RT (min)	Possible Identification	Conc. (µg/mL)	Peak Area
12.18	Siloxane	5.00	19604363.36

* Average of two injections

[1]Estimated Conc. $(\mu g/mL) = \frac{\text{Peak Area (Sample)} \times \text{Standard Conc.(5.00 µg/mL)}}{\text{Peak Area (Standard)}}$

[2]Mass per device (ug) $= \frac{\text{Estimated Conc. (µg/mL)} \times \text{Total Extract Vol. (520 mL)} \times \text{Dilution Factor (10)} \times}{\text{Number of Devices (1)}}$

N.A. – Not Applicable

Table 16
Summary of GCMS Results - Water Extracts

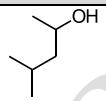
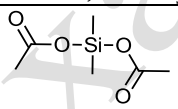
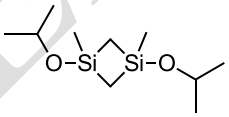
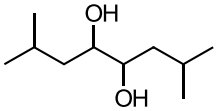
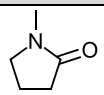
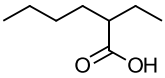
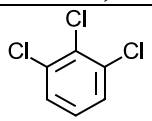
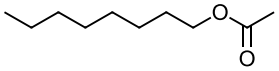
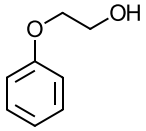
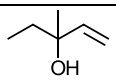
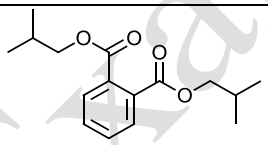
RT (min)	Possible ID	*CAS*	*Confident Level*	Peak Area*	Estimated Conc.[1] (µg/mL)	Mass per Device[2] (µg)
5.37	 2-Pentanol, 4-methyl-	108-11-2	*Tentative*	814306.73	0.21	1104.88
5.51	 Silanediol, dimethyl-, diacetate	2182-66-3	*Tentative*	785566.99	0.20	1065.89
6.10	 1,3-Diisopropoxy-1,3-dimethyl-1,3-disilacyclobutane	198066-66-9	*Tentative*	448497.40	0.11	608.54
6.47	 4,5-Octanediol, 2,7-dimethyl-	*N.A*	*Tentative*	143302.52	0.04	194.44

Table 16 Summary of GCMS Results - Water Extracts						
RT (min)	Possible ID	*CAS*	*Confident Level*	Peak Area*	Estimated Conc.[1] (µg/mL)	Mass per Device[2] (µg)
10.60	 2-Pyrrolidinone, 1-methyl-	872-50-4	*Confident*	303014.81	0.08	411.14
9.87 10.90-14.14, 15.66-29.63	Siloxanes	*N.A*	*Tentative*	25796437.48	6.58	35001.66
11.57	 Hexanoic acid, 2-ethyl-	149-57-5	*Tentative*	38357750.66	9.78	52045.36
12.28	 Benzene, 1,2,3-trichloro-	87-61-6	*Confident*	3150709.37	0.80	4275.01
12.50	 Acetic acid, octyl ester	112-14-1	*Tentative*	214105.96	0.05	290.51
12.74	 Ethanol, 2-phenoxy-	122-99-6	*Tentative*	793770.78	0.20	1077.02
13.22-15.54	Hydrocarbons	*N.A*	*Tentative*	1235540.21	0.32	1676.43
16.11, 19.55, 22.25	 1-Penten-3-ol, 3-methyl- and isomers	918-85-4	*Confident*	1375386.83	0.35	1866.18
18.06	 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester	84-69-5	*Tentative*	96072.79	0.02	130.36

Quantitation Standard Compound			
RT (min)	Possible Identification	Conc. (µg/mL)	Peak Area
12.18	Siloxane	5.00	19604363.36

* Average of two injections

[1]Estimated Conc. (µg/mL) = $\frac{\text{Peak Area (Sample)}}{\text{Peak Area (Standard)}} \times \text{Standard Conc. (5.00 µg/mL)}$

[2]Mass per device (ug) = $\frac{\text{Estimated Conc. (µg/mL)} \times \text{Total Extract Vol. (532 mL)}}{\text{Concentration Factor (0.1)} \times \text{Number of Devices (1)}}$

N.A. – Not Applicable

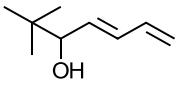
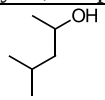
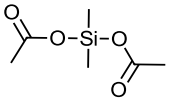
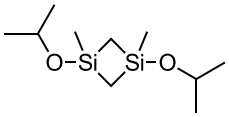
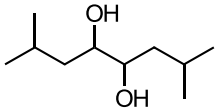
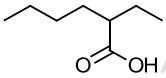
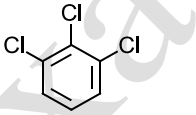
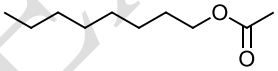
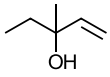
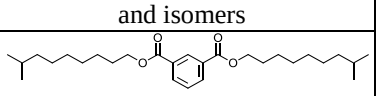
Table 17 Summary of GCMS Results - Saline Extracts						
RT (min)	Possible ID	*CAS*	*Confident Level*	Peak Area*	Estimated Conc.[1] (µg/mL)	Mass per Device[2] (µg)
4.44	 6,6-Dimethyl-1,3-heptadien-5-ol	81912-03-0	*Tentative*	286900.88	0.07	378.30
5.37	 2-Pentanol, 4-methyl-	108-11-2	*Tentative*	1006237.25	0.26	1326.81
5.51	 Silanediol, dimethyl-, diacetate	2182-66-3	*Tentative*	992720.30	0.25	1308.99
6.10	 1,3-Diisopropoxy-1,3-dimethyl-1,3-disilacyclobutane	198066-66-9	*Tentative*	167011.37	0.04	220.22
6.47	 4,5-Octanediol, 2,7-dimethyl-	*N.A*	*Tentative*	159206.05	0.04	209.93
9.87 10.90-14.14, 15.66-29.63	Siloxanes	*N.A*	*Tentative*	22289860.33	5.68	29391.05
11.57	 Hexanoic acid, 2-ethyl-	149-57-5	*Tentative*	17997310.08	4.59	23730.97
12.28	 Benzene, 1,2,3-trichloro-	87-61-6	*Confident*	3372673.18	0.86	4447.15
12.50	 Acetic acid, octyl ester	112-14-1	*Tentative*	221418.67	0.06	291.96
13.22-15.54	Hydrocarbons	*N.A*	*Tentative*	864763.75	0.22	1140.26
16.11, 19.55, 22.25	 1-Penten-3-ol, 3-methyl- and isomers	918-85-4	*Confident*	948590.17	0.24	1250.80
23.52		89-16-7	*Tentative*	340415.21	0.09	448.87

Table 17 Summary of GCMS Results - Saline Extracts						
RT (min)	Possible ID	*CAS*	*Confident Level*	Peak Area*	Estimated Conc.[1] (µg/mL)	Mass per Device[2] (µg)
	1,2-Benzenedicarboxylic acid, bis(8-methylnonyl) ester					
Quantitation Standard Compound						
RT (min)	Possible Identification	Conc. (µg/mL)	Peak Area			
12.18	Siloxane	5.00	19604363.36			

* Average of two injections

[1] $\text{Estimated Conc. } (\mu g/mL) = \frac{\text{Peak Area (Sample)}}{\text{Peak Area (Standard)}} \times \text{Standard Conc. } (5.00\ \mu g/mL)$

[2] $\text{Mass per device (ug)} = \frac{\text{Estimated Conc. } (\mu g/mL) \times \text{Total Extract Vol. } (517\ mL)}{\text{Concentration Factor } (0.1) \times \text{Number of Devices } (1)}$

N.A. – Not Applicable

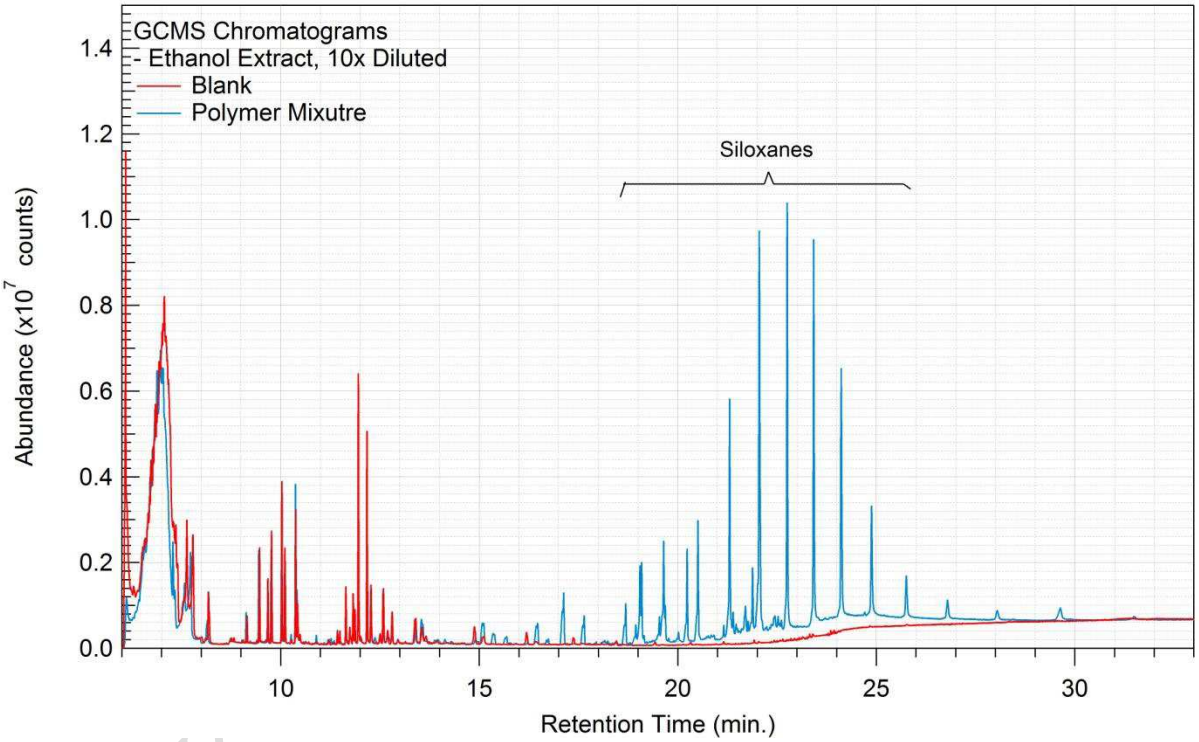


Figure 12. Overlay of GCMS chromatograms of ethanol extract

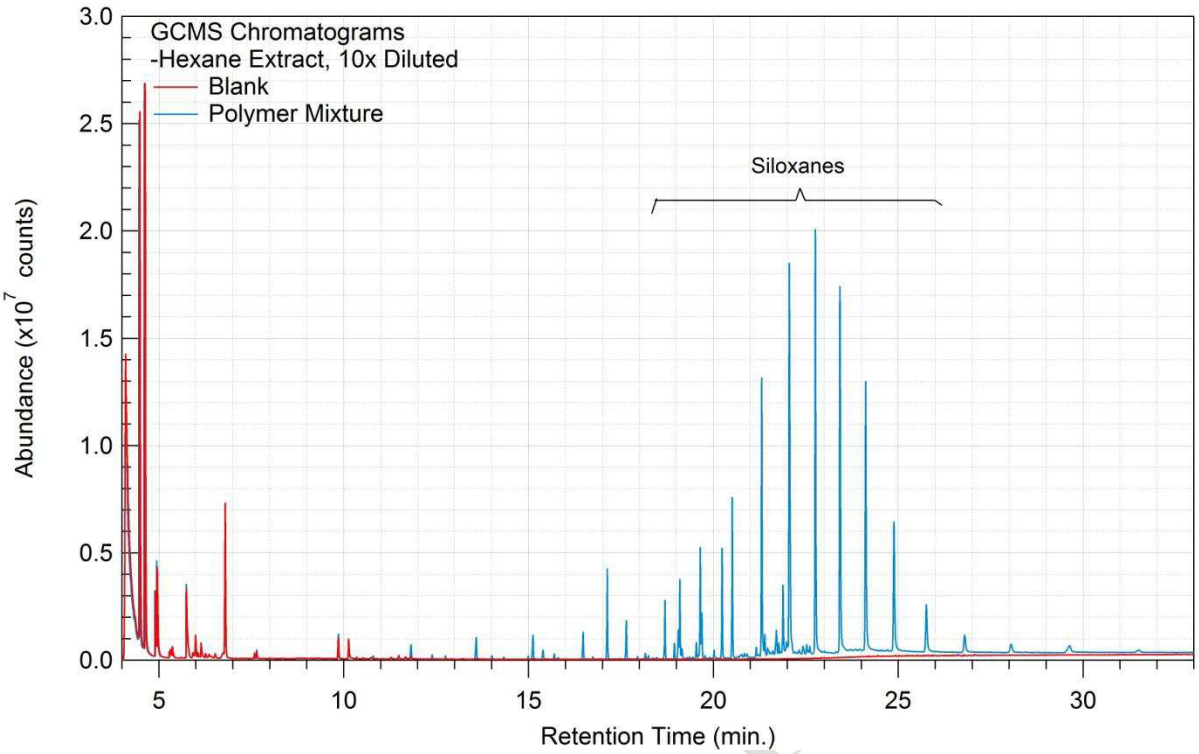


Figure 13. Overlay of GCMS chromatograms of hexane extract

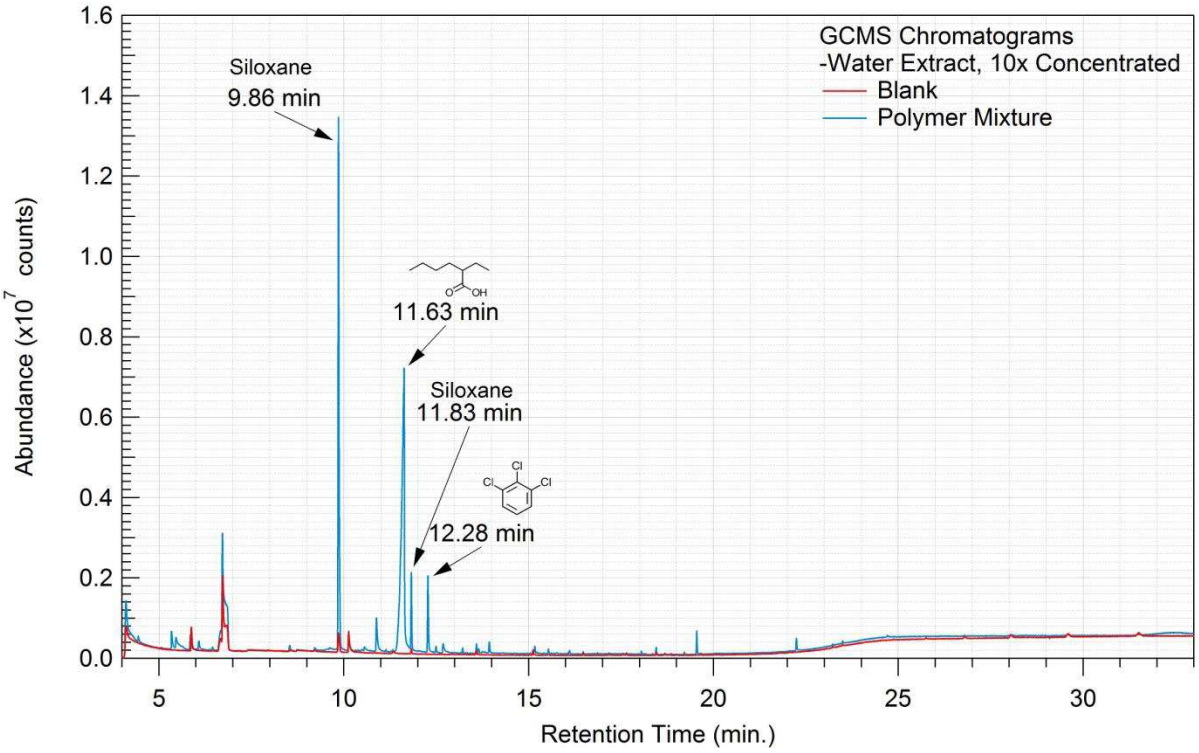


Figure 14. Overlay of GCMS chromatograms of water extract

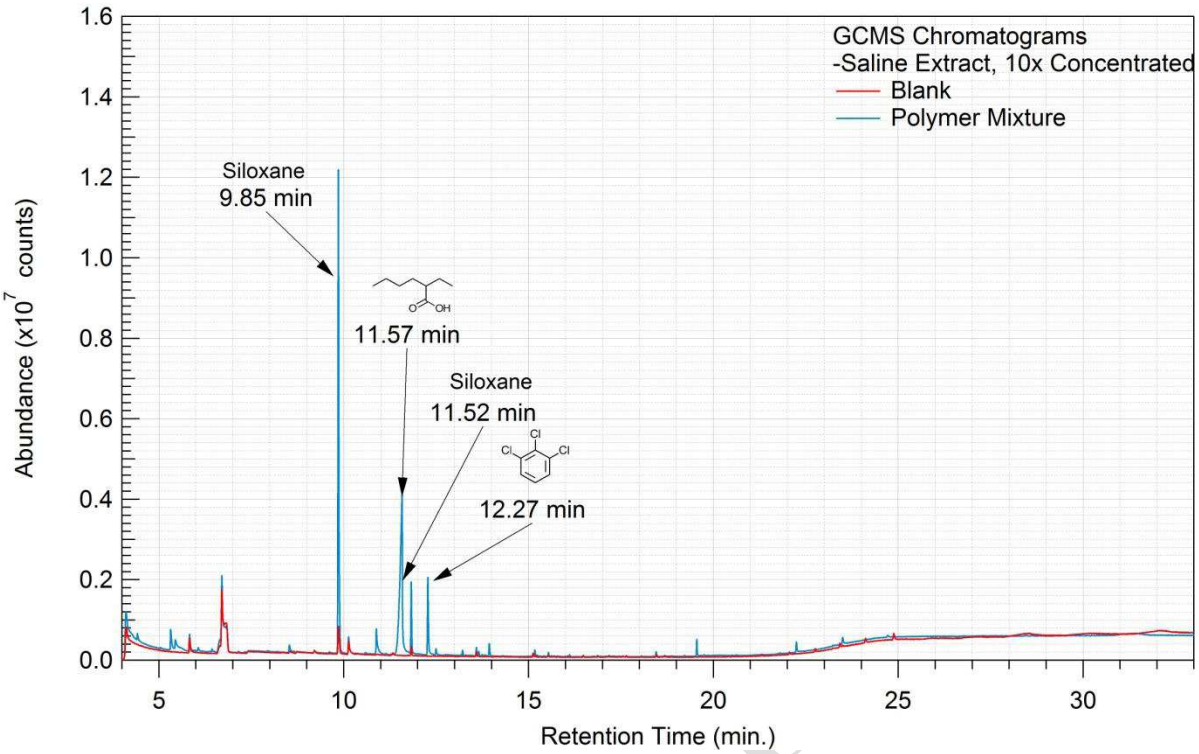


Figure 15. Overlay of GCMS chromatograms of Saline extract

HGCMS

A 1 mL aliquot of each sample of the water extract and saline extract was sealed in a 20ml headspace sampling vial and analyzed on HGCMS. **Figures 16-17** include overlays of the chromatograms collected from the samples and blanks. The results are summarized in **Tables 18-19**.

Table 18
Summary of HGCMS Results - Water Extracts

RT (min)	Possible ID	*CAS*	*Confident Level*	Peak Area*	Estimated Conc.[1] (µg/mL)	Mass per Device[2] (µg)
8.27	Ethoxytrimethylsilane	1825-62-3	*Confident*	84864.71	2.62	1367.10

Quantitation Standard Compound			
RT (min)	Possible Identification	Conc. (µg/mL)	Peak Area
12.18	Siloxane	5.00	162019.53

* Average of two injections

[1] $\text{Estimated Conc. } (\mu g/mL) = \frac{\text{Peak Area (Sample)}}{\text{Peak Area (Standard)}} \times \text{Standard Conc. } (5.00\ \mu g/mL)$

[2] $\text{Mass per device (ug)} = \frac{\text{Estimated Conc. } (\mu g/mL) \times \text{Total Extract Vol. } (532\ mL)}{\text{Number of Devices } (1)}$

Table 19
Summary of HGCMS Results - Saline Extracts

RT (min)	Possible ID	*CAS*	*Confident Level*	Peak Area*	Estimated Conc.[1] (µg/mL)	Mass per Device[2] (µg)
8.27	Ethoxytrimethylsilane	1825-62-3	*Confident*	76167.10	3.21	1657.14

Quantitation Standard Compound			
RT (min)	Possible Identification	Conc. (µg/mL)	Peak Area
12.18	Siloxane	5.00	118814.32

* Average of two injections

[1] $\text{Estimated Conc. } (\mu g/mL) = \frac{\text{Peak Area (Sample)}}{\text{Peak Area (Standard)}} \times \text{Standard Conc. } (5.00\ \mu g/mL)$

[2] $\text{Mass per device (ug)} = \frac{\text{Estimated Conc. } (\mu g/mL) \times \text{Total Extract Vol. } (517\ mL)}{\text{Number of Devices } (1)}$

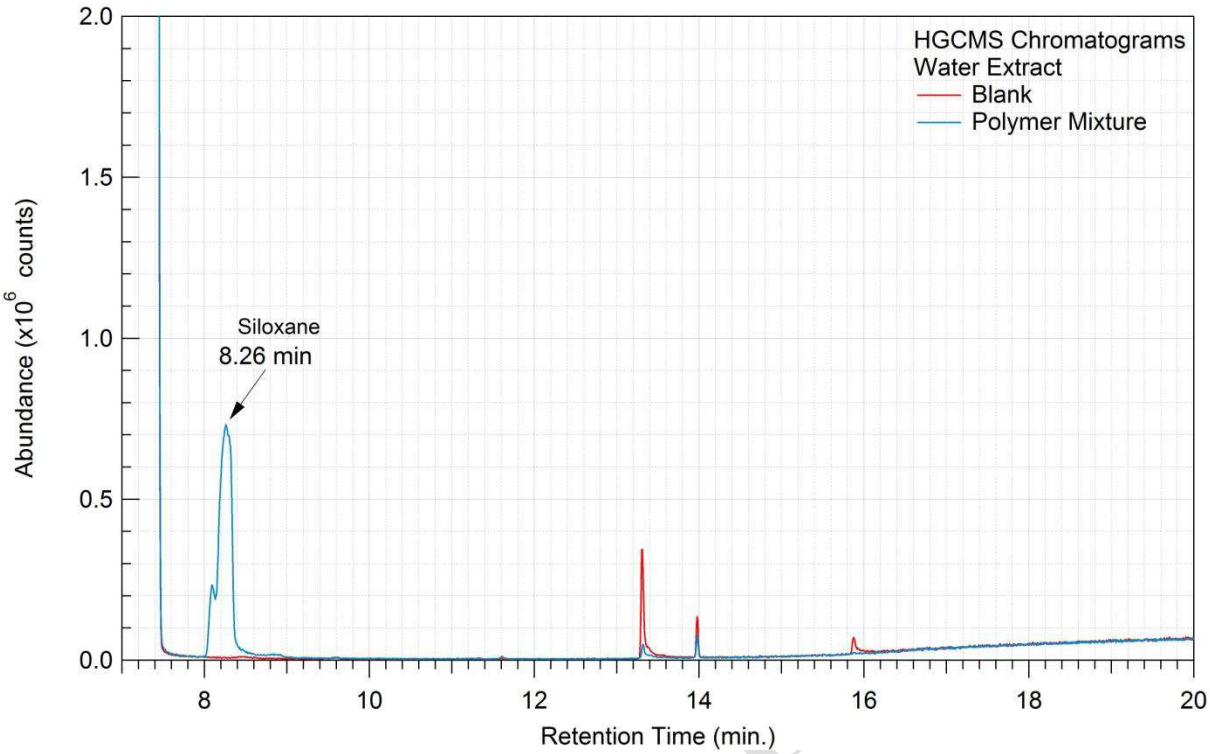


Figure 16 - Overlay of HGCMS chromatograms of water extract.

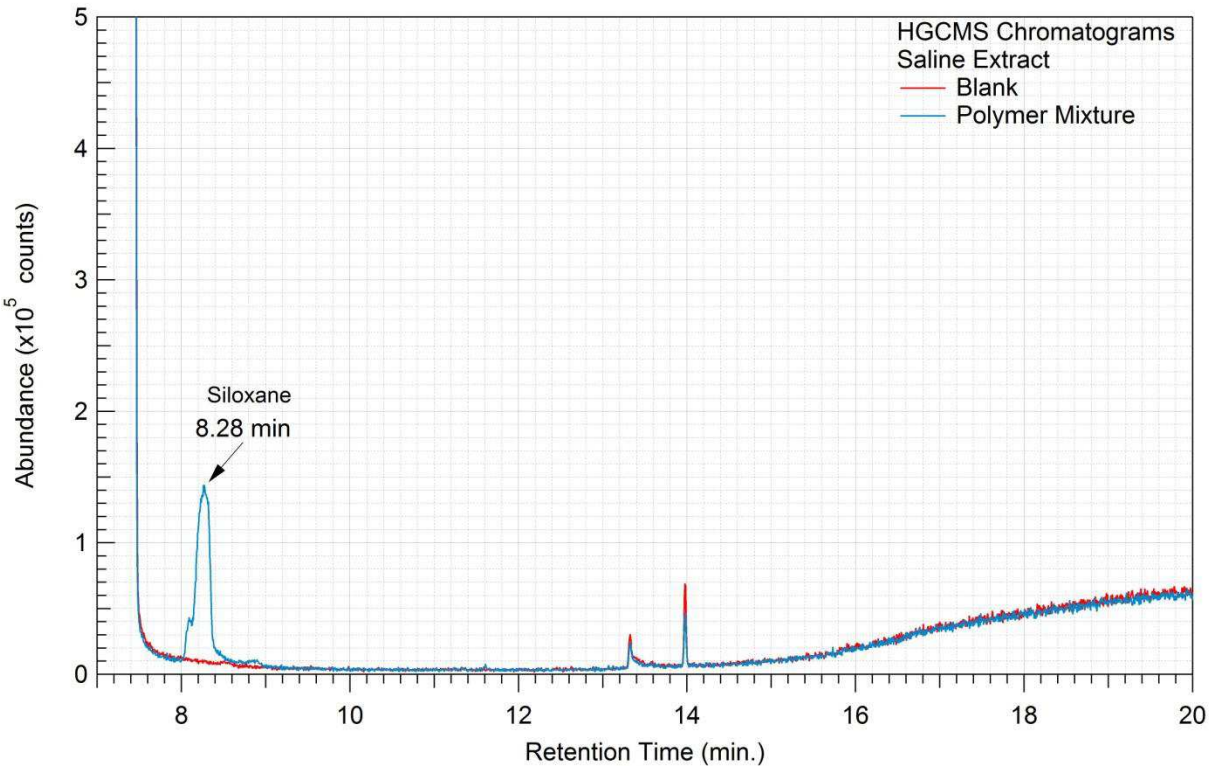


Figure 17 - Overlay of HGCMS chromatograms of saline extract.

ICP-MS

The elemental composition of each extract was determined using ICP-MS for seventy one (71) elements. A control blank extract was analyzed at the same time as the samples. The control had been subjected to all of the same extraction and concentration steps as the samples. Elements which were detected in the blank and sample at similar concentrations are consistent with background components and therefore the samples should not be considered a significant source of these elements. For all calculations, the amount observed in the blank is subtracted from that observed in the extracts. In cases where the blank showed higher concentration than the extract a value of zero (0) is reported.

Elements which show a significantly higher level in one or more of the extracts (3x the background level) are highlighted.

Table 20 and **Table 21** show the results for the extracts in water and saline, respectively.

Table 20 ICP-MS Result Water Extract					
Element	***Polymer Mixture***			***Control Blank***	
	Concentration [µg/L]	Mass per device (µg)[1]	Corrected Mass per device (µg)[2,3,4]	Concentration [µg/L]	Mass per device (µg)[1]
Li	1.22	0.65	0.61	0.08	0.04
Be	0	0	0	0	0
B	43.67	23.23	16.42	12.80	6.81
Na	573.18	304.93	265.78	73.59	39.15
Mg	398.27	211.88	195.27	31.23	16.61
Al	0.29	0.15	0	0.63	0.34
Si	26960.46	14342.96	14205.75	257.92	137.21
P	0	0	0	0	0
S	0	0	0	0	0
K	136.49	72.61	69.87	5.15	2.74
Ca	1516.80	806.94	717.60	167.92	89.33
Sc	0.03	0.02	0.02	0	0
Ti	0.03	0.02	0.02	0	0
V	0.02	0.01	0.01	0	0
Cr	0.07	0.04	0.04	0	0
Mn	1.39	0.74	0.24	0.94	0.50
Fe	0	0	0	0	0
Co	0.31	0.16	0.15	0.03	0.02
Ni	2.63	1.40	1.29	0.21	0.11
Cu	0.52	0.28	0.22	0.11	0.06
Zn	38.38	20.42	1.49	35.57	18.92
Ga	0.02	0.01	0.01	0	0
Ge	0.02	0.01	0.01	0	0
As	0.01	0.01	0.01	0	0
Se	0	0	0	0	0
Rb	0.04	0.02	0.02	0.01	0.01
Sr	2.44	1.30	1.11	0.36	0.19
Y	0	0	0	0	0
Zr	0.01	0.01	0.01	0	0
Nb	0	0	0	0	0
Mo	1.50	0.80	0.80	0	0
Ru	0	0	0	0	0
Rh	0	0	0	0	0
Pd	0	0	0	0	0
Ag	0	0	0	0	0
Cd	0.09	0.05	0.04	0.02	0.01
In	0	0	0	0	0
Sn	0	0	0	0	0
Sb	0.14	0.07	0.05	0.05	0.03
Te	0	0	0	0	0
Cs	0	0	0	0	0
Ba	4.46	2.37	1.15	2.29	1.22
La	0	0	0	0	0
Ce	0	0	0	0	0
Pr	0	0	0	0	0

Table 20
ICP-MS Result
Water Extract

Element	*Polymer Mixture*			*Control Blank*	
	Concentration [µg/L]	Mass per device (µg)[1]	Corrected Mass per device (µg)[2,3,4]	Concentration [µg/L]	Mass per device (µg)[1]
Nd	0	0	0	0	0
Sm	0	0	0	0	0
Eu	0	0	0	0	0
Gd	0	0	0	0	0
Tb	0	0	0	0	0
Dy	0	0	0	0	0
Ho	0	0	0	0	0
Er	0	0	0	0	0
Tm	0	0	0	0	0
Yb	0	0	0	0	0
Lu	0.02	0.01	0	0.04	0.02
Hf	0	0	0	0	0
Ta	0	0	0	0	0
W	0.04	0.02	0.02	0	0
Re	0	0	0	0	0
Os	0	0	0	0	0
Ir	1.96	1.04	0.74	0.56	0.30
Pt	2.38	1.27	1.26	0.01	0.01
Au	0.46	0.24	0	0.48	0.26
^{201}Hg	0	0	0	0	0
^{202}Hg	0	0	0	0	0
Tl	0	0	0	0	0
Pb	0.01	0.01	0.01	0	0
Bi	0	0	0	0	0
Th	0	0	0	0	0
U	0	0	0	0	0

[1]Mass per device (µg) $= \frac{\text{Concentration } \left(\frac{\mu g}{L}\right) \times \text{Total volume extract (0.532 L)}}{\text{number of devices (1)}}$

[2]Corrected Mass per device = Mass per device (Sample) – Mass per device (Control)

[3]If the blank shows the mass per device is below LOD, nothing will be subtracted from the sample because the mass in the blank is unknown.

[4]For elements which there was a higher concentration in the blank or control, a value of 0 is listed.

Table 21 ICP-MS Result Saline Extracts					
Element	***Polymer Mixture***			***Control Blank***	
	Concentration [µg/L]	Mass per device (µg)[1]	Corrected Mass per device (µg)[2,3,4]	Concentration [µg/L]	Mass per device (µg)[1]
Li	1.27	0.66	0.55	0.21	0.11
Be	0.01	0.01	0.01	0	0
B	23.17	11.98	0.00	28.11	14.53
Na	2402225.00	1241950.33	57605.69	2290802.00	1184344.63
Mg	252.73	130.66	89.86	78.92	40.80
Al	3.97	2.05	0.10	3.77	1.95
Si	6327.20	3271.16	2867.36	781.04	403.80
P	12.04	6.22	0.00	11.87	6.14
S	117.55	60.77	18.08	82.57	42.69
K	274.53	141.93	55.31	167.55	86.62
Ca	1212.76	627.00	0	1858.96	961.08
Sc	0.03	0.02	0.02	0	0
Ti	0.01	0.01	0.01	0	0
V	0.05	0.03	0	0.05	0.03
Cr	0.07	0.04	0.04	0	0
Mn	1.96	1.01	0	2.36	1.22
Fe	0.11	0.06	0.06	0	0
Co	0.32	0.17	0.13	0.07	0.04
Ni	4.67	2.41	2.16	0.49	0.25
Cu	2.21	1.14	0.83	0.60	0.31
Zn	268.71	138.92	21.25	227.61	117.67
Ga	0	0	0	0.02	0.01
Ge	0.01	0.01	0.01	0	0
As	0.18	0.09	0.07	0.04	0.02
Se	0.31	0.16	0	0.39	0.20
Rb	0.09	0.05	0	0.10	0.05
Sr	2.39	1.24	0	3.80	1.96
Y	0	0	0	0	0
Zr	0	0	0	0	0
Nb	0	0	0	0	0
Mo	0.22	0.11	0.10	0.03	0.02
Ru	0	0	0	0	0
Rh	0	0	0	0	0
Pd	0	0	0	0	0
Ag	0.93	0.48	0.38	0.19	0.10
Cd	0.92	0.48	0.44	0.07	0.04
In	0	0	0	0	0
Sn	0	0	0	0	0
Sb	0.08	0.04	0.03	0.03	0.02
Te	0	0	0	0	0
Cs	0.05	0.03	0	0.08	0.04
Ba	12.06	6.24	0	21.37	11.05
La	0	0	0	0	0
Ce	0.01	0.01	0	0.01	0.01
Pr	0	0	0	0	0

Table 21
ICP-MS Result
Saline Extracts

Element	*Polymer Mixture*			*Control Blank*	
	Concentration [µg/L]	Mass per device (µg)[1]	Corrected Mass per device (µg)[2,3,4]	Concentration [µg/L]	Mass per device (µg)[1]
Nd	0	0	0	0	0
Sm	0	0	0	0	0
Eu	0	0	0	0	0
Gd	3.29	1.70	1.25	0.87	0.45
Tb	0	0	0	0	0
Dy	0	0	0	0	0
Ho	0	0	0	0	0
Er	0	0	0	0	0
Tm	0	0	0	0	0
Yb	0.01	0.01	0.01	0	0
Lu	0	0	0	0.01	0.01
Hf	0	0	0	0	0
Ta	0	0	0	0	0
W	0.01	0.01	0	0.01	0.01
Re	0	0	0	0	0
Os	0	0	0	0	0
Ir	0.01	0.01	0.01	0	0
Pt	0.58	0.30	0.29	0.01	0.01
Au	0	0	0	0	0
^{201}Hg	0	0	0	0	0
^{202}Hg	0	0	0	0	0
Tl	0.01	0.01	0.01	0	0
Pb	0.27	0.14	0	0.35	0.18
Bi	0	0	0	0	0
Th	0	0	0	0	0
U	0	0	0	0	0

[1]Mass per device (µg) $= \frac{\text{Concentration } \left(\frac{\mu g}{L}\right) \times \text{Total volume extract (0.517 L)}}{\text{number of devices (1)}}$

[2]Corrected Mass per device = Mass per device (Sample) – Mass per device (Control)

[3]If the blank shows the mass per device is below LOD, nothing will be subtracted from the sample because the mass in the blank is unknown.

[4]For elements which there was a higher concentration in the blank or control, a value of 0 is listed.

Analysis Conditions

FTIR

Your samples were tested as-is on Thermo Nicolet iN10 MX FTIR microscope. Spectra were collected in attenuated total reflectance mode except where otherwise noted. The spectra generated were compared to ~ 23,000 entries in our library and the best match determined based upon absorbencies and peak intensities.

QTOF LCMS

The following conditions were used for the qualitative QTOF-LCMS analysis:

QTOF GCMS

The following conditions were used for the qualitative QTOF-GCMS analysis:

Headspace GCMS

The following run conditions were applied for Head Space analysis:

ICP-MS

The following run conditions were applied for ICP-MS analysis:

Closing Comments

Jordi Labs' reports are issued solely for the use of the clients to whom they are addressed. No quotations from reports or use of the Jordi name is permitted except as authorized in writing. The liability of Jordi Labs with respect to the services rendered shall be limited to the amount of consideration paid for such services and do not include any consequential damages.

Jordi Labs specializes in polymer testing and has 30 years experience doing complete polymer deformulations. We are one of the few labs in the country specialized in this type of testing. We will work closely with you to help explain your test results and solve your problem. We appreciate your business and are looking forward to speaking with you concerning these results.

Sincerely,

Joyce Zhao

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