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Biobased materials used in food contact applications: an assessment of the migration potential

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SUMMARY

The use of biobased materials as food packaging materials is increasing. The main advantages biobased materials have over fossil-based plastics are the use of renewable resources in their production and, in many cases, the biodegradability and/or compostability of the finished product which offers an alternative to disposal in landfill. As public interest and concern about environmental issues such as the use of non-renewable resources and the amount of waste being sent to landfill increases then it is foreseen that this will continue to rise.

Although it is well recognised that the use of materials derived from biomass is desirable it is important to ensure that the safety of the foods packaged within is not compromised. This project has assessed the suitability of biobased materials to be used in contact with food in terms of the determination of any potential migrants derived from the biological source itself or from any additives required to allow the packaging materials to fulfil their function and thereby ensuring that the materials meet the legislative requirements and that they don't endanger human health.

Following the preparation of a comprehensive literature review thirteen samples covering a range of biobased material types (starch, cellulose, poly(lactic acid), cassava and bagasse based) were obtained and tested. The identities of any potential migrants in the biobased materials as received and following a period of ageing (high temperature and high humidity) were determined using a suite of analytical methods selected to detect substances with a molecular weight below 1,000 Dalton. This molecular weight was selected in view of the relative ease of migration of such substances and their toxicological significance. The materials and extracts thereof were analysed by headspace GC-MS (to detect any volatile substances present), by GC-MS (to detect any semi-volatile substances) and by LC-TOF-MS (to detect any polar and non-volatile substances). The substances detected were material specific. Following a consideration of the nature of the substance and any restrictions placed on their use in food contact materials migration studies for selected substances were carried out into foods and food simulants. There was little measurable migration from the materials tested. Where migration was observed the simulants defined in the legislation (for plastics) overestimated or provided a good approximation to the migration into foods. This was in agreement with the suggestion made in FSA-commissioned project (A03040) on an investigation of the nature and extent of biodegradable polymers used in direct food contact applications that 'The methods of test for migration, using food simulants, are likely to be directly applicable to testing most biodegradable polymers…..' albeit for the limited number of material/migrant/ simulant/food combinations studied here.

The limitations of the overall migration methods as defined in the CEN standards highlighted in the aforementioned FSA-commissioned project (A03040) that test using olive oil as a simulant for overall migration may not be technically possible for humidity-sensitive materials were confirmed. Tests for overall migration into olive oil require preconditioning to constant weight and as expected for the absorbent materials tested here this could not be achieved. For these samples conditioning by vacuum drying should be followed. Exposure to water and other aqueous simulants altered the appearance of several of the samples included in this project. In several cases similar observations were made when the samples were used in contact with the aqueous foodstuffs and therefore it is recommended that samples should be labelled with respect to the appropriate use conditions to prevent deformation during use.

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ABBREVIATIONS

CEN	European Committee for standardisation
DEG	Diethylene glycol
DBP	Di-n-butyl phthalate
DiBP	Diisobutyl phthalate
DIPN	Diisopropyl naphthalene
DMT	Dimethyl terephthalate
EI	Electron impact
EFSA	The European Food Safety Authority
EU	European Union
FAO	Food and Agriculture Organization of the United Nations
Fera	The Food and Environment Research Agency
FSA	Food Standards Agency
GC	Gas chromatography
GC-MS	Gas chromatography-mass spectrometry
HDPE	High density polyethylene
HS-GC-MS	Headspace-gas chromatography-mass spectrometry
IS	Internal standard
JECFA	Joint FAO/WHO Expert Committee on Food Additives
LC	Liquid chromatography
LC-TOF-MS	Liquid chromatography-time-of-flight-mass spectrometry
LDPE	Low density polyethylene
LLDPE	Linear low density polyethylene
LOD	Limit of detection
LOQ	Limit of quantification
MEG	Monoethylene glycol
MS	Mass spectrometry
NIAS	Non-intentionally added substances
NNFCC	National Non-Food Crops Centre
PAR	Peak area ratio
PEG	Polyethylene glycol
PET	Polyethylene terephthalate
PG	Propylene glycol
PDO	1,3-Propanediol
PHA	Polyhydroxyalkanoate
PHB	Poly(3-hydroxybutyrate)

PHBH	Poly(hydroxybutyrate-co-hexanoate)
PHBV	Poly(3-hydroxybutyrate-co-valerate)
PLA	Polylactic acid
PP	Polypropylene
PS	Polystyrene
PTA	Terephthalic acid
PTT	Poly(trimethylene terephthalate)
PVC	Polyvinyl chloride
RCF	Regenerated cellulose film
SIM	Selected Ion Monitoring
SOP	Standard Operating Procedure
TEG	Triethylene Glycol
TIC	Total Ion Chromatogram
TOF	Time-Of-Flight
TPS	Thermoplastic Starch
USDA	United States Department of Agriculture
WHO	World Health Organisation
WRAP	Waste Resources Action Programme

1. INTRODUCTION

1.1 Background

Biobased materials are defined as being derived, directly or indirectly, from a renewable source of living matter. Examples include paper/board and biobased polymers. Recent years have seen a major increase in the use of biobased materials in food contact applications. As development in this area continues it is necessary to ensure that the materials being used have been manufactured such that they comply with Framework Regulation (EC) No. 1935/2004, i.e. they should not transfer their constituents to food in quantities which could endanger human health, bring about an unacceptable change in the composition of the food or bring about a deterioration in the organoleptic characteristics. Any biobased materials in contact with food have to meet this requirement.

There is increased public interest and concern about environmental issues such as the use of non-renewable resources and the amount of waste being sent to landfill, and consumers see packaging as a major environmental problem. Sustainability and concepts such as “closed-loop” or “cradle-to-cradle” resourcing are increasingly important in corporate responsibility, and there is increasing legislation aimed at reducing landfill waste. As a result, the packaging industry is under considerable regulatory and public pressure, and there is the potential for significant substitution of fossil-based plastics with those from natural and renewable sources.

The main advantages biobased plastics have over fossil-based plastics are the use of renewable resources in their production and, in many cases, the biodegradability and/or compostability of the finished product which offers an alternative to disposal in landfill. This is particularly important when the product is designed to be disposable, as in the case of packaging. Compostability is a particular advantage for food packaging, which is often not recycled because it is lightweight and food-contaminated and therefore costs more to clean and process than it is worth[1] – if compostable food packaging is used, the food waste and packaging can be disposed of in an environmentally-friendly way without separation. Biobased plastics also offer an alternative renewable source to recycled plastic which, again, is particularly useful for food packaging due to the shortage of the high quality feedstock required to produce food grade recycled plastic.[2]

The term “biobased” is often confused with, and used interchangeably with, that of “biodegradable” (or “compostable”). However, the former relates to the origin of the material and the latter to one of the means by which it can be broken down, which depends on the chemical structure of the finished material. In fact, biobased materials are not always biodegradable or compostable, and biodegradable or compostable plastics are not necessarily biobased. Biobased plastics based on naturally-occurring polymers are generally biodegradable, whereas those synthesised from naturally-occurring monomers can lose this property through chemical modification.[3,4]

1.2 Types of biobased materials used for food packaging

Paper and board are the most widely used examples of biobased materials,[5] and they are commonly used for food packaging. Because the use of paper/board materials as food packaging is well established and the migration from these materials has already been studied they were not included in the scope of this project. Biobased polymers (also known as biopolymers and bioplastics) can be divided into three broad categories based on their origins (Figure 1): [5,6]

- polymers directly extracted from biomass
- polymers produced by chemical synthesis using biobased monomers

- polymers produced by micro-organisms or bacteria

1.2.1 Polymers directly extracted from biomass

These include polysaccharides, proteins and lipids.

1.2.1.1 Polysaccharides – Starch[5-7]

Starch is the major storage carbohydrate in plants, and can be found in the seeds, roots, tubers, stems, leaves and fruits. The two main components of starch are polymers of glucose; amylose (a linear molecule with molecular weight 10^5-10^6) and amylopectin (a highly-branched molecule with molecular weight 10^7-10^9). Most plants contain 20-25% amylose, but some species of maize contain 50-90% amylose.[8] Starch is unique among carbohydrates in that it occurs naturally as discrete granules due to the crystalline helical structures formed by the short-branched amylopectin chains. These granules are hydrophilic and have strong inter-molecular association due to hydrogen bonding between hydroxyl groups on the granule surface. Raw materials for starch plastic can be obtained from corn (maize), wheat, potatoes, cassava, tapioca and rice. Waste flows can be used, e.g. potato peelings from the French fries industry.

Native starch can be pulped together with cellulose fibres and made into compostable trays and cups by hot-pressing methods similar to moulding paper pulp. Native starch cannot be thermally processed because its melting point is higher than its thermal decomposition temperature, so it is typically processed to make thermoplastic starch.

Thermoplastic starch (TPS) is made by processing native starch in an extruder under controlled conditions of temperature, pressure, shear and water content. This melts the crystalline structures, the starch granules swell and open and an inter-molecular rearrangement takes place transforming the semi-crystalline polymer to an amorphous polymer with greatly enhanced processability. Additives can be integrated into the process to provide the final resin composition in one step. Plasticisers such as glycerol and urea are added to reduce inter-molecular hydrogen bonding and stabilise the product. TPS can be processed into flexible or rigid plastics and fillers in the same way as processing a traditional plastic, e.g. film and bottle blowing, cast film, injection moulding or thermo forming. However, TPS is of limited usefulness due to high levels of hydrophilicity and limited mechanical properties.[5]

The problem of hydrophilicity can be addressed by chemically modified starch, in which hydroxyl groups on the starch molecule are replaced by ester or ether groups by chemical reaction, or are cross-linked with each other. Cross-linking inhibits the swelling of starch granules and gives increased stability to acid, heat and shear. This form of starch is expensive and its use is not widespread.[5]

The properties of TPS or native starch can be improved by blending with other materials. The starch content of blends varies from 30-80% depending on application, and the most commonly-used co-polymers are biodegradable polymers derived from fossil feedstocks, e.g. BASF's Ecoflex®. This makes most starch blends only partially biobased but fully biodegradable (e.g. Mater-Bi®), although fully biobased blends can be produced with PLA or PHA/B copolymers. Durable polymers can also be produced by blending starch with fossil-based polymers such as polypropylene (e.g. Cereplast Hybrid™) or polyurethane (Biopar® TPU).

Starch-based polymers constitute a major share of the total biobased polymer market,[9] and global production capacity reached 170,000 in 2007. Of this, 75% was produced in Europe, where capacity increased from 30,000 tonnes to 130,000 tonnes

from 2003-2007, an annual increase of almost 50%. The price (June 2009) of starch plastics in Europe ranges from €2-5 per kg. There is considerable scope for decrease although this may depend on fluctuations in the price of agricultural commodities.[5]

Manufacturers of commercially available starch-based polymers and the trade names of the materials produced are listed in Table 1.

The density of starch plastics is higher than that of conventional plastics and also most biobased plastics, making them less price competitive on a volume basis. TPS and starch blend films have reasonable transparency and are intrinsically anti-static. Barrier properties for oxygen and carbon dioxide are moderate to good, but the range of applications is restricted by sensitivity to moisture and high water vapour permeability. Mechanical properties are generally inferior to conventional plastics. The potential for starch plastics to substitute conventional plastics is greatest for polyolefins, mainly low density polyethylene (LDPE), high density polyethylene (HDPE) and polypropylene (PP), and polystyrene (PS).[5]

The largest application area for starch plastics is packaging applications, including soluble films, films for bags and loose fill material. Starch blends are widely used for food packaging applications in the form of wrap films, single-use foamed trays and boxes and table ware. Their relatively high water vapour permeability is an advantage for the fog-free packaging of warm foods.[5]

1.2.1.2 Polysaccharides – Cellulose[5-7]

Cellulose is the main cell wall constituent of all major plants and is the most abundant natural polymer on earth. All plants contain cellulose, but it is most abundant in wood pulp, cotton fibres, linen fibres, jute, and hemp.[10] Cellulose is also found in the cell walls of green algae and the membranes of most fungi. *Acetobacter xylinum* and *A. pasteurianus* can produce an almost pure form of cellulose with a chemical and physical structure identical to that found in plants.[6]

Cellulose, like starch, is a polymer of glucose, with different linkages between the glucose units and the configuration of the polymer chains. Cellulose's configuration provides an opportunity to form stronger hydrogen bonds between polymer chains, as well as a close interaction with other polymeric structures such as lignin, starch, pectin, hemicelluloses and proteins. Because of this mixed polymer morphology, cellulose is more resistant to hydrolysis than starch.

The most familiar application of cellulose based packaging is paper and board however, as stated above, migration from these materials are already well characterised and they are not discussed here. There is an increasing interest in using the natural plant fibres as received, i.e. without extracting the cellulose. The fibres can be processed into composites using small amounts of natural or synthetic binders, using technology similar to that used in the paper pulp industry.[5,7]

Cellulose films (cellophane) are produced by the chemical modification of natural cellulose, primarily from wood but also from linters (short cotton fibres). Cellulose is extracted by digesting wood pulp at high pressure in a series of chemical baths to remove impurities and break the long fibre chains. The resulting viscose liquid is filtered, extruded and then cast along a series of rollers and baths, during which the film is cleaned and softened.[11]

Cellulose can also be chemically modified to prepare esters such as cellulose acetate, cellulose acetate butyrate and cellulose propionate. Cellulose acetate is produce by reacting cellulose with acetic anhydride, and the cellulose acetate is then precipitated in water, dried and dissolved in acetone before being cast as a film by evaporating the acetone under controlled conditions.[10]

Regenerated cellulose can be made into fibres or films, fibres being much more economically important. Regenerated cellulose represents the bulk of the cellulose industry at 3.5 million tonnes worldwide, and cellulose esters account for > 1 million tonnes.[5] High processing costs currently prevent the bulk use of many cellulose derivatives.[6]

Manufacturers of commercially available cellulose-based films and packaging (i.e. excluding fibres) and the trade names of the materials produced are listed in Table 2.

Cellophane has high transparency and gloss. Uncoated cellulose films are highly permeable to water vapour but provide an excellent barrier to bacteria, flavours and aromas. They are naturally anti-static, and heat-resistant. Most cellulose films are coated, metallised or laminated in order to refine their natural attributes and for specific applications, e.g. oxygen or moisture barrier and heat sealing.[11] These coatings can be synthetic or biobased, although the most effective coatings are not biobased. Cellophane film is generally coated with nitrocellulose wax or polyvinylidene chloride. Cellulose acetate films are crystal clear, tough, scratch-resistant, anti-static and readily dyeable, although they must be plasticised for film production. Films with the highest level of acetate substitution have reduced moisture sensitivity but are also less biodegradable.[5]

Cellophane is popular for packaging applications because of its attractive appearance and transparency. Coated cellophane films are used for packaging baked foods, snacks, cheese, coffee, confectionary, crisps and spices. Of the cellulose esters, only cellulose acetate is widely used in food packaging, for baked goods and fresh produce. Unmodified cellulose composites can also be used for packaging, e.g. rigid trays.

1.2.1.3 Polysaccharides – Other[6]

Other polysaccharides include chitin/chitosan, hemicellulose, guar gum and pectin.

Chitin is a naturally occurring polymer present in the exoskeleton of invertebrates, and chitosan is a family of polymers derived from chitin. Chitosan readily forms films with high gas barrier properties, and has been used in the production of edible coatings. It may also be used to coat other biobased polymers which lack gas barrier properties. As with other polysaccharide-based polymers, care must be taken for moist conditions. Chitin has anti-microbial properties which may prove useful in food contact applications, and it has been shown that biodegradable laminate of chitosan-cellulose and polycaprolactone can be used in modified atmosphere packaging of fresh produce.

To date, no commercial applications of biobased plastics from chitin/chitosan and the other polysaccharides listed above have been identified.

1.2.1.4 Proteins[6]

Proteins can be divided into those of plant origin (e.g. gluten, soy, zein, pea and potato) and animal origin (e.g. casein, keratin, collagen and whey). Proteins contain numerous functionalities that can be modified to generate the polymer. Due to its abundance and low cost, research into the use of gluten in edible films, adhesives or for thermoplastic applications is being carried out. Soy proteins have been used in adhesives, inks and paper coatings. Zein proteins can be used to form films which, although brittle and requiring plasticisers, show potential for uses in edible coatings and biobased packaging. Casein is expensive, but it can be plasticised and made into stretchable films. Keratin is the cheapest of the proteins, as it can be derived from waste streams such as hair and feathers, but it is also the most difficult to process and yields plastic with poor mechanical properties. Collagen is the basic raw material for

the production of gelatine, which is a commonly-used food additive with the potential for film and foam production. Whey proteins have been extensively investigated as edible coatings and films.

The main drawback of all protein plastics, with the exception of keratin, is their sensitivity to humidity. Blending or lamination may be able to solve this problem, but research has been limited to date. To date, no commercial food contact applications have been identified for biobased plastic derived from these proteins.

1.2.1.5 Lipids

Origo-Bi™ by Novamont is a biodegradable copolyester made of 30-70% renewable raw materials from vegetable oil, blended with fossil-based biodegradable polymers (see section 3.2.2). Origo-Bi™ is more transparent than Novamont's more well established starch-based Mater-Bi®.[12]

1.2.2 Polymers produced by chemical synthesis using biobased monomers

The most common biobased plastic synthesised from biobased monomer is poly(lactic acid). Monomers for various conventionally fossil-based polymers can also be obtained from biobased sources for the synthesis of biobased (or partially biobased) versions of the plastics.

1.2.2.1 Polylactic acid (PLA)[5-7]

Poly(lactic acid) (PLA) is an aliphatic polyester produced by the polymerisation of lactic acid (2-hydroxypropionic acid). Lactic acid can be produced synthetically from hydrogen cyanide and acetaldehyde, or naturally by anaerobic fermentation by bacteria or certain fungi of carbon substrates, either pure (e.g. glucose and sucrose) or impure (e.g. starch). The range of raw materials used is expected to expand and move towards the utilisation of agricultural waste. Lactic acid exists in D- and L- forms, L-lactic acid being the naturally-occurring form, and fermentation offers the best route to optically-pure monomer by selection of an appropriate *Lactobacillus*.[5,13]

The condensation polymerisation of lactic acid generally yields low molecular weight polymers and this method is not used commercially. Higher molecular weights are obtained by ring-opening polymerisation of lactide, a cyclic dimer composed of two units of lactic acid. When racemic lactides are used, the result is an amorphous polymer which is not suitable for packaging.[13] Pure L-PLA has high melting point and crystallinity.[6] The availability of both optically pure lactides allows polymer producers to control the combination of L-PLA with D-PLA and thus enhance the characteristics of the finished polymer such as heat resistance.[14] The polymer of choice for most packaging applications is 90% L-lactide and 10% racemic D,L-lactide.[13]

PLA can also be blended with other polymers to further extend the applications. BASF produce Ecovio®, a blend of PLA and the fossil-based polyester EcoFlex®. Starch-PLA blends (e.g. Cereplast Hybrid™) are available and PLA/PHA blends are being investigated.[5] DuPont produce two additives specifically designed to enhance the properties of PLA; Biomax® Strong enhances impact strength, flexibility and viscosity, and Biomax® Thermal 300 increases the stability up to 95℃. [15]

Purac is world's largest supplier of lactic acid (100,000 tonnes per annum), derived from cane sugar or tapioca starch. Natureworks LLC also manufacture lactic acid, from corn starch, and are the world's only large volume producer of PLA (150,000 tonnes per annum). Their PLA costs €1.90 per kg. The majority of the other companies producing PLA products do so by processing Natureworks PLA. Demand

for PLA is increasing rapidly, and it is expected to be the largest bioplastic produced in the US by 2011 at > 300 million lbs.[16]

Manufacturers of commercially available PLA and the trade names of the materials produced are given in Table 3.

PLA can be converted to end products by a variety of plastics processing techniques including thermoforming, injection moulding, blow moulding extrusion, foaming, film extrusion and fibre extrusion. The mechanical properties of PLA compare well with fossil-based thermoplastics, and it is reasonably transparent, with high gloss and low haze. PLA films can hold creases or twists, a property normally lacking in plastic films. The physical properties of PLA make it a good candidate for replacement of fossil-based plastics in several application areas of LDPE, HDPE, PP and poly(ethylene terephthalate) (PET). High-value films, rigid containers and expanded foams are the most promising bulk applications.[5]

PLA is suitable for food contact applications. Residual lactide in the polymer is not a food safety concern because it hydrolyses to form lactic acid, which occurs naturally in food and in the body. Natureworks PLA, the most commonly used PLA for food contact applications (worldwide), is approved for direct contact with all aqueous, acidic and fatty foods below 60^{o}C and for acidic drinks served under 90℃. PLA has high resistance to grease and oils, and is therefore used in the packaging of viscous, oily liquids. It is also suitable for packaging dry products and those with a short shelf life. Although it is suitable for serving beverages it is not used for packaging carbonated drinks and other liquids due to its poor oxygen, carbon dioxide and water vapour barrier properties.[5] One of the largest barriers to the adoption of Natureworks PLA in the EU is the company's use of genetically modified crops, although grades which are certified GM-free are available.[7]

PLA is one of only a small number of synthetic polymers that are fully biodegradable and compostable,[9] however, the high temperature and humidity of industrial composting facilities are required, and PLA does not degrade in soil, seawater or landfill and is not home compostable. If not disposed of properly PLA will maintain its integrity in the near term. PLA can also be physically recycled or chemically converted back to lactic acid through hydrolysis if sorting facilities exist.[17]

1.2.2.2 Conventional polymers from biobased monomers[5]

Several types of conventional plastics can be synthesised using biobased monomers in place of the usual fossil-based sources. The finished plastics are indistinguishable from the fossil-based versions.

Polyesters - Poly(trimethylene terephthalate) (PTT) is produced by the condensation of 1,3-propanediol (PDO) and either terephthalic acid (PTA) or dimethyl terephthalate (DMT). Biobased PDO can be produced by aerobic fermentation of glycerol (a by-product of bio-diesel production) or glucose (from corn starch). PTT has similar properties to PET and can be produced in the same facilities. Biomax® PTT by DuPont contains up to 35% renewably sourced content in the form of Bio-PDO™. Biobased PDO can also be used in the manufacture of polyols and converted to polyurethane.[5,15]

BASF produce the biodegradable polyester poly(butylene adipate terephthalate) (PBAT), known as EcoFlex®, which is synthesised using fossil-based monomers. This plastic is used in many blends with biobased plastics to improve their properties, therefore a biobased alternative would be desirable. There is potential for the butanediol and adipic acid used in its production to be derived from biobased sources.

Novamont's Origo-Bi™ is PBAT with 30-70% renewable content derived from vegetable oil.[5]

The Coca-Cola Company recently unveiled PlantBottle™, a partially biobased PET bottle which contains up to 30% renewable materials.[18] The ethylene glycol used to produce the bottles is derived from sugar and molasses, and the company is currently researching biobased alternatives to terephthalic acid. The bottle has the advantage of being recyclable alongside conventional PET, and could potentially replace it in all applications.[19]

Polyurethanes - Polyurethanes are produced by reacting a polyol and an isocyanate. Isocyanates are, to date, derived exclusively from fossil-based feedstocks but the polyol component can be derived from vegetable oils.

Polyamides - Commercially available bio-based polyamides include PA11 (monomer 11-aminoundecanoic acid derived from castor oil) and PA610 (partially biobased using sebacic acid from castor oil). Other monomers can potentially be obtained from biobased sources, e.g. adipic acid from fermentation of sugar could be used to produce partially biobased PA66, and azelaic acid, a monomer of PA69, can be derived from oleic acid found in vegetable oils.[5]

Polyolefins - Polyethylene (PE) can be produced using bio-ethanol. Brazil has 350 ethanol production units making 20 million cubic metres of ethanol per year from the anaerobic fermentation of sugar cane.[14] Sugar beet or starch crops such as corn and wheat can also be used. The bio-ethanol is then used to produce ethylene. By far the most important product made from ethylene is polyethylene (LDPE, LLDPE, HDPE), but ethylene is also used in large quantities to produce PVC, PET and PS. Biobased propylene, for the production of polypropylene is at the pilot production scale.[5]

Biobased PE was produced in the 1980s, but production ceased when oil prices fell. The recent increases in oil prices have regenerated interest and biobased polyethylene will become commercially available in 2010. Production capacity is expected to reach 550,000 tonnes in 2012. As the properties are identical to that of fossil-based PE, biobased PE could potentially substitute in all applications. LDPE and HPDE currently account for 34% of the plastics used in Western Europe, and 57% of this PE is used in packaging.[5]

Bio-ethanol can also be used in the production of poly(vinyl chloride) PVC. However, the production of PVC involves the formation of toxic by-products, and toxic substances can also be released during its use (plasticisers) and disposal (dioxins from incineration). The use of biobased ethylene is unlikely to lessen the environmental impact of PVC production. The PVC industry, particularly packaging, has come under criticism due to the challenges of separating PVC from other post-consumer waste, and the amount of PVC used for packaging has been substantially reduced.[5]

Of these conventional polymers synthesised from biobased monomers, biobased PE is the most important in terms of bulk commercial availability and food packaging applications. Braskem and Dow Chemical Company will both produce biobased PE for the food packaging industry.[5]

Manufacturers of commercially available biobased monomers and polymers synthesised using them are listed in Table 4.

1.2.3 Polymers produced by micro-organisms or bacteria

1.2.3.1 Polyhydroxyalkanoates[5-7,14,20]

Polyhydroxyalkanoates (PHAs) are, like PLA, aliphatic polyesters produced via the fermentation of renewable feedstocks such as corn. In contrast to the two stage process of PLA production, i.e. fermentation to monomer followed by polymerisation, PHAs are produced directly within the micro-organism. The semi-crystalline polymer accumulates as granules within the cytoplasm of the cells, from where it can be collected by solvent extraction or using enzymes. The choice of bacteria strain (e.g. *E. coli*, *Ralstonia eutrophus*) and feedstock affects the composition, and hence the final properties, of the PHA polymer.[21-22]

The most common member of the PHA group of polymers is poly(3-hydroxybutyrate) (PHB) which is produced by the polymerisation of 3-hydroxybutyrate monomer. The properties of the PHA polymer can also be modified by coplymerisation. Poly(hydroxybutyrate-co-hexanoate) (PHBH), a family of copolymers made up of 3-hydroxybutyrate and other 3-hydroxyalkanoates with side groups ≥ 3 carbon units. The incorporation of these side groups lowers the crystallinity and melt temperature of the polymer, making it easier to process, and provides ductility and toughness in the finished product.[23] Also available is poly(3-hydroxybutyrate-co-valerate) (PHBV) in which the copolymer 3-hydroxyvalerate adds flexibility. Commercial grades contain 5% valerate and grades containing up to 15% valerate are currently being tested.

PHAs are in the early stages of commercialisation, with a number of plants due to begin production in 2009-2010. Tianan's production capacity reached 2000 tonnes in 2007 and an increase to 10,000 tonnes was announced in 2009. Green Bio/DSM began production in 2009 with a capacity of 10,000 tonnes, as did Telles with 50,000 tonnes. Kaneka plan to produce 50,000 tonnes in 2010. Meredian have announced plans for production facilities with an annual capacity of 270,000 tonnes. The price of PHAs is currently much higher than other biobased plastics, despite a considerable decrease in the last five years. In 2009 Tianan's PHBV cost $4.40 per kg and the company expects the price to fall further with the use of lower cost feedstocks and more efficient processes.[5] Metabolix have developed genetically-modified designer bacteria that are more efficient in making plastics,[14] although there are wider concerns related to the use of GM organisms.

Manufacturers of commercially available PHA polymers and the trade names of the materials produced are listed in Table 5.

PHAs are fully biodegradable in soil, compost, rivers and oceans, but in the absence of biodegradation conditions they are durable (although the PHB homo-polymer becomes brittle on ageing). The various types of PHA can be converted into films, moulded articles, fibres, elastics, laminates and coatings, fabrics and foams, using a variety of conventional conversion processes. PHA films are translucent and injection moulded articles have high gloss. PHAs have low water vapour permeability, which is of particular relevance to food packaging applications. They are most applicable as substitutions for PVC, HDPE, LDPE and PP.

Injection moulded PHAs are used for cutlery, packaging (bags, boxes and foams) and a wide variety of non-packaging applications. Packaging and agricultural films are the most important markets. Potential food contact applications for Mirel™ include hot cups, lids, food containers and beverage cartons. Meredien and Green Bio PHAs can be used for food service ware.[5]

1.2.3.2 Bacterial cellulose[5-6]

As mentioned in section 3.1.2, cellulose can be produced by bacteria. *Acetobacter xylinum* and *A. pasteurianus* can produce an almost pure form of cellulose with a chemical and physical structure identical to that found in plants. Bacterial cellulose is processed under ambient conditions, in contrast to the harsh chemical and high temperature treatment required for the processing of plant cellulose, resulting in a higher degree of polymerisation in the finished cellulose. Bacterial cellulose (biocellulose) is chemically pure and very strong, however, low yields and high costs are currently barriers to large-scale production and its applications for bulk products are limited.

1.3 Market size and share

1.3.1 Current market

The production of plastics is a large industry which has shown almost continuous growth, on average by 9% each year since 1950, although it suffered a downturn in 2008 due to the global economic crisis. The industry is the largest application of crude oil after energy and transport (although only 4% of fossil fuel is used as raw material for plastics production). Global production was 245 million tonnes in 2008 (down from 260 million tonnes in 2007), of which 25% was produced in the EU, 1.5% in the UK. The demand by converters in the EU in 2008 was 48.5 million tonnes, 4 million tonnes in the UK. The net export of plastics produced in the EU in 2008 was 6.8 million tonnes, worth €8.7 billion, and of converted plastic products 1.2 million tonnes, worth €4.4 billion. Both these figures were lower than in 2007.[24-25]

Packaging remains the largest end-use application of plastics at 38%.[24-25] The global packaging industry is worth about £300 billion and the UK industry over £9 billion.[26]

Biobased plastics currently have a share of less than 1% of the total plastics market, estimated at 0.36 million tonnes in 2007. However, there has been a large amount of research and development into these materials in recent years, into new materials and improving the performance of existing materials, and the global market for biobased plastics is experiencing rapid growth, on average 38% per year between 2003-2007. The production capacities of most biobased polymer manufacturing plants are still small when compared with conventional plants, but increased demand will result in larger capacities of commercially available materials, allowing accelerated growth. Company announcements predict a production capacity of 2.33 million tonnes in 2013 and 3.45 million tonnes in 2020.[5]

England is among the European countries with the highest consumption of biobased plastics, along with Germany, France, Italy and the Netherlands.[3] The USA is the largest single market for biodegradable packaging, where demand is expected to reach 97 million lbs by 2011.[16]

1.3.2 Factors influencing market share

Plastics are so widely used because their enormous diversity makes them suitable for a wide range of applications. The types of biobased plastics that are currently available have the properties required to cover approximately 5-10% of the current plastics market.[3] This coverage could theoretically increase to 90% in the long term.[5] The range of applications of existing biobased plastics can be extended by combining different types to form blends or multi-layer films, or by combining biobased plastics and paper. Biobased plastics may also be combined with fossil-based plastics, and synthetic additives are frequently used in small quantities to improve the functional

properties of the finished product. Biobased plastics can be processed into a vast number of products using all the conventional plastics processing technologies.[3]

The increasing interest in biobased plastics is driven mainly by environmental concerns, principally the use of non-renewable resources in the manufacture of fossil-based plastics and their persistence in the environment after disposal. Consumers in Europe in 2008 generated 24.9 million tonnes of post-consumer plastic waste. Of this, just over half was recovered (i.e. recycled or used in energy recovery) and the remainder (12.1 million tonnes) went to landfill. Packaging contributes the major part of what is today recycled, with over 90% of crates and boxes and 40% of bottles and industrial films being recycled, although recycling of mixed plastics is low (less than 10%). Diversion of waste from landfill is increasing slowly – the recycling of post-consumer packaging in the EU grew from 28% in 2007 to 29% in 2008. In the UK only around 25% of post-consumer plastic waste was recovered, 20% by recycling.[24-25]

Each household in the UK generates 23 kg of waste each week, of which 4 kg (18%) is packaging. This equates to around 200 kg of packaging waste per household per year, or 147 kg per person. A total of 10.6 million tonnes of packaging waste was generated in the UK in 2007, of which 20% (2.1 million tonnes) was plastic packaging. Household waste contributes 9% of total waste, 44% of total packaging waste (4.7 million tonnes), and 64% of plastic packaging waste (1.4 million tonnes) per year. Packaging from households contributes less than 2% of total waste in England. Food and drink make up 66% of the products purchased by a typical household.[1]

Packaging cannot be eliminated because it plays a key role in preventing spoilage and damage in the supply system. The use of food packaging is beneficial in reducing food waste by greatly extending shelf-life, and is essential in a society where food is produced some distance away from where it is consumed and there is a demand for out-of-season fruit and vegetables. Food wastage in developing countries can be as high as 50% whereas in the UK the use of packaging means that only 3% of food is wasted before reaching the shops (although UK consumers then waste 30% of their food after purchase).[1] In supermarkets, loose fruits and vegetables create 26% more food waste compared with packaged produce. Packaging is also a way of carrying product information that is increasingly required by law. Packaging accounts for only 10% of the energy used in the food chain, and packaging waste has less than one tenth the environmental impact of food waste. Without plastic packaging it is estimated that the tonnage of alternative packaging materials would increase by a factor of 4.[25]

Despite the essential nature of packaging, and its low contribution to total waste, consumers believe that packaging is the top environmental problem in relation to the products they buy.[27] The efforts of consumers who, when surveyed, stated that they did “quite a lot” to minimise their waste generation were largely centred on avoiding free carrier bags and packaged foods.[28] The Waste Resources Action Programme (WRAP) lists reducing the impact of packaging as one of its four main priorities, and the Courtauld Commitment, a voluntary agreement between WRAP and major retailers, aims to design out packaging waste.[29]

In addition to consumer pressure, legislation is increasingly being applied to landfill waste reduction. The EU Landfill Directive 1999/31/EC requires a reduction of biodegradable waste sent to landfill, and the Finance Act and Landfill Tax Regulations 1996 charges a fee for each tonne of landfilled waste as a disincentive. The Packaging and Packaging Waste Directive 94/62/EC (revised by 2004/12/EC) requires volumes and weights of packaging to be the minimum necessary to maintain safety and hygiene and makes producers of waste responsible for proving their packaging is diverted from landfill. The government’s waste strategy requires an increase in recycling and composting of household waste, and the Household Waste and

Recycling Act 2003 requires all local authorities in England to provide doorstep collection for at least two types of recyclable material by 2011.[30]

Biobased plastics, particularly those which are biodegradable/compostable, offer a solution to both the principal problems associated with plastic, i.e. the use of non-renewable resources and the disposal of waste. When used for packaging they appeal to consumers because they can have the advantages that packaging brings whilst still feeling that they have "done their bit for the environment". Biobased plastics are based on renewable resources, most commonly plants, which take carbon dioxide from the atmosphere as they grow. Any carbon dioxide released after their disposal, e.g. during composting or in energy recovery from waste, does not result in a net increase of greenhouse gas because it is part of a rapidly renewing carbon cycle. Composting is a less energy intense alternative to landfill than recycling of conventional plastics.

One disadvantage of biobased plastics is their cost. Biobased plastics remain 2-4 times more expensive than their fossil-based counterparts[31] despite recent rises in the cost of fossil-based plastics (due to dependence on oil prices) and reductions in the cost of many biobased plastics as a result of improved technologies and increased levels of production. However, biobased plastics are not inherently more expensive than fossil-based plastics, and this gap is expected to narrow further in the future.[3]

A major issue preventing the uptake of biobased plastics by UK retailers is the lack of appropriate disposal routes. Biodegradability and compostability only offer an alternative to landfill if the correct facilities are available. Around 40% of people with a garden produce home compost,[32] however, some consumers do not have access to a garden and Asda estimate that only 3% of the population are regular home composters.[33] For biobased plastics which require industrial composting disposal is more problematic – around 8% of local authorities collect garden green waste for composting, but not all of these include kitchen waste.[34] Food packaging is classified as kitchen waste and can only be composted in one of the 50 industrial composting sites in the UK that are licensed for animal by-products.[32] There is, therefore, no established infrastructure for the collection of biodegradable packaging for composting in the UK. The burden of extra cost on a product cannot be justified by the retailers if the intended benefit, in this case environmentally-friendly disposal, cannot be realised.

Another concern is that consumers will not recognise biobased plastics, or understand how they should be disposed of. This can be addressed through product labelling. Until UK product labelling is clarified, consumer confusion and lack of access to the correct disposal routes mean that biobased plastics will most likely end up in landfill, where conditions are usually too dry for biodegradation to occur, negating the advantage over fossil-based plastics. If sufficient moisture is present, home compostable materials may biodegrade, anaerobically or aerobically depending on the amount of oxygen available. Anaerobic biodegradation releases methane, a greenhouse gas 23 times more harmful than the carbon dioxide released by aerobic biodegradation, potentially making biobased plastics more harmful for the environment than fossil-based plastics. Industrially-compostable bioplastics will remain in landfill without breaking down regardless of conditions. Alternatively, biobased plastics might be disposed of in plastic recycling streams, where they can cause contamination even at low (< 1%) levels.[35] PLA is recyclable, and can be recognised by infra-red automatic sorting equipment,[17] but there are as yet no collection facilities in the UK to recycle PLA.

Other, wider, issues include:

- the diversion of crops and land / water use from the production of food to raw materials for biobased plastics – the use of waste by-products is preferred to primary crops[35]

- the use of genetically modified crops or micro-organisms – PLA has historically been manufactured from genetically-modified feedstock[35]
- energy use – comparisons between fossil-based and biobased plastic productions is difficult and may be biased due to the smaller scale of the latter
- packaging weight – most EU legislation on waste disposal sets weight-based targets,[27] and biobased plastics may require a heavier gauge to provide the same performance as conventional plastics. Recent case studies of the Courtauld Commitment (2005-2010) are based mainly on lightweighting of existing packaging materials rather than the use of alternatives.[36] Phase 2 of the Courtauld Commitment was launched in March 2010 and its targets are:

  – to reduce the weight, increase recycling rates and increase the recycled content of all grocery packaging, as appropriate. Through these measures the aim is to reduce the carbon impact of this grocery packaging by 10%.

  – to reduce UK household food and drink waste by 4%.

  – to reduce traditional grocery product and packaging waste in the grocery supply chain by 5% - including both solid and liquid wastes

1.4 Standards and product labelling

Consumers cannot choose to purchase products in biobased packaging, or dispose of it appropriately, if they cannot recognise it, therefore it is important that it is clearly identified. Confusion also arises due to the lack of clarity with respect to the terminology used to describe such materials. As stated above, the terms “biobased” and “biodegradable” are often used interchangeably even though the former relates to the origin of the material and the latter to one of the means by which it can be broken down. Another term, “bioplastic”, is used by the European Bioplastics Association to describe both biobased plastics (regardless of durability) and biodegradable or compostable plastics (regardless of their origin).[3]

Similarly, the terms “biodegradable”, “degradable” and “compostable” are often interchanged, despite the different processes involved, but their distinction is essential in determining the most appropriate method of disposal/recovery.

Biodegradable [37-38] - Biodegradability is a measure of the actual metabolic, microbial conversion, under composting conditions, of the packaging sample into water, carbon dioxide and new cell biomass. There are no defined time limits or criteria to determine what can be called biodegradable, making it a very loose term (almost everything will biodegrade, given enough time). The rate of biodegradation depends on the type and thickness of material, and the environment where biodegradation takes place (e.g. in compost, fresh water, sea water, etc.). The relevant international and European Union standards are geared to specific biodegradation environments and set maximum timescales during which the material must biodegrade (e.g. BS EN 13432). To meet this standard within a maximum of 6 months, biodegradation of the test sample must generate an amount of carbon dioxide that is at least 90% as much as the carbon dioxide given off from the control / reference material.

Degradable [35,37-38] - Terms such as degradable, biodegradable, oxo-degradable and oxo-biodegradable are used to describe conventional fossil-based plastics containing additives which accelerate fragmentation into smaller particles when exposed to heat or UV radiation. Although it is claimed that these small particles biodegrade, the process is very slow (18 months or more even in optimum conditions)[35] therefore there is a risk of their accumulation in the environment. The terms cannot be verified due to the absence of a standard specification, and a less misleading term for these

materials would be “fragmentable”. No degradable plastic has yet been certified as compostable to EU standards, and they are also unsuitable for recycling because the additives make the recycled plastic unstable, and there is potential for the contamination of both these waste streams due to consumer confusion. WRAP have said that degradable plastic is not suitable for retail primary packaging,[35] the Green Alliance recommends that retailers avoid its use for all packaging applications and carrier bags[39] and European Bioplastics is distancing itself from this industry.[38]

Compostable [37] - Compostable materials biodegrade through the action of naturally-occurring micro-organisms, and do so to a high extent within a specified timeframe. They can be composted together with other organic waste without hindering the composting process and do not affect the quality of the final compost. An EU harmonised standard EN 13432 “Packaging: requirements for packaging recoverable through composting and biodegradation – testing scheme and evaluation criteria for the final acceptance of packaging” was introduced in 2000 and adopted by many of the EU member states, including the UK where it is published by the British Standards Institution as BS EN 13432. The scope of the standard is compostability and anaerobic digestability of packaging.

To comply with EN13432, a compostable material must have the following characteristics:[37,39]

- Biodegradability

  the metabolic, microbial conversion, under composting conditions, of over 90% of mass into water, carbon dioxide and biomass within 6 months.

- Disintegrability

  the fragmentation, under composting conditions, of over 90% of mass into fragments sized < 2 mm within 3 months.

- No negative effects on the composting process.

- Low levels of heavy metals.

- No adverse effect on the quality of the final compost

  including bulk density, pH, salinity, volatile solids, elemental composition and appearance.

- Support the germination and growth of plants

  germination rate and plant biomass > 90% that of plants grown in control compost.

In the UK, the Association for Organics Recycling (formerly the Composting Association) operates a certification scheme in partnership with the German certification body Din Certco, aligned to the requirements of BS EN 13432. The final product, and not just its constituent parts (polymer, additives, etc.) must pass all tests. To simplify the process, certification bodies such as Din Certco maintain a list of substances which are certified for use in the manufacture of compostable packaging. Products are usually certified to a maximum thickness, e.g. 100 µm, which may be many times thicker than the finished product. When evidence is presented that the product meets all the requirements of the standard then the product is issued with a unique certification number and permitted to carry European Bioplastics’ “compostable” seedling logo (Figure 2).[37]

European Bioplastics is the owner of this logo and licences certification bodies to award its use to manufacturers and converters of compostable packaging. A product that carries the seedling logo must also display its certification number to allow end user to trace the product to its source. Product certificates are valid for three years,

and (in the UK) the Association for Organics Recycling takes samples from the market for testing during this three year period to ensure standards are still being met.[37]

The test conditions specified by BS EN 13432 simulate industrial scale composting and anaerobic digestion, therefore the seedling logo certifies packaging as suitable for industrial composting only. At present there are no specific international or national standards for home compostability. Home composting does not reach the same temperatures as industrial composting, therefore materials that are industrially compostable may not break down under these conditions. However, the word "compostable" on packaging is assumed by consumers to be equally true for industrial composting via green waste collection and food waste collection or home compost bins,[40] so there is a need for clear labelling.

The Belgian certification body Vinçotte operate an "OK Compost" certification scheme (aligned to EN 13432) and, alongside it, an "OK Compost Home" certification scheme. The test criteria are similar to those in EN 13432 but the test temperatures and durations are different, to simulate the home composting environment (Table 6).[37]

Due to the increasing number of compostable packaging in the UK market, the Association for Organics Recycling is currently working with the National Non-Food Crops Centre (NNFCC) and WRAP towards establishing a "home compostable" certification service and logo (Figure 3) for the UK.[37,40-41] In the longer term, a logo advising the consumer to include the packaging with their food waste collection may be added (Figure 4), but this can only be used on products sold in areas where the local authority provides the appropriate collection and composting facilities.[42] These logos are designed to sit alongside the British Retail Consortium's new on-pack labelling system for recyclable materials (Figure 5).

There are a number of other "green" logos which may be found on food packaging, such as the Mobius loop, which indicates goods which are recyclable or include recycled content (Figure 6) and the Green Dot®, which denotes compliance with an authorised packaging recovery scheme in some EU member states (this scheme does not operate in the UK but products displaying this logo are sold) (Figure 7).

In March 2009 a new on-pack label scheme was launched by the British Retail Consortium with the aim of clarifying on-pack guidance for consumers to increase recycling. WRAP's "Recycle Now" logo is used along with specific details of the type of material and how it can be recycled (Figure 5).[40-41]

There is a risk that this large array of logos and information on packaging may confuse consumers. In the UK, recommendations on the use of recycling logos is included in the Defra's "Green Claims Code"[45], which offers guidance on the use of the international standard on environmental claims ISO 14021 "Environmental Labels and Declarations – Self-Declared Environmental Claims (Type II Environmental Labelling)".[43-44] The guidance given advocates the use of recognised standards and industry methods to verify any claims that are made, and discourages the use of generalised terms such as "sustainable" and "environmentally friendly". The guidance does not cover claims of biodegradability or compostability, or the use of the seedling logo; nor does Defra's "A Shopper's Guide to Green Labels".[46]

None of the logos described above relates to the biobased content of a material. The biobased content of a material can be determined by measuring the ^{14}C content, which is found in biobased carbon but not fossil carbon.[47] In order to make sustainable products more accessible to consumers, the US Department of Agriculture (USDA) recently proposed a voluntary labelling scheme associated with its BioPreferred℠ programme.[48] The programme was created in 2002 to increase the purchase and use of biobased products by the Federal Government. The label (Figure 8) would appear

on products composed wholly or significantly of biobased ingredients, i.e. meeting or exceeding USDA-established minimum biobased content requirements.

European standards for biobased products are being developed as part of the European Commission's Lead Market Initiative.[47]

1.5 UK application of biobased food contact materials

Food packaging is intended to ensure the safety and quality of food products. Food packaging is a demanding area – the properties of the polymer which must be considered when determining suitability for a particular application include gas and water vapour permeability, mechanical properties, sealing capability, thermoforming properties, resistance to water, grease, acid, UV, machinability on the packaging line, transparency, anti-fogging capacity, printability, availability and cost. Increasingly, a consideration of the source of the raw materials and the ultimate disposal of the packaging is also necessary,[21] and this is where biobased plastics have the largest potential for advantage.

For biobased packaging to appeal to a food manufacturer or retailer, it must first meet or exceed the performance characteristics of the conventional packaging. There are a variety of bioplastics, blends, laminates and coatings available with properties suitable for a wide range of food contact applications, however, they may differ significantly from conventional plastics in properties such as gloss, barrier effects, antistatic behaviour, printability and touch.[3] Consumers can be put off by the lower transparency of some biobased plastics.[49] However, the change in properties may prove to be an advantage, e.g. a higher moisture transmission is beneficial for foods such as fruit and vegetables, increasing their shelf-life.[3]

Biobased packaging can be found in several major European supermarkets, with Sainsbury's in the UK being one of the first to introduce it.[3] The emphasis for the retailers is on those bioplastics which are home compostable. Compostable packaging is particularly suitable for fresh produce because in addition to appealing to the environmentally-aware consumer, it also allows the supermarkets to dispose of unsold fruit and vegetables in their packaging, saving the cost of separation. Retailers also have more control over this area of packaging.

The Green Alliance has developed a guidance document[39] and flowchart[50] to assist in determining which foods are suitable for packaging in biobased materials. The flowchart takes into account the existing dominant choice of packaging for a particular product, along with the context of the product's use and disposal, as well as the type of food to be packaged.

Products which are considered to be good candidates for compostable packaging are:

- Foods which are to be consumed in bulk and disposed of along with the packaging, e.g. at outdoor events
- Fresh whole fruit and vegetables – the inherent properties of compostable packaging, including their high water vapour transmission rates are generally considered an advantage in the packaging of fresh fruit and vegetables as they extend shelf life
- Long shelf-life ambient products which are not moisture-sensitive, such as pasta, rice and pulses.
- Confectionary – small, lightweight packaging is rarely recycled even if recyclable. Compostable wrappers would decrease the impact of street litter, although they should not be labelled as compostable in case this encourages littering.

Products which are good candidates but are currently restricted by technical constraints are:

- Cut salads – the high water vapour transmission rate contributes to drying
- Wet produce such as cut fruits tend to soften compostable packaging, causing the seals to fail
- Shrink wrapping products, e.g. cucumbers
- Meat and dairy products
- Moisture-sensitive dry products, e.g. crisps, cereals and biscuits (again due to the high water vapour transmission rate)
- Food which requires heating in the packaging
- Modified atmosphere packaging

Biobased packaging is also not recommended where there is an established recycling stream for the existing dominant choice of packaging, e.g. drinks bottles, due to risks of consumer confusion and contamination of the recycling stream.

Information gathered from UK supermarkets shows that the largest application of biobased plastic for food packaging is currently organic fruit and vegetables. Use is restricted to organic ranges because consumers purchasing organic produce are considered to be more likely to home compost and will therefore be able to dispose of the packaging as intended. Consumers of organic products have also already indicated their willingness to pay a price premium (there may, therefore, be potential to extend application to luxury products such as “Finest” or “Extra Special” ranges). However the application to organic produce can be restricted by the use of genetically modified organisms in the production of some biobased plastics. The Soil Association does not allow any GM derived packaging for organic certified products.[39] The application of biobased plastic packaging to fruit and vegetables is particularly appropriate because consumers purchasing these types of foods will automatically have other compostable waste such as vegetable peelings, to dispose of along with the packaging. This would not necessarily apply to a consumer purchasing, for example, a ready meal in a compostable tray.

The foodstuffs most commonly packed in biobased polymers include fruit and vegetables (mainly organic produce), sweets/chocolates, cereals, tea, bakery products and pasta. Primarily these are dry foods which do not place high demands in terms of barrier properties on the polymers.

As mentioned above, in addition to their use as food packaging materials, biobased plastics can be used in the manufacture of other food contact articles such as catering supplies. High levels of recycling are very difficult to achieve at outdoor events and other places where people consume large amounts of food and drink in a short time, e.g. cafeterias. If the food contact articles used are compostable, and can be collected, these events can provide a contaminant free feedstock for composting. Large scale events such as the Glastonbury festival are already instigating policies requiring traders to use compostable tableware.[37]

A number of companies supply biodegradable food service ware in the UK, including plates, bowls, cups and cutlery. These items are often based on plant fibres rather than biobased plastics. Examples of manufacturers, trade names and material types are given in Table 7.

1.6 Migration from biobased polymers

All food contact materials and articles, including those that are biobased, contain chemicals with the potential to migrate into any foods with which they come into contact. These may be biobased monomers used in their productions, residual levels of chemicals used in the refining/processing/manufacture of the materials and any chemicals added to the formulation to provide the required characteristics of these materials. Chemical migration is defined as "the mass transfer from an external source into food by sub-microscopic processes". The extent to which any substance migrates into a foodstuff is controlled by diffusion processes which are subject to both kinetic and thermodynamic control. These processes can be described by Fick's second law and the extent of any chemical migration is dependent on:

- the nature of the food contact material
- the nature of the foodstuff
- the nature of the migrating substance
- the concentration of the migrating substance
- the extent of contact (direct or indirect) between the food contact material and the foodstuff
- the duration of the contact
- the temperature of the contact

1.6.1 Legislation

The safety of all food contact materials and articles, irrespective of their source, is controlled in the European Union (EU) by the Framework Regulation (EC) No. 1935/2004.[51] It defines what is meant by food contact materials and articles and sets basic requirements that they must fulfil. These requirements are set to ensure food safety and consumer protection, and to harmonise rules to prevent barriers to trade within the EU. Article 3 of this Regulation, which specifies general requirements, states:

> "1. Materials and articles, including active and intelligent materials and articles, shall be manufactured in compliance with good manufacturing practice so that, under normal or foreseeable conditions of use, they do not transfer their constituents to food in quantities which could:
>
> (a) endanger human health;
>
> or
>
> (b) bring about an unacceptable change in the composition of the food;
>
> or
>
> (c) bring about a deterioration in the organoleptic characteristics thereof.
>
> 2. The labelling, advertising and presentation of a material or article shall not mislead the consumers."

The Framework Regulation also empowers the European Commission to set requirements for specific materials. Specific Directives lay down requirements for Plastics (Directive 2002/72/EC, as amended).[52] Rules for migration testing of such materials and articles are described in Directive 82/711/EEC, as amended[53] and Directive 85/572/EEC, as amended.[54] Plastics, according to Directive 2002/72/EC, as amended, are defined as organic macromolecular compounds obtained by

polymerisation, polycondensation, polyaddition or any other similar process from molecules with a lower molecular weight or by chemical alteration of natural macromolecules. Therefore it may be considered that each of the types of biobased polymers that meet this description fall within this definition. It should be noted that regenerated cellulose film (a biobased material) is specifically excluded from this Directive and instead is controlled by Directive 2007/42/EC relating to materials and articles made of regenerated cellulose film intended to come into contact with foodstuffs.[55]

To protect the consumer, any biobased materials classed as plastics must be manufactured using authorised starting substances and additives. Authorisation to use a starting substance or additive is provided by the European Food Safety Authority (EFSA) following submission of a petition. This positive list of authorised substances is given in Directive 2002/72/EC, as amended, which also sets restrictions for their use such as specific migration limits. In addition to these restrictions there is also a basic requirement that plastic food contact materials should be inert. To this purpose an overall migration limit is set at 60 mg/kg of packaged food. Furthermore the Directive contains rules for the enforcement of the migration limits.

Biobased starting substances and additives included in the authorised list - Polymers extracted from biomass that are currently used commercially for food contact applications in the UK are based on either cellulose or starch. Both of these substances are authorised as starting substances for use in the manufacture of food contact plastics. Neither has been assigned a specific migration limit and therefore their migration is controlled by the overall migration limit of 60 mg/kg.

Poly(lactic acid) is the most common biobased plastic obtained by chemical synthesis using biobased monomers which is used for food contact applications in the UK. Lactic acid is listed as an approved monomer for food contact applications in Directive 2002/72/EC, as amended. Lactic acid has not been assigned a specific migration limit and therefore its migration is controlled by the overall migration limit of 60 mg/kg.

Polymers produced by micro-organisms which have potential food contact applications (although no current UK applications were found) include polyhydroxyalkanoates (PHAs), which are a specific class of polyesters produced by bacterial fermentation of sugar. Article 5 of Directive 2002/72/EC states: “Only the products obtained by means of bacterial fermentation listed in Annex IV may be used in contact with foodstuffs.” The copolymer 3-hydroxybutanoic acid-3-hydroxypentanoic acid is the only substance included in this Annex, therefore this is the only permitted starting material from this source, providing it meets a list of extensive specifications. The polymer is produced by the bacteria and it must be extracted, isolated from the bacteria and purified. The extensive specifications necessary reveal the fact that this purification process can never be as complete for a polymer as it can be for a low molecular weight monomer. Monomers can be purified by highly effective physical processes including distillation or recrystallisation but these procedures cannot be applied at all (distillation) or applied with such effectiveness (recrystallisation) to polymers. It is likely that other biodegradable polymers derived from the action of micro-organisms should also have specifications placed on them to control the possibility of chemical migration to a food in contact.

No information was obtained on the identities of the additives used in the manufacture of the biopolymers as this is proprietary information. However it has been reported that all PLA polymer additives have appropriate EU national regulatory status (the identities of these additives are not provided).[56] Any substances permitted for use in food contact plastics (as defined in Directive 2002/72/EC, as amended) may also be used in the manufacture of biobased plastics. Some additives are available from renewable resources. These include:

- fatty acids, their salts, esters and amides – used as lubricants, processing aids, heat stabilisers, emulsifiers
- pine derivatives: pine tar, rosin, terpene – used as tackifiers and processing aids
- vulcanized vegetable oils or factices – used in rubber formulations
- phenol derivatives – used as antioxidants
- liquid depolymerised natural rubber – used as a cross-linkable polymeric plasticizer
- epoxidized soya bean oil – used as a plasticizer

1.6.2 Migration potential

1.6.2.1 Overall migration

Test methods for overall migration have been standardized by CEN. In these tests plastic materials and articles are exposed to food simulants using conditions equivalent to the worst foreseeable use. The simulants and exposure conditions to use are defined in the EU legislation (Directive 82/711/EEC, as amended and 85/572/EEC, as amended). It may be expected that if biobased polymers are used in direct contact with moist and aqueous foods, or are tested with aqueous simulants, then high migration of the water-soluble polymers will occur. Overall migration tests using simulant D (olive oil or its alternatives) are not possible with materials that are very moisture-sensitive such as biobased polymers. The CEN standard methodology is not applicable to these systems. In such cases substitute test media can be used – this is defined in the existing legislation and test methods have been standardized for the use of the substitute test media.

1.6.2.2 Specific migration

As mentioned above, many of the starting substances for biobased materials are authorised for use in food contact plastics with the only restriction on their migration being the overall migration limit. Therefore the main areas of interest regarding migration of specific substances from these materials are:

- the migration of additives used to make the biobased material suitable as a food contact material
- the migration of source contaminants present in the natural material
- co-polymerisation with other starting substances such that the functional properties of biobased materials are improved by this chemical reaction
- the migration of any non-intentionally added substances (NIAS) formed during the manufacture or subsequent storage of the packaging material

Migration of additives - Any chemicals that are added to the plastic to enable it to fulfill its function as a food contact material may migrate into foods in which they come into contact. For example plasticisers are needed to overcome the natural brittleness of starch and make materials with usable physical properties. Glycerol, sorbitol, other low molecular weight polyols and urea may be used to achieve this and, as low molecular weight constituents, these substances have the potential to migrate. Similarly the addition of plasticisers to cellulose acetate films (with 53 to 56% acetyl groups) is required to improve biodegradability – it is reported that when common plasticisers are substituted by specific esters and other low molecular weight components (at least

30% by weight), the plastic then has the same desirable thermoplastic properties but will decompose in soil or water.[57] The migration of these low molecular weight plasticisers could occur if placed in contact with food. PLA can be plasticised with monomeric or oligomeric lactic acid, and made into blown films, injection moulded articles, coatings etc.[58]

Migration of source contaminants - Another potential source of migrants could be contaminants of the biobased source material, which might remain in the finished plastic. For example if fusarium toxins are present on maize which is harvested for use in the production of a biobased material, will they remain in the final material or article when it is used to package the foodstuff? Similarly will any pesticide residues remain in the final product?

Migration of chemicals used to modify polymer function - The functional properties of natural polymers are commonly improved by chemical reaction. Examples of these treatments include:

- pre-treatments of jute fibres with sodium hydroxide and then copolymerization with acrylonitrile[59]
- using an acrylic acid-grafted polyethylene-octene copolymer as a compatibiliser of corn starch-reinforced metallocene polyethylene-octene elastomer blends[60]
- modifying starch by reaction with acrylamide

Acrylic acid, acrylonitrile and acrylamide are themselves very reactive molecules. The point of concern would be if these treatments of the natural polymer gave rise to oligomers or reaction side products that had a low molecular weight and the potential to migrate. As mentioned above, all food contact materials and articles must meet the criteria included in Article 3 of the Framework Regulation. This is achieved by taking into account both (a) known ingredients and (b) their impurities, reaction products and breakdown products. Therefore it is not only the migration of the known chemicals in the formulation that need to be controlled but also any impurities, reaction products or breakdown products that form. It is now clarified in recital 13 of Directive 2007/19/EC (the fourth amendment to Plastics Directive 2002/72/EC) that there is a general requirement to assess the safety of all potential migrants, including impurities, reaction and breakdown products. Therefore the issue of oligomers and reaction products has been recognised for conventional plastics and similar information would need to be provided for biodegradables.

As mentioned earlier, the extent to which any substance migrates into a foodstuff is controlled by diffusion processes and the extent of any chemical migration is dependent on the factors listed in Section 1.6.

The conditions for the majority of the current food contact applications of biobased materials are not aggressive, i.e. they are usually used to package dry foods such as fruit and vegetables, stored for short periods of time at ambient temperature. The contact between the food and the packaging generally occurs over a small area of the overall surface. Therefore for such applications it may be expected that only low levels of migration will occur. However, PLA used in disposable cups makes continuous contact with the beverage, in many cases at elevated temperature. These are conditions which may be expected to give rise to elevated migration of any starting substances and additives that are present.

1.6.3 Reported chemical migration from biobased materials

There are only a small number of reports/scientific publications describing migration from biobased materials. These are described below.

1.6.3.1 Migration from cellulose-based materials

Migration of plasticisers from regenerated cellulose film (RCF), has been reported.[61,62] The plasticisers were propylene glycol (PG), mono- (MEG), di- (DEG) and triethylene glycol (TEG). MEG and DEG were withdrawn from this specific use in 1985 and industry proposed replacing them with PG, TEG, polyethylene glycol (PEG), glycerol and urea. These softeners are polar, water-soluble molecules, and so migration would not be favoured given that these polymers tend to be used to package dry foods only.

1.6.3.2 Migration from starch-based materials

Avella *et al*.[63] determined the extent of migration of minerals from biodegradable starch/clay nanocomposite films developed for use in food packaging. The experimental work involved putting vegetable samples (lettuce and spinach) into bags made from either potato starch or potato starch-polyester blend, and their respective composites with nano-clay. The bags were heated at 40℃ for 10 days, cooled, acclimatised, and migration of minerals determined by atomic absorption method after digestion of the vegetables. The results of the tests indicated an insignificant trend in the levels of iron and magnesium in the vegetables, but a consistent increase in the amount of silicon (the main component of nano-clay). The concentrations of silicon detected in the vegetables were 16-19 ppm in the case of nano-clay composites of potato starch, and potato starch-polyester bend, compared to 13 ppm for the same polymers without nano-clay, and around 3 ppm in control vegetables. The migrants determined were all associated with the nano-clay.

1.6.3.3 Migration from PLA

Studies have shown that the level of lactic acid monomer that migrates to food from packaging containers is much lower than the amount used in common food ingredients. When PLA was tested for migration into 8% ethanol solution and olive oil, under test conditions of 10 days at 43℃, the overall migration was 0.85 and 0.15 mg/dm^2 into the two simulants, respectively. The migration studies were conducted on samples of the polymer following guidelines issued by the Food and Drug Administration. The migrate was comprised of lactic acid, lactoyl lactic acid (acyclic dimer), trimer, and lactide (cyclic dimer). Dimers and oligomers hydrolyse in aqueous systems (i.e. *in vivo*) to lactic acid, which is a common food ingredient that has been shown to be safe in food at levels far in excess of any small amount that might result from the intended uses of PLA.[64] PLA stored for 15 days at 40°C with two different food simulants, 10% ethanol and 95% ethanol, showed that the total amount of substances that migrate from PLA polymer into simulant is lower than the average daily intake of lactic acid from all proposed uses as indirect food additives (22 mg per day).[65]

In another study,[66] the overall migration from a PLA film was less than 1 mg/dm^2 into 3% acetic acid solution, 15% ethanol solution and olive oil, under test conditions of 10 days at 40ºC, and into isooctane under conditions of 30 minutes at 40ºC. Mutsuga *et al*.[67] subjected different types of PLA sheet to migration tests under various conditions and the lactic acid, lactide and oligomers content of the migration solutions were determined using LC/MS. PLA was found to be stable at 40℃ for 180 days; the

total migration level of lactic acid, lactide and oligomers was 0.028 - 1.5 mg/dm^2. When stored at 60℃ for 10 days PLA decomposed and the total migration level was increased to 0.073 - 284 mg/dm^2.

2. AIMS AND OBJECTIVES

Objective 01. *Gather information on the use of biobased materials for food contact*

Objective 02. *Obtain samples*

Objective 03. *Sample characterisation*

Objective 04. *Identification of potential migrants*

Objective 05. *Ageing tests*

Objective 06. *Specific migration tests into foods and food simulants*

Objective 07. *Comparison of migration data for foods and food simulants*

Objective 08. *Assess the applicability of the overall migration test methods for biobased materials*

Objective 09. *Final report*

3. SAMPLES

Numerous manufacturers and potential users of biobased food contact materials were contacted. From these contacts and by purchasing samples from the supermarkets and the internet thirteen different samples were obtained (Table 8). Each was assigned a unique sample number. The material types identified are also given in Table 8.

4. IDENTIFICATION OF POTENTIAL MIGRANTS

Although it is well recognised that the use of materials derived from biomass is desirable it is important to ensure that the safety of the foods packaged within is not compromised. The identities of any potential migrants in the packaging materials were determined using a suite of analytical methods selected to detect substances with a molecular weight below 1,000 Dalton. This molecular weight was selected in view of the relative ease of migration of such substances and their toxicological significance. The materials and extracts thereof were analysed by headspace GC-MS (to detect any volatile substances present), by GC-MS (to detect any semi-volatile substances) and by LC-TOF-MS (to detect any polar and non-volatile substances).

Many (but not all) biobased materials are intended to be biodegradable/compostable. This means they have an intrinsic sensitivity to degradation by water and microorganisms. Clearly the functional properties should not degrade during the anticipated life of the products otherwise the packaging or food contact article could not perform its intended function. However in terms of the chemical composition it is also possible that high humidity and temperature could alter the substances present resulting in additional substances being formed with the potential to migrate into foods with which they come into contact. Therefore the materials were also subjected to the same suite of tests following storage for 4 months at 40℃ and in the presence of water

and heat (5 mL water added to the samples stored in sealed glass vials for 5 days, the water was allowed to evaporate and then the vial containing the samples was sealed and stored for 4 months at 40℃).

4.1 Sample preparation

4.1.1 Sample preparation for HS-GC-MS

The biobased packaging materials (before and after ageing) were cut into small pieces (~0.5 x 0.5 cm). A known mass of each biobased packaging material was transferred into a 10 mL headspace GC-MS vial. Material was added to half fill the vial and the mass was accurately recorded. A further 2 vials were then prepared containing the same mass of each sample. 100 µL of a 1 mg/mL solution of d_{10}-ethylbenzene in ethanol was added to each vial and the vials were tightly capped prior to analysis

4.1.2 Sample preparation for GC-MS

The biobased packaging materials (before and after ageing) were cut into small pieces (~0.5 x 0.5 cm). A known mass (1 g) of each biobased packaging material was transferred into a glass vial. Ethanol (20 mL) and d_{10}-benzophenone internal standard (100 µL of a 1 mg/mL solution) were added and the vials were capped and shaken for 18 hours at room temperature. A portion of the extraction solvent was transferred to a glass vial suitable for GC-MS analysis. Triplicate samples were prepared in this way. Additional samples were extracted in the same way but using isooctane as the extraction solvent.

4.1.3 Sample preparation for LC-TOF-MS

The biobased packaging materials (before and after ageing) were cut into small pieces (~0.5 x 0.5 cm). A known mass (1 g) of each biobased packaging material was transferred into a glass vial. Ethanol (20 mL) and d_{10}-benzophenone internal standard (100 µL of a 1 mg/mL solution) were added and the vials were capped and shaken for 18 hours at room temperature. A portion (5 mL) of the extraction solvent was evaporated to dryness and reconstituted in 1 mL acetonitrile. Triplicate samples were prepared in this way. Additional samples were extracted in the same way but using isooctane as the extraction solvent.

4.2 Sample analysis

4.2.1 Analysis by HS-GC-MS

The samples were incubated for 30 minutes at 80℃. The resulting volatile compounds were analysed using an Agilent 6980 gas chromatograph (Agilent, Palo Alto, CA, USA) coupled with an Agilent 5973inert mass selective detector by splitless injection of 1 mL of the headspace gas onto an DB-VRX capillary column (30 m x 0.25 mm i.d. x 1.2 µm film thickness; J & W Scientific, Folson, Ca, USA). Following injection, the oven was held at 40℃ for 3 minutes and then raised at 10℃/ minute to 280℃ and held for 5 minutes. The injector was held at 250℃. Helium (1 mL/min constant flow) was employed as the carrier gas. The MS was operated in electron impact mode with scanned monitoring between 40 – 450 amu.

4.2.2 Analysis by GC-MS

The sample solvent extracts were analysed by GC-MS using an Agilent 6980N gas chromatograph (Agilent, Palo Alto, CA, USA) coupled with an Agilent 5973inert mass selective detector. Splitless injection of 1 µL of extract was carried out into a DB-5MS capillary column (30 m x 250 µm i.d., 0.25 µm film thickness; J & W Scientific, Folson, Ca, USA). Following injection the oven was held at 60℃ for 5 minutes and then raised at 10℃/minute to 320℃. The injector was held at 250℃. Helium (1 mL/min constant flow) was employed as the carrier gas. The MS was operated in electron impact mode with scanned monitoring between 40 - 600 amu.

4.2.3 Analysis by LC-TOF-MS

The sample solvent extracts were analysed by LC-TOF-MS using an Agilent LC/MSD TOF (Agilent, Santa Clara, CA, USA) consisting of a 1200 Series LC and a time of flight mass spectrometer. Two separate LC-MS methods were used to increase the coverage of compounds that could be detected. In both cases separation was facilitated using an Agilent ZORBAX Eclipse XDB-C18, 100 x 2.1 mm, 3.5 µm column. For positive mode electrospray the mobile phase consisted of 0.1% aqueous acetic acid (channel A) and acetonitrile (channel B). For negative mode electrospray the mobile phase was 5 mM ammonium formate at pH 5.5 (channel A) and 0.1% 5 mM ammonium formate at pH 5.5 in acetonitrile (channel B). The mobile phase gradient for both positive and negative mode electrospray was the same: a starting mixture of 80% A and 20% B that changed to 50% B over 15 minutes. This was held for 5 minutes and then went to 100% B at 30 minutes. This was held for a further 10 minutes before returning to the original mobile phase composition and the column was equilibrated for 10 minutes prior to the next injection. The flow rate was 0.2 mL/min with an injection volume of 5 µL.

TOF-MS analysis was carried out in positive and negative mode electrospray with nebuliser pressure 45 psi, capillary 4000 V, gas temperature 325^{o}C, drying gas flow at 10 L/min, skimmer 60 V, fragmentor 150 V and octopole RF voltage 250 V. The mass range measured was 100 – 1100 m/z. The TOF-MS data produced was processed using Agilent MassHunter software. The ion species allowed in positive electrospray mode were $[M+H]^+$, $[M+NH_4]^+$, $[M+Na]^+$ and $[M+K]^+$, and those allowed in negative electrospray mode were $[M-H]^-$, $[M+Cl]^-$ and $[M+CH_3COO]^-$. The lowest relative peak height and absolute peak height were 5% and 10000 counts respectively. Atoms included in the molecular formula finder were C (0 – 60), H (0 – 120), O (0 – 30), N (0 – 10), S (0 – 5), Cl (0 – 5), P (0 – 5) and Br (0 – 5).

Following LC-TOF-MS analysis the data generated was processed using Agilent MassHunter Qualitative software. This software employs algorithms to automatically identify all the detectable compounds or molecular features in accurate mass data even when analysing very complex mixtures. Key among these are the Molecular Feature Extractor and Empirical Formula Generation algorithms. Molecular Feature Extractor is a data-mining tool that generates a list of molecular features with retention time, neutral mass and ion abundance. All of the related ions of a molecular feature (isotopes, charge states, adducts and multimers) are grouped together, and areas of noise are removed. As the name suggests, the Empirical Formula Generator calculates potential empirical formulae for TOF-MS peaks. It uses accurate mass MS, isotope spacing, and mass peak abundance information to decrease the number of potential formulae generated and then lists them in order of likeliness using a unique scoring system. The software generates a report describing the peaks detected, including retention time, accurate mass, predicted empirical formulae and score.

4.3 Results

As mentioned above the samples were analysed as received and following a period of forced ageing at elevated temperature and at elevated temperature with high humidity. Heating the samples (no water) did not result in any apparent physical changes (by eye). The effects of the elevated temperature and humidity storage conditions on the physical appearance of the samples are given in Table 9.

The results described below give the proposed identities (best library matches) and estimated concentrations (determined by assuming and 1:1 peak area response with the internal standard) of the substances detected in the materials tested as received, after storage at elevated temperature and after storage at high humidity and temperature.

Information found on the restrictions on the use of the substances proposed to be present in the food contact materials is given below. This information was derived by searching existing EU legislation for plastics (although some of the materials tested would not fall under the classification of a plastic Directive 2002/72/EC, as amended, was used as a guide), Joint FAO/WHO Expert Committee on Food Additives (JECFA) evaluations and the internet (the substances and the 'words' TDI, ADI and restriction where entered into the internet search engine.

4.3.1 Volatile substances detected by headspace GC-MS

Tables 10 to 22 show the estimated concentrations and best library matches for each of the peaks detected by headspace GC-MS of the samples analysed as received. A cut-off concentration of 1 mg/kg was applied (a rough rule of thumb a level of 1 mg/kg in packaging is assumed to give a maximum of 10 µg/kg migration potential[68]). No substances were detected using this technique when the samples were analysed after storage. This is proposed to be due to reduced sensitivity of the method as a consequence of the presence of water in the samples that will have altered the partitioning between the samples and the headspace gas. Therefore it was not possible to directly compare the chromatograms. Each of the substances detected by headspace GC-MS are discussed below:

Tetrahydrofuran has been assigned a specific migration limit (SML) of 0.6 mg/kg in Directive 2002/72/EC.

n-Butyl acetate is listed as an approved additive for plastics in Directive 2002/72/EC, as amended and no restriction is given. Thus the overall migration limit of 60 mg/kg in the food applies. No information was found for isopropyl or n-propyl acetate.

Pentanal, hexanal and 2-pentylfuran are permitted for use as flavouring agents. The JECFA evaluation for hexanal as a flavouring compound gives the acceptable daily intake (ADI) as acceptable.

The ADI for xylene is given as not established by JECFA.

For the cellulose films toluene and isopropyl acetate as solvents have been assigned a restriction in the Regenerated cellulose film Directive (2007/42/EC)[55] such that the total quantity of the substances (solvents) should not exceed 0.6 mg/dm^2 of the coating on the side in contact with the foodstuff. Taking into account the mass of the films per unit area this restriction was not exceeded.Of specific interest are the perfluorinated compounds detected in sample S09-012093. This sample is a bagasse bowl. Perfluorinated compounds have previously been reported to be present in grease-proof packaging such as popcorn bags and therefore it is proposed that they are applied to these bowls to impart the same properties. Although only one peak was detected above the cut-off concentration of 1 mg/kg when the ions associated with this class of

compound (m/z 69 and m/z 131) were specifically searched for six peaks were detected (Figure 9).

4.3.2 Semi-volatile substances detected by GC-MS

Tables 23 – 44 compare the semi-volatile substances detected in the solvent extracts of the samples before ageing and after heat with and without water. Where no data is given there were no peaks detected in the chromatograms of the extracts above the cut-off concentration. The same cut-off concentration as for the headspace GC-MS was applied. Any restrictions associated with the substances detected are described below:

Sample S09-012077 and Sample S09-012078 – A range of carboxylic acids, carboxylic acid esters, aldehydes, alkenes and alkanes were detected in the solvent extracts.

N,N-Dimethyl-1-tetradecanamine, N,N-dimethyl-1-hexadecanamine and N,N-dimethyl-1-octadecanamine were detected in the extracts stored with water. Such tertiary amines are used as chemical intermediates for the manufacture of quaternary ammonium compounds. Quaternary ammonium compounds have been reported to be used as surface modifiers on organophilic clays such as bentonite to make them compatible with plastic materials. The quaternary ammonium compounds are frequently used to modify starch based materials.

9-Octadecenoic acid (Z)-, 2-hydroxy-1-(hydroxymethyl)ethyl ester was also detected in the extracts of both samples. The use of quaternary ammonium compounds may be accompanied by the use of an amphiphilic copolymer. Those listed in the patent [69] are PEG 30 dipolyhydroxystearate and polyglyceryl-2-dipolyhydroxystearate. Therefore it is possible that this compound is related to their use.

Di-(2-ethylhexyl) phthalate was detected in Sample S09-012078 aged in the presence of water. This substance is included in the list of permitted additives in Directive 2002/72/EC, as amended. It should only be used as a plasticiser in repeated use materials and articles contacting non-fatty foods or as a technical support agent in concentrations up to 0.1% in the final product. An SML of 1.5 mg/kg food simulant has been assigned. The concentration detected in the sample was 74.1 mg/kg, i.e. much less than 0.1%.

Sample S09-012079 – Sample S09-012079 is a starch containing co-extruded film. The co-extruded material is believed to be a regenerated cellulose film (RCF). Glycerol, a permitted softener for use in RCF films[55] was detected in the extracts. Softeners are permitted at levels of “not more than 27% (w/w) in total”. Therefore the levels detected (< 5000 mg/kg) are acceptable. No migration limit is assigned for this substance as it is permitted for use as a food additive (E number E422).

Benzoic acid, nonanoic acid, (Z)-9-octadecenamide, cis-11-eicosenamide, (Z)-13-docosenamide and erucic acid are approved additives for plastics in Directive 2002/72/EC, as amended and no restriction is given. Thus the overall migration limit of 60 mg/kg in the food applies.

Glycerol esters with acetic acid (e.g. triacetin) are included on the list of monomers and starting substances which may be used in the manufacture of plastic materials and articles in Directive 2002/72/EC, as amended, no restriction is given and therefore any migration should comply with the overall migration limit.

Diisopropyl naphthalenes (DIPN) and diisobutyl phthalate (DiBP) are both typical contaminants of recycled cartonboard and therefore their presence here is not expected. The levels measured in the extracts (DIPN = 6.9-11.3 mg/kg and DiBP = 10.5-21.3 mg/kg) are low. The film weight is 1 g/dm^2. Therefore the concentrations of

DIPN and DiBP in the film (11.3 and 21.3 µg/kg respectively) are equivalent to 11.3 and 21.3 µg/dm^2. Assuming the conventional food contact ratio of 1 kg of food exposed to 6 dm^2 of material then the migration potential is 68 µg/kg for DIPN and 128 µg/kg for DiBP. A TDI of 0.024 mg/kg b.w./day has been proposed for DIPN[70] which is equivalent to 1.44 mg/kg food. A restriction for DiBP of 1 mg/kg in food has been proposed. The worst case migration (i.e. 100% transfer) from the material into any food with which it comes into contact would not exceed these proposed restrictions. 2-(Phenylmethoxy) naphthalene is reported to be used in coatings for paper/board materials, no restrictions were found.

Restrictions are in place for the use of di-(2-ethylhexyl) phthalate in plastic food contact materials and articles. Di-(2-ethylhexyl) phthalate should only be used as a plasticiser in repeated use materials and articles contacting non-fatty foods or as a technical support agent in concentrations up to 0.1% in the final product. An SML of 1.5 mg/kg food simulant has been assigned. The concentration detected in the film was in the range 15.5 - 19.1 mg/kg, i.e. much less than 0.1%.

Other substances proposed to be present in the extracts and for which no information was found were; 1,6-dioxacyclododecane-7,12-dione, diethyl phthalate, isooctyl dodecanoate, p-terphenyl and 2-naphthyl benzoate.

Some of the substances detected in the extracts of this sample may have been derived from the transportation of the material to our laboratory. The sample was received in a recycled cartonboard sleeve and therefore it is possible that transfer of these substances from this sleeve to the sample occurred during transportation and storage.

Samples S09-012080, S09-012081 and S09-012082 – All three films are cellulose based. All contain the permitted softener glycerol at less than 27% as is permitted in the RCF Directive.[55]

Acetic acid and a series glycerol-like substances were detected in Sample S09-012080. Acetic acid and glycerol esters are permitted for use as additive in RCF films at total levels of < 1% in uncoated RCF products. The levels estimated in the films did not exceed this restriction.

Sample S09-012081 contained numerous alkanes as well as glycerol, two glycol related substances, 1,6-dioxacyclododecane-7,12-dione and the natural terpene dehydroabietene.

Only glycerol was detected in sample S09-012082.

Sample S09-012083 – No restrictions were found for 1,6-dioxacyclododecane-7,12-dione and 1-propene-1,2,3-tricarboxylic acid, tributyl ester. Acetyl tributyl citrate is a plasticiser. It is permitted for use as an additive for plastic food contact materials and articles and no restriction is given. It's presence in this sample is proposed to be in the ink applied to the external surface of the cup.

Phosphoric acid, diphenyl 2-ethylhexyl ester has been assigned an SML of 2.4 mg/kg. The concentrations estimated to be present in the cup were 2.0-2.1 mg/kg. Each cup weighs 12.3 g and hold 300 mL of beverage. Therefore assuming total transfer the worst case concentration would be 2.1 mg/kg in the cup resulting in 0.086 mg/kg in the beverage, i.e. less than the SML for this substance.

Sample S09-012092 – A series of unspecified alkanes were detected in the solvent extracts of the cups. The other substances detected are all natural compounds that are expected to be derived from the cassava starting material.

Sample S09-012093 – DiBP and di-n-butyl phthalate (DBP) were detected in the isooctane extracts after storing with water and heat. As mentioned above a restriction for DiBP of 1 mg/kg in food has been proposed. DBP should only be used as a

plasticizer in repeated use materials and articles contacting non-fatty foods or as a technical support agent in polyolefins in concentrations up to 0.05% in the final product. The SML of 0.3 mg/kg food simulant is derived from the TDI of 0.05 mg/kg b.w./day.

The bowl weighs 12.0 g and hold 250 mL of foodstuff. DiBP was detected at a concentration of 1.1 mg/kg which would be equivalent to a migration of 0.05 mg/kg food assuming total transfer, i.e. less than the proposed restriction. The concentration of DBP was estimated to be 1.6 mg/kg, assuming total transfer and that a 60 kg adult consumes 1 kg of food served in this product then the exposure would be 0.001 mg/kg b.w./day.

Sample S09-012094 – DL-lactide monomer was detected in the extracts of the PLA performed prior to storage.

Sample S09-012095 – DL-lactide formed from lactic acid monomer was detected in the extracts of the PLA performed prior to storage. Lactic acid is permitted for use as a monomer and no restriction is given.

Hexanoic acid is an approved additive for plastics in Directive 2002/72/EC, as amended and no restriction is given. Thus the overall migration limit of 60 mg/kg in the food applies.

Other palmitic acid and stearic acid esters are listed in Directive 2002/72/EC, as amended and no restrictions are given as they are expected to hydrolyse to innocuous substances and by analogy the same may be expected to be true for the isopropyl palmitate and isopropyl stearate proposed to be present here.

Sample S09-012096 – DL-lactide formed from lactic acid monomer and (Z)-13-docosenamide were detected in the extracts of the sample performed prior to storage. Lactic acid is permitted for use as a monomer and no restriction is given. (Z)-13-Docosenamide is listed as an approved additive.

Sample S09-012097 – DL-lactide formed from lactic acid monomer and (Z)-13-docosenamide were detected in the extracts. Lactic acid is permitted for use as a monomer and no restriction is given. (Z)-13-Docosenamide is listed as an approved additive.

Stearic acid esters are listed in Directive 2002/72/EC, as amended and no restrictions are given as they are expected to hydrolyse to innocuous substances and by analogy the same may be expected to be true for the ethyl stearate (ethyl octadecanoate) proposed to be present here.

Ethyl eicosanoate is another fatty acid ester that may be expected hydrolyse to innocuous substances.

In most cases other substances were detected in the extracts for which no good library match was obtained. It should be noted that all identities were proposed based on matches with MS libraries and that they have not been confirmed by the analysis of the authentic standards.

In most cases the total ion chromatograms derived from the analysis of the samples that had been stored at elevated temperature were similar to those derived from the original screening analysis. The addition of water to many of the materials resulted in dissolution of the polymer and in doing so broke the polymer backbone thereby allowing greater interaction with the material and more complete extraction of the substances present. For example for sample S09-012077 a corn starch tray only gave one peak in the total ion chromatogram of the solvent extracts analysed as received. Addition of water to the polymer resulted in dissolution and numerous polar compounds were released into the ethanol extraction solvent. Clearly this has

implications in terms of any migration that takes place from such materials as if wet contact were to occur between the foodstuff and the polymer then the number of potential migrants and the concentrations at which they migrate may increase. However those materials that did dissolve in the water are only intended for contact with dry foodstuffs.

4.3.3 Polar and/or non-volatile substances detected by LC-TOF-MS

Tables 44 to 67 show the accurate masses and proposed identities for the substances detected in the LC-TOF-MS analysis of the solvent extracts analysed in positive and negative mode. Where no data/table is given then no substances were detected in the extracts that were not present in the procedural blanks. Identities were proposed by searching an (incomplete) in-house database of food contact material starting substances and searching the internet for the molecular formulae generated. The presence of the peaks in the extracts analysed either as received, following storage at elevated temperature and following storage at elevated temperature in the presence of water is indicated. Annex 1 gives the masses and all proposed formulae for each peak.

The concentrations of the substances present in the extracts could not be determined. Instead the peak areas are provided (Tables 68 to 85) to allow comparisons to be made between the levels present in the samples treated with heat and the samples treated with heat in the presence of water. As a consequence of day to day differences in instrument sensitivity and the lack of suitable internal standard it was not possible to compare the peak area data as the analyses were not performed at the same time. For negative electrospray mode ionisation a suitable internal standard was not included. For positive electrospray mode ionisation d_{10}-benzophenone was added as internal standard. However, this peak was not detected in the total ion chromatograms of any sample. It is believed that this is because of the high concentrations of the components in the extracts that are dominating the ionisation process, causing suppression of the internal standard.

Of the substances detected the majority for which identities could be proposed were oligomeric units or fatty acids. Octylphenol ethoxylate was detected in the extracts of Sample S09-012079. This is a surfactant that is biodegradable, e.g. [71].

4.3.4 Changes in chemical composition following treatment

Although changes in composition may have occurred it is also possible that the dissolution of the polymer occurring as a result of the addition of water may have altered the extraction properties. Either:

> The more polar compounds may have remained in the ‘wet' polymer resulting in an apparent decrease or loss of a given analyte.
>
> or
>
> The addition of water may have broken the polymer backbone thereby allowing greater interaction with the material and more complete extraction of the substances present resulting in an apparent increase in or formation of a given analyte.

Therefore the results presented here do not prove that the observed changes occurred as a consequence of the presence of the water.

5. MIGRATION STUDIES

It was reported in FSA-commissioned project (A03040) on an investigation of the nature and extent of biodegradable polymers used in direct food contact applications that 'The methods of test for migration, using food simulants, are likely to be directly applicable to testing most biodegradable polymers. Since the methods of test are supposed to be directly related to the actual conditions of use in contact with food, and mimic these, then if a biodegradable polymer is suitable for a particular application in contact with food then a correctly specified test procedure should be applicable also….'.

Therefore although the levels of the substances detected in the solvent extracts did not give rise to concern in terms of their migration potential migration experiments were carried out to compare the extent of the migration into foods with that into conventional simulants as defined in the legislation for plastics to confirm that these methods are applicable to these materials. The samples, simulants, foods, migrants and test conditions selected for the migration studies are summarised in Table 86.

5.1 Migration from Sample S09-012077

Of the substances detected in the extracts of Sample S09-012077 N,N-dimethyl-1-tetradecanamine, N,N-dimethyl-1-hexadecanamine, N,N-dimethyl-1-octadecanamine and 9-octadecenoic acid (Z)-, 2-hydroxy-1-(hydroxymethyl)ethyl ester were selected for the migration studies.

5.1.1 Exposure to food simulant

Sample S09-012077 is a chocolate mould. The food simulant specified for chocolate in Directive 85/572/EEC, as amended, is olive oil applying a reduction factor of 5. 1 dm^2 of the mould was immersed in 50 mL of olive oil for 10 days at 40℃ (conditions equivalent to long term storage at ambient temperature as defined in Directive 82/711/EEC, as amended). Total immersion was chosen as the mode of testing as this allowed a higher surface area to volume ratio to be used.

5.1.2 Exposure to foodstuff

Milk chocolate was melted in a glass bowl by heating in a microwave for 3 minutes with occasional stirring. The chocolate was poured in the mould hot and was then stored for 10 days at 40℃ (as above). 125 g of chocolate was added to one mould of total area ~ 3.5 dm^2.

5.1.3 Extraction and analysis by GC-MS

Following the exposure period the mould was removed from the oil, the oil was shaken to mix and a portion (10 g) was transferred to a 40 mL glass vial. 100 µL of a 10 µg/mL solution of d_{10}-benzophenone internal standard was added to the oil which was then extracted with acetonitrile (10 mL) by shaking at room temperature for 4 hours. A portion of the acetonitrile extract (5 mL) was transferred to a clean vial and concentrated to 0.5 mL under a gentle stream of nitrogen. The chocolate was removed from the mould, homogenised and extracted in the same way.

Blank (unexposed) olive oil and blank (unexposed) chocolate were extracted in the same way.

Blank (unexposed) olive oil and blank (unexposed) chocolate were overspiked with N,N-dimethyl-1-octadecanamine (one of the potential migrants selected for this sample) to achieve concentrations of 100 and 500 µg/kg in the oil/chocolate and were extracted in the same way.

The concentrated extracts were analysed by GC-MS (as section 4.2.2) but the MS was operated in selected ion mode monitoring m/z 82, 110 and 192 for the d_{10}-benzophenone internal standard and m/z 58 and 297 for the N,N-dimethyl-1-octadecanamine. Ion m/z 58 was also monitored for N,N-dimethyl-1-tetradecanamine and N,N-dimethyl-1-hexadecanamine and ions m/z 55, 67 and 264 were monitored for 9-octadecenoic acid (Z)-, 2-hydroxy-1-(hydroxymethyl)ethyl ester to allow these substances to be detected if present in the extracts (no standards were available for overspiking purposes).

The unexposed and exposed simulant and food were also analysed with the MS operated in full scan mode to detect the presence of any other substances migrating for which specific ions were not monitored.

5.1.4 Results

Responses were seen for the ions associated with the N,N-dimethyl-1-tetradecanamine, N,N-dimethyl-1-hexadecanamine and N,N-dimethyl-1-octadecanamine in the extracts of the olive oil exposed to the test sample that were not present in the blank oil (Figure 10). An interference was observed in the channel that gave the greatest response (m/z 58) close to the retention time of the N,N-dimethyl-1-octadecanamine. Therefore quantification was achieved using the less sensitive ion (m/z 297 monitored for this compound). Quantification was achieved by comparing the response in the exposed sample with that in the overspiked blank oil samples. The average concentration (three samples were exposed) of N,N-dimethyl-1-octadecanamine detected in the exposed oil was 2.1 mg/kg.

No standards were obtained for N,N-dimethyl-1-tetradecanamine, N,N-dimethyl-1-hexadecanamine therefore the concentrations present were determined by comparing the response (m/z 58) with that of the N,N-dimethyl-1-octadecanamine (taking into account the aforementioned interference). The estimated concentrations of these two substances in the oil were 93 and 963 µg/kg respectively.

The estimated concentrations of these three substances extracted from the sample in the screening exercise equate to a worst case migration (assuming 100% transfer) of 0.5, 7.2 and 11.1 mg/kg for N,N-dimethyl-1-tetradecanamine, N,N-dimethyl-1-hexadecanamine and N,N-dimethyl-1-octadecanamine respectively. Therefore between 13 and 20% migration was observed into the simulant.

The ions for 9-octadecenoic acid (Z)-, 2-hydroxy-1-(hydroxymethyl)ethyl ester were also monitored but were not detected in the exposed olive oil samples. As no authentic standard was obtained the detection limit could not be estimated.

None of the potential migrants were detected in the exposed chocolate as a consequence of the high levels of co-extractives present.

No differences were observed in the total ion chromatograms for the blank and exposed simulant and the blank and exposed chocolate. This is due to the decreased sensitivity of this technique and the number of substances extracted from the unexposed oil/chocolate that could mask any migrating substances.

5.2 Migration from Sample S09-012079

Of the substances detected in the extracts of Sample S09-012079 glycerol, di-(2-ethylhexyl) phthalate and (Z)-13-docosenamide were selected for the migration studies. Sample S09-012079 is a film that may be used for dry foodstuffs. No food simulant is given in Directive 85/572/EEC, as amended, to test migration into dry foods however the use of Tenax has been proposed and therefore this adsorbent was chosen here.

5.2.1 Exposure to food simulant

Sample S09-012079 is a thin film. As mentioned above Tenax was selected as the food simulant to mimic the migration into dry foods such as cereal. 0.75 dm^2 of the film was interspersed in 4 g of Tenax and stored for 10 days at 40℃ (conditions equivalent to long term storage at ambient temperature as defined in Directive 82/711/EEC, as amended).

5.2.2 Exposure to foodstuff

1.5 dm^2 of the film (Sample S09-012079) was interspersed in 30 g of cereal and stored for 10 days at 40℃ (conditions equivalent to long term storage at ambient temperature as defined in Directive 82/711/EEC, as amended).

5.2.3 Extraction and analysis by GC-MS

Following the exposure period the film was removed from the Tenax and the simulant was mixed with a metal spatula. A portion (1.0 g) was transferred to a 40 mL glass vial. 100 µL of a 10 µg/mL solution of d_{10}-benzophenone internal standard was added to the Tenax which was then extracted with diethyl ether (10 mL) by shaking at room temperature for 4 hours. A portion of the diethyl ether extract (5 mL) was transferred to a clean vial and concentrated to 0.5 mL under a gentle stream of nitrogen.

Following the exposure period the film was removed from the cereal and the cereal was mixed with a metal spatula. A portion (3.0 g) was transferred to a 40 mL glass vial. 200 µL of a 10 µg/mL solution of d_{10}-benzophenone internal standard was added to the cereal which was then extracted with dichloromethane (20 mL) by shaking at room temperature for 4 hours. A portion of the dichloromethane extract (10 mL) was transferred to a clean vial and concentrated to 1 mL under a gentle stream of nitrogen.

Blank (unexposed) Tenax and blank (unexposed) cereal were extracted in the same way.

Blank (unexposed) Tenax and blank (unexposed) cereal were overspiked with glycerol, di-(2-ethylhexyl) phthalate and (Z)-13-docosenamide to achieve concentrations of 1 and 10 mg/kg in the Tenax and 0.3 and 3.3 mg/kg in the cereal and were extracted in the same way.

The concentrated extracts were analysed by GC-MS (as section 4.2.2) but the MS was operated in selected ion mode monitoring m/z 82, 110 and 192 for the d_{10}-benzophenone internal standard and m/z 43 and 61 for glycerol, m/z 149, 167 and 279 for di-(2-ethylhexyl) phthalate and m/z 59, 72 and 337 for (Z)-13-docosenamide. Concentrations of the analytes in the Tenax and in the foodstuff were determined using matrix matched standards.

The unexposed and exposed simulant and food were also analysed with the MS operated in full scan mode to detect the presence of any other substances migrating for which specific ions were not monitored.

5.2.4 Results

Di-(2-ethylhexyl) phthalate and (Z)-13-docosenamide both migrated into the Tenax and the cereal. From the concentration measured in the simulant/foodstuff the migration assuming the conventional food contact ratio of 6 dm^2 of film per kg of food was calculated. The migration of di-(2-ethylhexyl) phthalate into the Tenax was 173 µg/kg and into the cereal was 52 µg/kg. For this analyte the migration into the Tenax was greater than that into the foodstuff and therefore this adsorbent may be considered to be a suitable simulant, i.e. it overestimated the migration into the dry foodstuff. The migration of (Z)-13-docosenamide into the Tenax was 973 µg/kg and into the cereal was 944 µg/kg. Here the migration into Tenax was a good approximation for that into the cereal. No glycerol was detected in either the exposed Tenax or cereal.

The estimated concentration of the (Z)-13-docosenamide extracted from the sample in the screening exercise equates to a worst case migration (assuming 100% transfer) of 11.9 mg/kg. Therefore less than 10% migration was observed into both the simulant and the food. The levels of di-(2-ethylhexyl) phthalate measured were of a similar order of magnitude as measures were not taken to minimise contamination by this ubiquitous compound then the low levels present in the Tenax, food and film are expected to be derived from the environment surrounding the sample rather than from the sample itself.

The total ion chromatograms for the blank and exposed Tenax and the blank and exposed cereal are compared in Figures 11 and 12. Additional substances were detected in the extracts of the exposed Tenax/cereal that were not present in the blank. These substances are marked in these Figures. In addition to the (Z)-13-docosenamide mentioned above, 1,6-dioxacyclododecane-7,12-dione, 1-phenylmethoxynaphthalene and two unknowns were detected. The migration was estimated by comparing the response in the total ion chromatogram with that of the d_{10}-benzophenone internal standard. The migration levels (applying the conventional food contact ratio) were estimated to be:

1,6-Dioxacyclododecane-7,12-dione migration into Tenax = 306 µg/kg

1,6-Dioxacyclododecane-7,12-dione migration into cereal = 133 µg/kg

1-Phenylmethoxynaphthalene migration into Tenax = 521 µg/kg

1-Phenylmethoxynaphthalene migration into cereal = 182 µg/kg

The unknown eluting at ~ 22 minutes migration into Tenax = 2967 µg/kg. This unknown was not detected in the TIC of the cereal due to the presence of an interference in the chromatogram. This ‘unknown’ is the substance that was detected at the highest concentration in the solvent extracts.

The unknown eluting at ~ 26.5 minutes migration into Tenax = 71 µg/kg. This unknown was not detected in the TIC of the cereal due to the presence of an interference in the chromatogram.

The unknown eluting at ~ 28.5 minutes migration into Tenax = 34 µg/kg. This unknown was not detected in the TIC of the cereal due to the presence of an interference in the chromatogram.

For these migrants in this sample the results obtained indicate that Tenax is a suitable simulant for migration into dry foods.

5.3 Migration from Sample S09-012081

Of the substances detected in the extracts of Sample S09-012081 glycerol was selected for the migration studies. Sample S09-012081 is an RCF film that may be

used for dry foodstuffs. No food simulant is given in Directive 85/572/EEC, as amended, to test migration into dry foods however the use of Tenax has been proposed and therefore this adsorbent was chosen here. The exposures, extraction and analysis were as described above for Sample S09-012079.

5.3.1 Results

Glycerol was detected in the Tenax exposed to the film however the calibration standards were not appropriate (glycerol gave a poor peak shape in the GC-MS chromatogram) and therefore the migration was only estimated for this substance. No migration was observed into the cereal.

The total ion chromatograms for the blank and exposed Tenax are compared in Figures 13. Additional substances were detected in the extracts of the exposed Tenax/cereal that were not present in the blank. These substances are marked in these Figures. The concentrations were estimated relative to the internal standard:

3-Methoxy-1,2-propanediol = 67 µg/kg

Glycerol = 341 µg/kg

Unknown = 18 µg/kg

Pentacosane = 31 µg/kg

Octacosane = 18 µg/kg

5.4 Migration from Sample S09-012092

Of the substances detected in the extracts of Sample S09-012092 the alkanes were selected for the migration studies. Sample S09-012092 is a cup which may be expected to come into contact with acidic fruit juices or alcoholic beverage. The food simulants specified for these beverages in Directive 85/572/EEC, as amended, are 3% (w/v) aqueous acetic acid and 10% (v/v) aqueous ethanol. Apple juice was selected as the foodstuff.

5.4.1 Exposure to food simulant

The cup was filled with the simulant (125 mL) to within 0.5 cm of the rim, covered with a glass dish and stored for 2 hours at 70℃. These conditions are defined as equivalent to hot fill in the EU legislation for plastics (used here as a guide).

5.4.2 Exposure to foodstuff

Apple juice was heated in a microwave until just boiling. The cup was filled with the hot apple juice (125 mL) and was allowed to stand for 2 hours at room temperature.

5.4.3 Extraction and analysis by GC-MS

Following the exposure period the simulant/apple juice was removed from the cup and a portion (10 g) was transferred to a 40 mL glass vial. 100 µL of a 10 µg/mL solution of d_{10}-benzophenone internal standard was added to the liquid which was then extracted with dichloromethane (10 mL) by shaking at room temperature for 4 hours. A portion of the dichloromethane extract (5 mL) was transferred to a clean vial and concentrated to 0.5 mL under a gentle stream of nitrogen.

Blank (unexposed) 10% ethanol, 3% acetic acid and apple juice were extracted in the same way.

Blank (unexposed) 10% ethanol, 3% acetic acid and apple juice were overspiked with dodecane and heptadecane (selected as representative alkanes but not present in the sample) to achieve concentrations of 100 and 500 µg/kg in the simulants/apple juice and were extracted in the same way.

The concentrated extracts were analysed by GC-MS (as section 4.2.2) but the MS was operated in selected ion mode monitoring m/z 82, 110 and 192 for the d_{10}-benzophenone internal standard and m/z 57, 71 and 85 to screen for the alkanes of interest. Concentrations of the analytes in the Tenax and in the foodstuff were determined using matrix matched standards.

The unexposed and exposed simulant and food were also analysed with the MS operated in full scan mode to detect the presence of any other substances migrating for which specific ions were not monitored.

5.4.4 Results

No alkane migration was detected into any of the simulant or apple juice samples. This may be a consequence of the low solubility of these non-polar compounds in the aqueous beverage and simulants.

Comparing the total ion chromatograms for the blank and exposed simulants and the blank and exposed apple juice two peaks were detected in the extracts of the exposed samples that were not present in the blanks. The best library matches for these substances were vanillin (a natural product from vanilla) and 4-hydroxy-1-methoxycinnamaldehyde both detected in test sample that had been stored at elevated temperature in the presence of water. It was visible by eye that strong interaction occurred between the aqueous beverages/simulants and the cups and although little migration was observed the cups were not suitable for the long term storage of the beverages.

5.5 Migration from Sample S09-012093

Of the substances detected in the extracts of Sample S09-012093 the perfluorinated compounds detected by headspace GC-MS were selected for the migration studies. Sample S09-012093 is a bowl which may be expected to come into contact with acidic soups or fatty sauces under conditions equivalent to hot fill. The food simulants specified for these foodstuffs in Directive 85/572/EEC, as amended, are 3% (w/v) aqueous acetic acid and olive oil. Tomato soup was selected as the foodstuff.

5.5.1 Exposure to food simulant

The bowl was filled with the simulant (250 mL) to within 0.5 cm of the rim, covered with a glass sheet and stored for 2 hours at 70℃. Thes e conditions are defined as equivalent to hot fill in the EU legislation for plastics (used here as a guide).

5.5.2 Exposure to foodstuff

Tomato soup was heated in a glass bowl a microwave until just boiling. The bowl was filled with the hot soup (250 mL) and was allowed to stand for 2 hours at room temperature.

5.5.3 Extraction and analysis by GC-MS

Following the exposure period the simulant/soup was removed from the bowl and a portion (4 g) was transferred to a 10 mL glass headspace vial. 100 µL of a 10 µg/mL solution of d_{10}-ethylbenzene internal standard was added and the vial was capped.

Blank (unexposed) 3% acetic acid, olive oil and soup were prepared in the same way.

The samples were analysed by headspace GC-MS (as section 4.2.1). No standards could be obtained and therefore any migration was established by specifically searching for the ions characteristic of these compounds (m/z 69 and 131).

5.5.4 Results

No perfluorinated compounds were detected in the exposed simulants/soup. This may be a consequence of the sensitivity of the analysis in these matrices. The estimated detection limit based on the response of the internal standard was 7 µg/kg in 3% acetic acid and 10 µg/kg in the soup.

5.6 Migration from Sample S09-012097

Of the substances detected in the extracts of Sample S09-012093 (Z)-13-docosenamide was selected for the migration studies. Sample S09-012093 is a bag that may be used for dry foodstuff such as sandwiches. No food simulant is given in Directive 85/572/EEC, as amended, to test migration into dry foods however the use of Tenax has been proposed and therefore this adsorbent was chosen here.

5.6.1 Exposure to food simulant

Sample S09-012093 is a food bag. As mentioned above Tenax was selected as the food simulant to mimic the migration into dry foods such as bread. 0.75 dm^2 of the film was interspersed in 4 g of Tenax and stored for 24 hours at 40℃ (conditions equivalent to storing a sandwich for between 4 and 24 hours at ambient temperature as defined in Directive 82/711/EEC, as amended).

5.6.2 Exposure to foodstuff

One slice of bread was stored in 1 bag (6.8 dm^2 of the film) 24 hours at 40℃ (i.e. storing a sandwich for between 4 and 24 hours at ambient temperature).

5.6.3 Extraction and analysis by GC-MS

Following the exposure period the film was removed from the Tenax and the simulant was mixed with a metal spatula. A portion (1.0 g) was transferred to a 40 mL glass vial. 100 µL of a 10 µg/mL solution of d_{10}-benzophenone internal standard was added to the Tenax which was then extracted with diethyl ether (10 mL) by shaking at room temperature for 4 hours. A portion of the diethyl ether extract (5 mL) was transferred to a clean vial and concentrated to 0.5 mL under a gentle stream of nitrogen.

Following the exposure period the bread was removed from the bag and the bread was homogenised using a food mixer. A portion (3.0 g) was transferred to a 40 mL glass vial. 100 µL of a 10 µg/mL solution of d_{10}-benzophenone internal standard was added to the bread which was then extracted with dichloromethane (10 mL) by shaking at room temperature for 4 hours. A portion of the dichloromethane extract (5 mL) was

transferred to a clean vial and concentrated to 0.5 mL under a gentle stream of nitrogen.

Blank (unexposed) Tenax and bread were extracted in the same way.

Blank (unexposed) Tenax and bread were overspiked with (Z)-13-docosenamide to achieve concentrations of 1 and 10 mg/kg in the Tenax and 0.3 and 3.3 mg/kg in the bread and were extracted in the same way.

The concentrated extracts were analysed by GC-MS (as section 4.2.2) but the MS was operated in selected ion mode monitoring m/z 82, 110 and 192 for the d_{10}-benzophenone internal standard and m/z 59, 72 and 337 for (Z)-13-docosenamide. Concentrations of the analytes in the Tenax and in the foodstuff were determined using matrix matched standards.

The unexposed and exposed simulant and food were also analysed with the MS operated in full scan mode to detect the presence of any other substances migrating for which specific ions were not monitored.

5.6.4 Results

No migration of the (Z)-13-docosenamide was observed into Tenax or the bread.

There were no differences in the total ion chromatograms obtained from the analysis of the blank and exposed Tenax and the blank and exposed bread.

5.7 Changes in the composition of the materials exposed to simulants/foods

Changes were observed in Samples S09-012092 and S09-012093 when exposed to both foods and simulants. Both materials softened when in contact with the food/simulant and after the two hour exposure period both samples started to leak through the base. However as the same changes were apparent with both foods and aqueous based simulants then it was considered that the simulants mimicked the contact with the foodstuff.

5.8 Migration studies summary

There was little measurable migration from the materials tested. Where migration was observed the simulants defined in the legislation overestimated or provided a good approximation to the migration into foods and therefore the suggestion made in FSA-commissioned project (A03040) on an investigation of the nature and extent of biodegradable polymers used in direct food contact applications that 'The methods of test for migration, using food simulants, are likely to be directly applicable to testing most biodegradable polymers. Since the methods of test are supposed to be directly related to the actual conditions of use in contact with food, and mimic these, then if a biodegradable polymer is suitable for a particular application in contact with food then a correctly specified test procedure should be applicable also….' was confirmed albeit for the limited number of material/migrant/simulant/food combinations studied here.

6. ASSESSING THE APPLICABILITY OF THE OVERALL MIGRATION TEST METHODS FOR BIOBASED MATERIALS

As mentioned above it was reported in FSA-commissioned project (A03040) on an investigation of the nature and extent of biodegradable polymers used in direct food

contact applications that 'The methods of test for migration, using food simulants, are likely to be directly applicable to testing most biodegradable polymers…….. However the text continued to state 'One caveat is that tests for overall migration may not be technically possible for humidity-sensitive materials'.

Tests for overall migration into olive oil require preconditioning to constant weight. To establish whether or not conditioning is required the test specimen is transferred from an environment at 80% relative humidity to one at 50% relative humidity and recording the weight. If the difference between the masses of the test specimen as determined is greater than 2 mg/dm², then conditioning of the test specimens will be necessary before carrying out the overall migration test.

As expected for these absorbent materials all of the samples tested Sample S09-012077, Sample S09-012079, Sample S09-012080, Sample S09-012092 and Sample S09-012093 changed in mass between the two chambers. If constant weight cannot be reached within 5 days then the CEN standards describing the test method for overall migration into olive oil by total immersion (EN1186 Part 2) states:

> "NOTE 1: Long conditioning periods are not satisfactory due to oxidation of the olive oil which may occur upon prolonged conditioning."

Therefore this method is not applicable for such samples. For these samples conditioning by vacuum drying should be followed. This procedure is defined in EN1186 Part 2. Therefore if biobased polymers were to be subjected to the same overall migration tests as defined for plastics then this conditioning method should be followed.

As mentioned earlier in this report exposure to water and aqueous simulants/foods altered the appearance of several of the samples included in this project (Table 9). In several cases similar observations were made when the samples were used in contact with the aqueous foodstuffs and therefore it is recommended that samples should be labelled with respect to the appropriate use conditions to prevent deformation during use.

7. CONCLUSIONS

Although numerous, material specific, substances were detected in the thirteen packaging materials tested there was little measurable migration into food simulants and foods. Where migration was observed the simulants defined in the legislation (for plastics) overestimated or provided a good approximation to the migration into foods. This was in agreement with the suggestion made in FSA-commissioned project (A03040) on an investigation of the nature and extent of biodegradable polymers used in direct food contact applications that 'The methods of test for migration, using food simulants, are likely to be directly applicable to testing most biodegradable polymers…..' albeit for the limited number of material/migrant/ simulant/food combinations studied here. The limitations of the overall migration methods as defined in the CEN standards highlighted in the aforementioned FSA-commissioned project (A03040) that test using olive oil as a simulant for overall migration may not be technically possible for humidity-sensitive materials were confirmed and for such samples conditioning by vacuum drying should be followed when carrying out migration testing using olive oil. Exposure to water and other aqueous simulants altered the appearance of several of the samples included in this project. Therefore the limitation of these material types in terms of their ability to maintain their shape and perform their function should be recognised and it is recommended that materials and articles should be labelled to define the contact conditions for which they will function.

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9. SOURCES OF INFORMATION

To identify the types of biobased materials that are currently available the information sources listed below were searched.

Internet search engine:

Google	www.google.co.uk

Organisation websites:

Association for Organics Recycling	www.organics-recycling.org.uk
European Bioplastics	www.european-bioplastics.org
Green Alliance	www.greenalliance.org.uk
Let's Recycle	www.letsrecycle.com
The National Non-Food Crops Centre	www.nnfcc.co.uk
Plastics Europe	www.plasticseurope.org
Waste and Resources Action Programme	www.wrap.org.uk

Packaging magazines and trade journals:

Biomass Magazine	www.biomassmagazine.com
Bioplastics Magazine	www.teamburg.de/bioplastics
Biobased News	www.biobasednews.com
Flexible Packaging	www.flexpackmag.com
Packaging Digest	www.packagingdigest.com
Packaging Europe	www.packagingeurope.com
Packaging News	www.packagingnews.co.uk
The Packaging Professional	www.iom3.org/content/packaging-professional
Packaging Today	www.packagingtoday.co.uk
Retail Packaging magazine	www.retailpackagingmag.co.uk

Keywords included in the search:

Biobased, Biopolymer, Bioplastic
Food Packaging, Food Contact
Migration
Starch
Cellulose
Polylactic acid
Polyhydroxyalkanoate PHA PHB PHBH PHBV
Biobased PE
Biobased PET
Compostable
Biodegradable
EN13432
Din Certco
Vincotte
Plastic waste
Landfill legislation
Alcan Jordans
Amcor NaturePlus Ceramis
Arkema Rilsan Plantamid
BASF Ecoflex Ecovio Ultramid
Biograde
Biolice
Biomatera
Biomer
Biop Biopar

Biopac
Bioresins
Biosphere
Biostarch
Biotec Bioplast
Braskem
Cardia
Celebration
Cereplast
Cerestech Cereloy
Clarifoil
Coca-cola Plantbottle
Compostable Packaging
Coopbox Italia NaturalBox
Dow Chemical Company
DSM Tianjin Green Bio
DuPont Biomax Sorona Cerenol
Earthcycle
Eastman Tenite
Eco-Products
Evercorn
Fabri-Kal Greenware
FkuR Biograde Bioflex
Futerro Loopla
Grace Biotech Grace Bio
Grenidea Agroresin
Harbin Livan
Hisun
Huhtamaki Bioware
Innovia Natureflex
Instone Verdepack
International Paper Ecotainer
Kaneka
Limagrain Biolice
Mazzuccheli Bioceta
Meredian Nodax
Mitsubishi Biogreen

Natureworks Ingeo
Novamont Mater-Bi Origo-Bi
NVYRO
Paperfoam
PHB Industrial Biocycle
Plantic
Potatopak
Purac
Renewable Products Earthshell
Rodenburg Solanyl
Roots Biopack
Shell Corterra
Sidaplax Earthfirst
Solvay
Stanelco Biome
Sustainable Adhesives Biotak
Synbra Biofoam
Telles Metabolix Mirel
Tepha Tephaflex Tephelast
Tianan Biologic Enmat
Treofan Biophan
Valueform Trugreen
Vegeplast Vegemat
Vertupak Procurasell Vegasse
Wentus Wenterra

Scientific literature search engine:
Web of Science
Food Sci.&Tech.Abs
Foodline
Pascal 1973-2010
MEDLINE(R) 1950-2010
Adis Clinical Trials Insight 1990-2010
Polymer Online
Plastic Properties Database 1999
RAPRA Rubber & Plastics 1972-2010
CA SEARCH(R) 1967-2010

Keywords included in the search:

Biobased, Biopolymer, Bioplastic

Food Packaging, Food Contact

Migration

Starch

Cellulose

Polylactic acid

Polyhydroxyalkanoate

Biobased manufacturers contacted:

Several manufacturers, converters and suppliers of bio-based resins and products were contacted, or their websites searched, for further information on the food contact applications of their products and their availability in the UK.

UK retailers contacted:

Asda

Co-op

Marks and Spencer

Morrisons

Sainsbury's

Tesco

Waitrose

Figure 1. An overview of the different types of biobased polymers (adapted from [6,7])

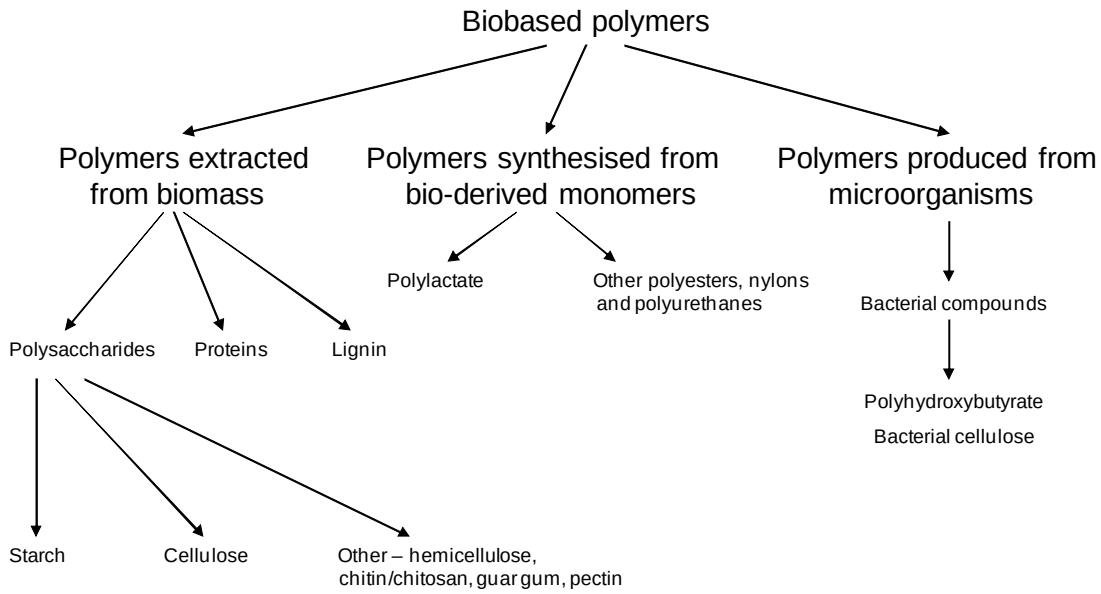


Figure 2. European Bioplastics logo for materials certified compostable to EN 13432

Figure 3. Proposed logo for UK home compostable certification scheme

Figure 4. Proposed logo for UK food waste collection

Figure 5. Example of British Retail Consortium on-pack recycling information

Figure 6. Mobius loop indicating (left) recyclable material and (right) material containing x% recycled content

Figure 7. Green Dot® indicating compliance with authorised packaging recovery scheme

Figure 8. Labelling of the USDA BioPreferred℠ programme

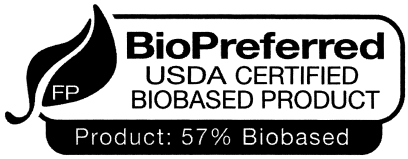


Figure 9. Chromatograms showing the ions associated with perfluorinated compounds detected in sample S09-012093

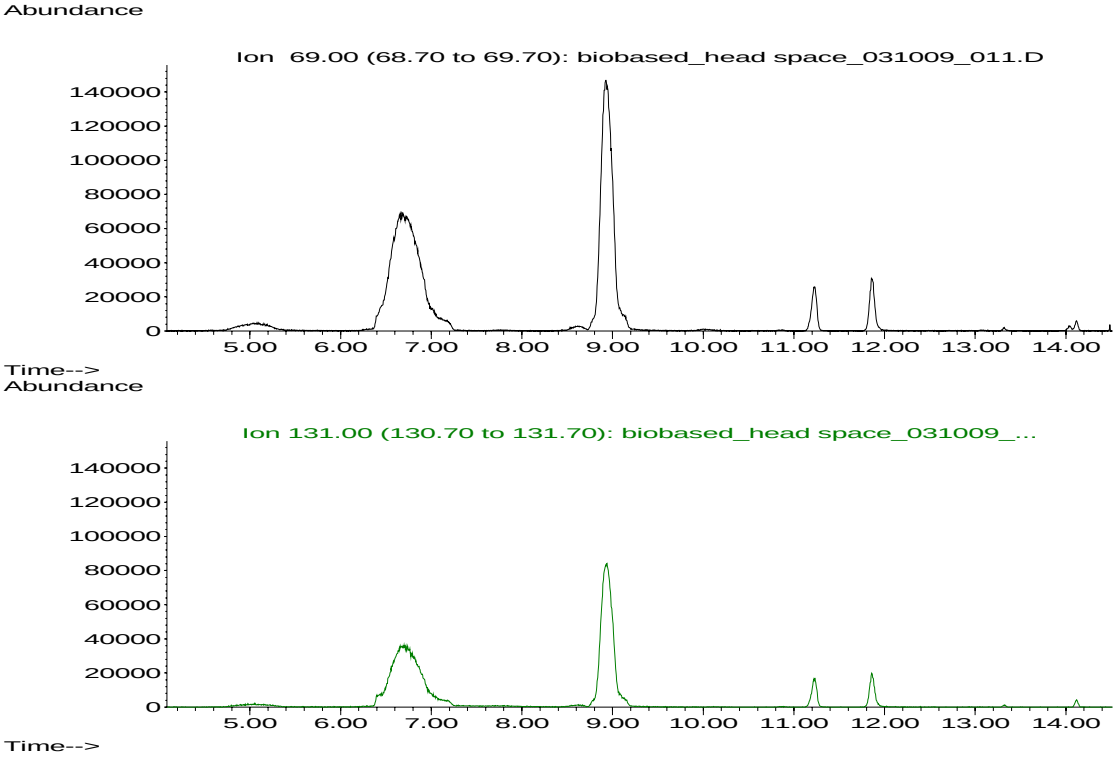


Figure 10. Chromatograms showing the ions associated with N,N-dimethyl-1-tetradecanamine, N,N-dimethyl-1-hexadecanamine and N,N-dimethyl-1-octadecanamine in a blank olive oil and olive oil exposed to Sample S09-012077

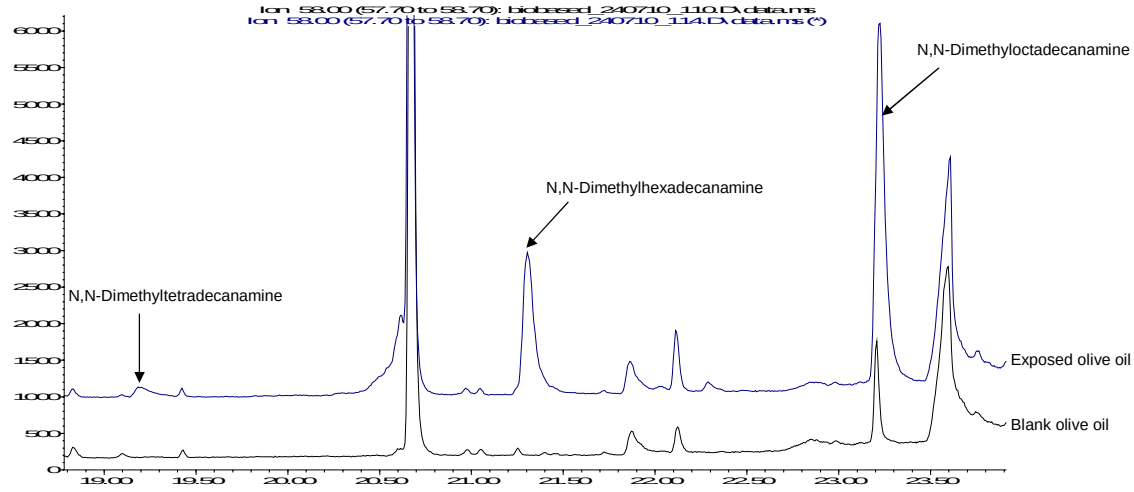


Figure 11. Comparing the total ion chromatograms for the blank Tenax and the Tenax exposed to Sample S09-012079

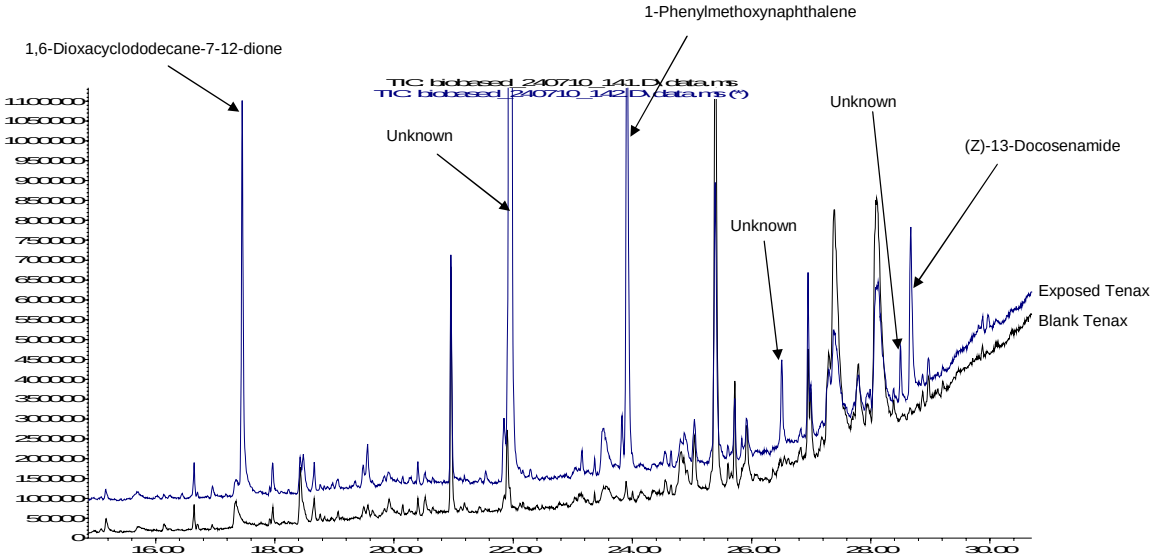


Figure 12. Comparing the total ion chromatograms for the blank cereal and the cereal exposed to Sample S09-012079

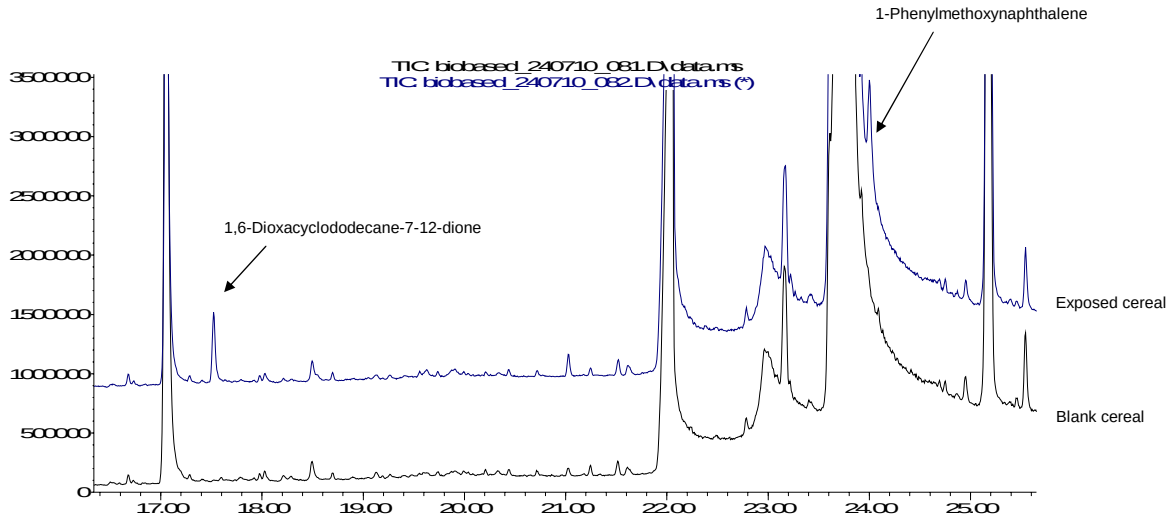


Figure 13. Comparing the total ion chromatograms for the blank Tenax and the Tenax exposed to Sample S09-012081

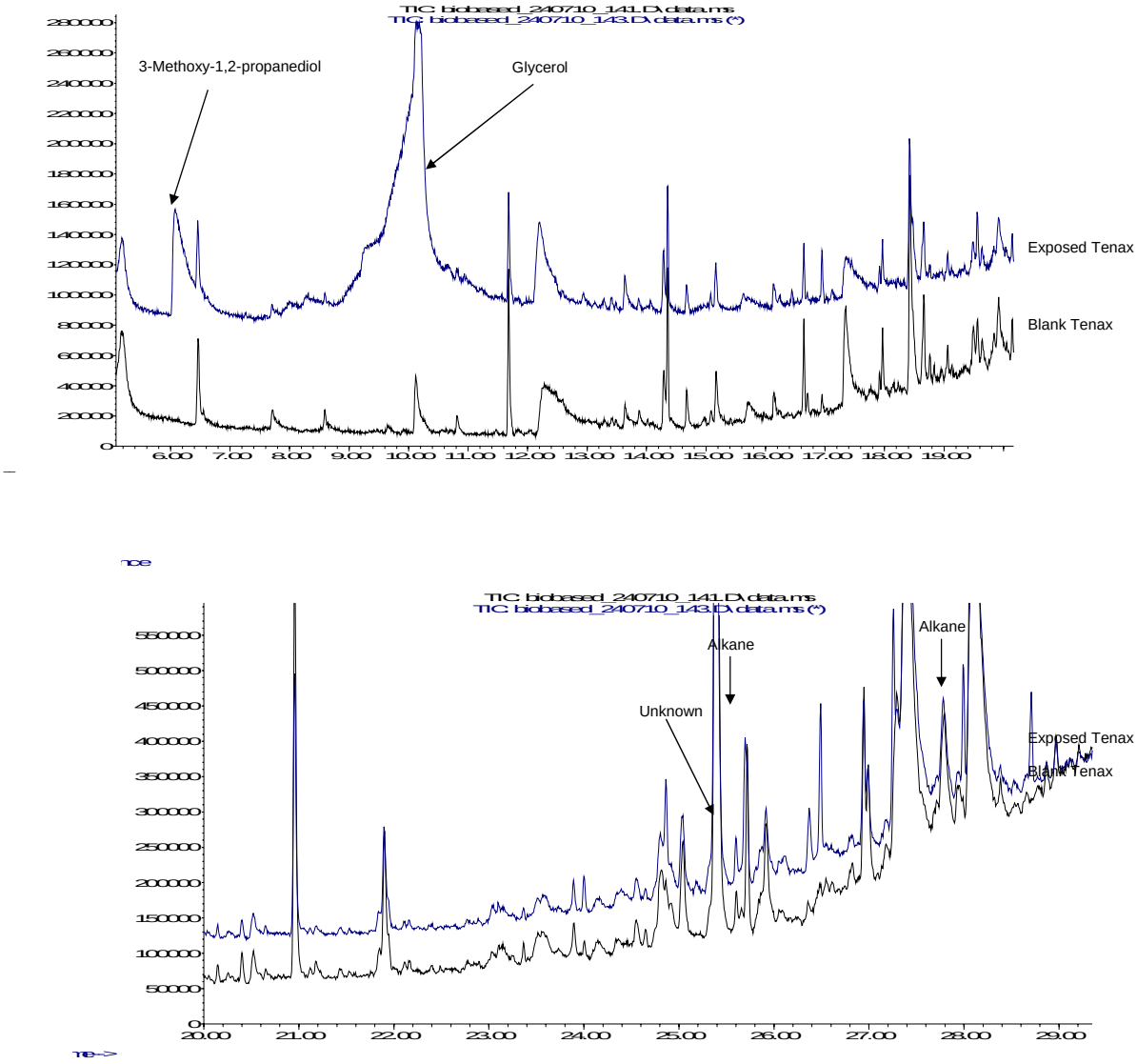


Table 1. Manufacturers of commercially available starch-based polymers

Company	**Trade Name**
Amcor Flexibles / Alcan Packaging	NaturePlus
Biop Biopolymer Technolgies AG	Biopar®
Biosphere Industries LLC	Biosphere®, Renew-a-Pack®, BlueWare®
Biostarch	Biostarch®
Biotec GmbH & Co.	Bioplast®
Cardia Bioplastics (formerly Biograde)	Cardia Biohybrid™
Cereplast Inc.	Cereplast Compostable®, Cereplast Hybrid®
Cerestech Inc.	Cereloy™
Dupont	Biomax® TPS
Evercorn Inc. / Japan Cornstarch Co. Ltd.	EverCorn™
Grace Biotech	Grace-Bio
Harbin Livan Biodegradable Product Co. Ltd.	Livan
Limagrain Céréales	Biolice®
New Ice Inc. / Instone	Verdepack™
Novamont	Mater-Bi®
PaperFoam b.v.	PaperFoam®
Plantic Technologies Ltd.	Plantic®, ecoPlastic™
Potatopak	Potatopak
Renewable Products Inc. / EarthShell	EarthShell®
Rodenburg Bioploymers b.v.	Solanyl®
Stanelco plc. / Biome Bioplastics Ltd.	Biome
Vegeplast©	Vegemat®
Wentus Kunstoff GmbH	Wenterra®

Table 2. Manufacturers of commercially available cellulose-based polymers

Company	**Trade Name**
Amcor Flexibles / Alcan Packaging	NaturePlus
Clarifoil	Clarifoil
Earthcycle Packaging Ltd.	Earthcycle
Eastman Chemical Company	Tenite™
FkuR Kunststoff	Biograde®
Grenidea	Agroresin™
Innovia Films	NatureFlex™
Mazzuccheli	Bioceta
New Ice Inc. / Instone	Verdepack™
Roots Biopack	
ValueForm Ltd.	TruGreen™
Vertupak Ltd. / ProcuraSell	Vegasse®

Table 3. Manufacturers of commercially available PLA

Company	**Trade Name**
Amcor Flexibles / Alcan Packaging	Ceramis®-PLA
BASF	Ecovio®
Cereplast	Cereplast Compostables™
Coopbox Italia	Naturalbox
Fabri-Kal	GreenWare®
FkuR Kunstoff GmbH	Bio-Flex®
Futerro (Galactic + Total Petrochemicals)	Loopla®
Hisun Biomaterials Co. Ltd.	
Huhtamaki UK Ltd.	BioWare®
International Paper	Ecotainer®
Natureworks LLC (Cargill / Teijin)	Natureworks PLA Ingeo™
Purac	
SidaPlax / Plastic Suppliers Inc.	EarthFirst®
Synbra Technology b.v.	BioFoam®
Treofan	Biophan

Table 4. Manufacturers of commercially available biobased monomers and polymers derived from these monomers

Company	**Trade Name**
Antron	Bio_Legacy nylon
Arkema	Rilsan®, Platamid®
BASF	Ultramid® Balance
Braskem	Bio-PE
Coca-Cola Company	PlantBottle™
Dow Chemical Company / Crystalserve	
DuPont	Biomax® PTT, Sorona®, Cerenol®
Shell Chemicals	Corterra™
Solvay	

Table 5. Manufacturers of commercially available PHA polymers

Company	**Trade Name**
Biomatera	Biomatera
Biomer	Biomer®
DSM / Tianjin Green BioSciences Ltd. Co.	Green Bio
Kaneka Corporation.	Kaneka
Meredian Inc.	
Mitsubishi Gas Chemicals	Biogreen®
PHB Industrial	Biocycle®
Telles (Metabolix Inc. / Archer Daniels Midland Co.)	Mirel™
Tepha	TephaFlex®, TephElast™
Tianan Biologic Material Co. Ltd.	Enmat™

Table 6. Comparison of industrial and home composting test conditions

Criteria	**Industrially Compostable** (EN 13432)	**Home Compostable** (EN 13432 with variations)
Biodegradation	Tested at 58 ℃ +/- 2℃, breakdown into CO_2 of ≥ 90% cf. control within 6 months.	Tested at 20-30℃, breakdown into CO_2 of ≥ 90% cf. control within 12 months.
Disintegration	Test performed at temperatures achieved in vessels of at least 140 L capacity. At 12 weeks (max.) ≤ 10% of original dry weight of test material > 2 mm remaining.	Test performed at 20-30℃ in vessels of at least 14 0 L capacity. At 6 months (max.) ≤ 10% of original dry weight of test material > 2 mm remaining.
Logo(s)		

Table 7. Manufacturers, trade names and material types of biobased food service ware

Company	Trade Name	Notes
Biopac	BioWrap, BioCell, BioForm	Cups - Cornstarch-lined board. Cold cups - PLA. Plates - sugarcane waste, palm leaves, bulrush. Food containers - cornstarch, cane, board. Bags - potato starch.
Celebration	Enviroware™, Ecotainer™, Naturegreen™	PLA. Fibreboard manufactured from sugar cane / reed / bamboo fibre. Cutlery - cellulose and limestone, or PLA.
Compostable packaging	Compostable Packaging, Greenware™	Injection moulding of compostable food service ware. Supply Greenware PLA cups.
Earthshell		Starch-based dinnerware.
Eco-Products		Plates and bowls - bagasse. Supply PLA cold cups and deli containers and PLA-lined hot cups.
Huhtamaki UK Ltd.	Bioware	PLA-coated paper board, biopolymers and moulded fiber products.
New Ice Inc. / Instone	Verdepack™	Starch-based fast food trays.
NVYRO Ltd.	NVYRO	Starch-based single use cups, plates, bowls, trays and cutlery.
Renewable Products Inc	EarthShell®	Cups and plates - starch and limestone.
Roots Biopack		Cups, plates and Trays - bagasse.
Vegware		Cups, plates, trays, bags, lids, cutlery – bagasse, PLA, cassava, corn starch.

Table 8. Sample details

LIMS code	Sample description	Food contact use
S09-012077	Corn starch tray	Chocolate mould
S09-012078	Corn starch tray	Chocolate mould
S09-012079	Starch containing co-extruded film	Packaging dry foodstuffs
S09-012080	Cellulose containing film	Packaging dry foodstuffs
S09-012081	Cellulose containing film	Packaging dry foodstuffs
S09-012082	Cellulose containing film	Packaging dry foodstuffs
S09-012083	Poly(lactic acid) cups	Serving beverages/soup
S09-012092	Cassava cups	Serving beverages/soup
S09-012093	Bagasse bowls	Serving liquid foodstuffs
S09-012094	Poly(lactic acid) cups	Serving beverages/soup
S09-012095	Bio hot cup lids	Serving beverages/soup
S09-012096	Hot-cups starch lined	Serving beverages/soup
S09-012097	Poly(lactic acid) bags	Sandwich/food bags

Table 9. Effect of storage at elevated temperature and high humidity on the physical appearance of the samples

LIMS code	Sample description	Effect of water and heat
S09-012077	Corn starch tray	Polymer dissolved
S09-012078	Corn starch tray	Polymer dissolved
S09-012079	Starch containing co-extruded film	No change (by eye)
S09-012080	Cellulose containing film	No change (by eye)
S09-012081	Cellulose containing film	No change (by eye)
S09-012082	Cellulose containing film	Loss of silver colour
S09-012083	Poly(lactic acid) cups	No change (by eye)
S09-012092	Cassava cups	Cups absorbed water
S09-012093	Bagasse bowls	Bowls absorbed water
S09-012094	Poly(lactic acid) cups	Polymer became opaque
S09-012095	Bio hot cup lids	No change (by eye)
S09-012096	Hot-cups starch lined	No change (by eye)
S09-012097	Poly(lactic acid) bags	No change (by eye)

Table 10. Estimated concentrations and best library matches for the substances detected in the corn starch tray (S09-012077) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
10.8	6.2	Pentanal
13.1	38.0	Hexanal
16.9	1.4	2-Pentyl furan

Table 11. Estimated concentrations and best library matches for the substances detected in the corn starch tray (S09-012078) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
10.8	3.6	Pentanal
13.1	28.1	Hexanal

Table 12. Estimated concentrations and best library matches for the substances detected in the starch containing co-extruded film (S09-012079) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
13.1	1.4	Hexanal

Table 13. Estimated concentrations and best library matches for the substances detected in the cellulose containing film (S09-012080) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
7.5	7.2	Carbon disulphide
12.6	11.0	Toluene

Table 14. Estimated concentrations and best library matches for the substances detected in the cellulose containing film (S09-012081) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
7.5	7.3	Carbon disulphide
8.9	1.8	Trichloromethane

Table 15. Estimated concentrations and best library matches for the substances detected in the cellulose containing film (S09-012082) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
7.5	11.1	No good library match
8.9	1.4	No good library match
9.3	20.0	Tetrahydrofuran
9.9	1.4	Acetic acid, 1-methylethyl ester
12.6	312.1	Toluene
13.3	8.7	Acetic acid, butyl ester
14.9	1.2	Xylene

Table 16. Estimated concentrations and best library matches for the substances detected in the poly(lactic acid) cups (S09-012083) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
12.6	10.4	Toluene

Table 17. Estimated concentrations and best library matches for the substances detected in the cassava cups (S09-012092) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
8.9	6.4	Ethyl acetate
13.1	1.4	Hexanal

Table 18. Estimated concentrations and best library matches for the substances detected in the bagasse bowls (S09-012093) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
8.9	8.3	Unspecified perfluorinated compound (characteristic ions m/z 69 and 131 observed)

Table 19. Estimated concentrations and best library matches for the substances detected in the poly(lactic acid) cups (S09-012094) by headspace GC-MS

Retention time (minutes)	Average concentration (µg/kg)	Best library match
No peaks detected with an estimated concentration of > 1 mg/kg		

Table 20. Estimated concentrations and best library matches for the substances detected in the bio hot cup lids (S09-012095) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
10.8	1.8	Pentanal
13.1	5.3	Hexanal

Table 21. Estimated concentrations and best library matches for the substances detected in the hot-cups starch lined (S09-012096) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
No peaks detected with an estimated concentration of > 1 mg/kg		

Table 22. Estimated concentrations and best library matches for the substances detected in the poly(lactic acid) bags (S09-012097) by headspace GC-MS

Retention time (minutes)	Average concentration (mg/kg)	Best library match
11.1	1.0	n-Propyl acetate

Table 23. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the corn starch tray (S09-012077) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1			16.4	Pentanoic acid
2			25.6	Hexanoic acid
3			9.8	1-Tridecene
4			16.6	Unspecified alkane
5			6.4	N,N-Dimethyl-1-tetradecanamine
6			6.6	Heptadecanal
7			96.2	N,N-Dimethyl-1-hexadecanamine
8			16.6	Pentadecanoic acid, 14-methyl-, methyl ester
9			24.1	n-Hexadecanoic acid
10			8.9	Octadecanal
11			6.4	9,12-Octadecadienoic acid (Z,Z)-, methyl ester
12			6.9	9-Octadecenoic acid (Z)-, methyl ester
13	3.4		147.4	N,N-Dimethyl-1-octadecanamine
14			10.5	Octadecanoic acid, methyl ester
15			84.1	No good library match
16			123.1	Propylene glycol monooleate
17			26.1	No good library match
18			80.5	No good library match
19			117.8	No good library match
20			46.7	No good library match

Table 24. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the corn starch tray (S09-012078) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1			8.3	Unspecified alkane
2			9.5	No good library match
3			4.4	Heptadecanal
4			87.9	N,N-Dimethyl-1-hexadecanamine
5			40.4	Pentadecanoic acid, 14-methyl-, methyl ester
6			89.7	n-Hexadecanoic acid
7			3.6	N,N-Dimethyl-1-heptadecanamine
8			7.1	Octadecanal
9			13.8	9,12-Octadecadienoic acid (Z,Z)-, methyl ester
10			12.7	9-Octadecenoic acid (Z)-, methyl ester
11			133.6	N,N-Dimethyl-1-octadecanamine
12			22.5	Octadecanoic acid, methyl ester
13			174.5	No good library match
14			104.8	9-Octadecenoic acid (z)-, 2-hydroxy-1-(hydroxymethyl)ethyl ester
15			27.1	No good library match
16			84.2	No good library match
17			116.9	No good library match
18			46.7	No good library match

Table 25. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the starch containing co-extruded film (S09-012079) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	1401.0	4581.0	1653.7	Glycerol
2		15.2	8.3	No good library match
3		78.4	38.0	Benzoic acid
4		5.7	4.5	Nonanoic acid
5	5.1	9.5	3.9	Triacetin
6	117.6	207.5	204.7	1,6-Dioxacyclododecane-7,12-dione
7	6.9	11.3	9.6	Diisopropyl naphthalene
8	10.5	21.3	16.7	Diisobutyl phthalate
9	1072.7	1865.9	2004.8	No good library match
10		6.9	9.5	No good library match
11		14.8	13.8	No good library match
12		225.6	176.9	1-(Phenylmethoxy) naphthalene
13	12.1	14.2	10.7	No good library match
14	4.1	3.3	9.0	2-Naphthyl benzoate
15	14.4	9.7	16.8	(Z)-9-Octadecenamide
16	28.9	35.2	40.7	No good library match
17	15.5	17.1	19.1	Di-(2-ethylhexyl) phthalate
18	21.3	11.9	19.4	Cis-11-Eicosenamide
19	6.4			No good library match (similar ions to peak 20)
20	4.8			No good library match (similar ions to peak 19)
21	25.9	38.3	46.1	Erucic acid
22	2792.4	1665.1	2090.5	(Z)-13-Docosenamide
23	21.7			No good library match (similar ions to peak 26)

Table 25 continued. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the starch containing co-extruded film (S09-012079) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
24	10.8			Squalene
25	783.8	652.6	755.0	No good library match
26	28.1			No good library match (similar ions to peak 23)
27	507.2	264.1	217.3	No good library match
28	347.7	121.3	106.5	No good library match
29	419.0	132.1	101.6	No good library match
30	621.0	285.9	239.2	No good library match

Table 26. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the cellulose containing film (S09-012080) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	371.2		6607.8	Glycerol
2	302.4		762.6	Acetic acid
3	77.9		668.8 [#]	No good library match
4	221.2		1369.0	Glycerol related compound
5	297.2		2457.9	Glycerol related compound
6	624.3		3874.7	Glycerol related compound
7	249.3		2223.2	Glycerol related compound

[#] Includes background interference

Table 27. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the cellulose containing film (S09-012081) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	253.5			3-Methoxy-1,1-propanediol (glycol related compound)
2	22.9			Glycol related compound
3	37615.7	41520.9	46953.0	Glycerol
4	4.1			No good library match
5		3.0	3.8	Dehydroabietene
6		4.3	3.7	Unspecified alkane
7	10.3			Unspecified alkane
8	22.0	35.9	38.0	Unspecified alkane
9	6.6	2.8	4.9	Unspecified alkane
10	39.7	49.5	41.9	Unspecified alkane
11	8.2	4.1	7.6	No good library match
12	27.9	25.3	38.5	No good library match
13	48.7	69.4	59.1	Unspecified alkane
14	54.8	70.7	48.0	Unspecified alkane
15	57.4	71.4	36.4	Unspecified alkane
16	49.9	63.1	22.4	Unspecified alkane
17	50.8	61.9	12.0	Unspecified alkane
18	46.0	12.4	38.9	No good library match
19	44.3	51.5	7.1	Unspecified alkane
20	51.9	52.0	6.8	Unspecified alkane
21	54.0	56.4	5.1	Unspecified alkane
22	66.0	45.9	15.0	No good library match and unspecified alkane
23	32.2	20.5	21.8	Unspecified alkane

Table 28. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the cellulose containing film (S09-012082) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	895.5		39766.8	Glycerol

Table 29. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the poly(lactic acid) cups (S09-012083) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1		54.5	44.9	1,6-Dioxacyclododecane-7,12-dione
2	2.1			No good library match
3	2.0			No good library match
4	4.1	2.8	3.1	No good library match
5	1.0			No good library match
6		3.7	4.9	1-Propene-1,2,3-tricarboxylic acid, tributyl ester
7	128.0	232.8	176.2	Acetyl tributyl citrate
8	30.5	78.0	67.2	No good library match (2 peaks co-eluting – same mass spectra)
9	51.8	65.3	50.6	Same mass spectrum as peak 8
10	12.1	4.9	7.5	Same mass spectrum as peak 8
11	5.2	11.2	11.8	Unspecified alkane
12	4.7	5.1	6.5	Squalene
13	133.9	134.9	123.3	No good library match
14	64.5	50.3	45.3	No good library match
15	18.6			No good library match

Table 30. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the cassava cups (S09-012092) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1			42.0	Benzoic acid
2			33.6	2,3-Dihydrobenzofuran
3			27.4	4-Hydroxybenzaldehyde and vanillin
4			15.5	4-Hydroxy-3,5-dimethoxybenzaldehyde
5	2.5	6.8	9.4	Unspecified alkane
6			43.8	4-Hydroxy-2-methoxycinnamaldehyde
7			179.3	n-Hexadecanoic acid
8	1.5		2.4	Unspecified alkane
9		9.3	9.0	Unspecified alkane
10		1.7	4.3	No good library match
11	28.8	37.5	31.2	Unspecified alkane
12	119.3	145.4	85.7	Unspecified alkane
13	260.4	291.7	113.3	Unspecified alkane
14	493.8	527.0	127.6	Unspecified alkane
15	11.9	14.5	6.6	Unspecified alkane
16	4.2	4.9	4.7	Unspecified alkane
17	623.3	605.1	83.1	Unspecified alkane
18	17.1	19.7	4.1	Unspecified alkane
19	13.3	18.8	9.9	Unspecified alkane
20	700.2	651.7	53.9	Unspecified alkane
21	27.9	24.3	4.3	Unspecified alkane
22	14.3	18.8	5.2	Unspecified alkane
23	541.5	482.1	25.8	Unspecified alkane
24	27.4	29.2		Unspecified alkane

Table 30 continued. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the cassava cups (S09-012092) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
25		19.4		Unspecified alkane
26	443.4	405.7		Unspecified alkane
27	281.9	245.3		Unspecified alkane
28	15.0			Unspecified alkane
29	180.1	145.7		Unspecified alkane
30	87.1	80.3		Unspecified alkane
31	30.2	27.8		Unspecified alkane

Table 31. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the bagasse bowls (S09-012093) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	2.4	15.0	62.3	No good library match
2	2.8	9.9	33.4	No good library match
3	1.5	2.7	181.8	No good library match
4	1.9	2.4	147.9	No good library match

Table 32. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the poly(lactic acid) cups (S09-012094) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	3.3			D,L-Lactide

Table 33. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the bio hot cup lids (S09-012095) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1		3.5	9.0	Hexanoic acid
2	27.7	3.7	7.6	D,L-Lactide
3	71.1			Glycol related compound
4		1.3	6.1	No good library match
5		3.9	16.9	No good library match
6	20.7	16.2	118.6	Isopropyl palmitate
7	13.2	9.2	74.3	Isopropyl stearate
8	2.4			No good library match

Table 34. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the hoy-cups starch lined (S09-012096) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	2.8			D,L-Lactide
2	9.9			Glycol related compound
3	4.6			(Z)-13-Docosenamide

Table 35. Estimated concentrations and best library matches for the substances detected in the ethanol extracts of the poly(lactic acid) bags (S09-012097) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	45.3	4.2	68.5	D,L-Lactide
2	80.4	27.6	18.0	Glycol related compound
3	393.3	23.5	34.6	Ethyl octadecanoate
4		3.3	5.6	No good library match
5	5.7			Ethyl eicosanoate
6		16.1	26.4	No good library match
7	101.6			No good library match
8	94.8			No good library match
9		22.3	35.1	No good library match
10		26.0	42.9	No good library match
11		76.5	100.3	No good library match
12	272.9			No good library match
13	192.7			No good library match
14	46.4	20.3	52.0	(Z)-13-Docosenamide
15	8.9			No good library match

Table 36. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the corn starch tray (S09-012078) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1			74.1	Di-(2-ethylhexyl) phthalate

Table 37. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the starch containing co-extruded film (S09-012079) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	4.7	7.0	3.9	Triacetin
2	165.0	158.9	124.1	1,6-Dioxacyclododecane-7,12-dione
3	3.4	7.5	6.9	Diethyl phthalate
4	1.2			Diisopropylnaphthalene isomer
5	1.0			Diisopropylnaphthalene isomer
6	1.5			Diisopropylnaphthalene isomer
7	5.8	12.4	9.8	Diisopropylnaphthalene isomer
8	7.8	17.2	13.6	Diisobutyl phthalate
9	1744.9	1261.1	1043.1	No good library match
10	1.8	4.8	5.0	Isooctyl dodecanoate
11	7.0	11.4	8.8	No good library match
12	4.9	6.8	5.6	p-Terphenyl
13	131.2	202.3	131.3	1-(Phenylmethoxy) naphthalene
14		11.3	11.5	9-Octadecenamide, (Z)-
15	22.1	59.7	46.5	No good library match
16	6.6	20.0	372.9	Di-(2-ethylhexyl) phthalate
17		10.6	9.5	Cis-11-Eicosenamide
18		3.1	2.1	No good library match
19		4.4	3.5	No good library match
20	12.0	54.3	34.0	Erucic acid
21	1248.9	1375.7	1107.9	(Z)-13-Docosenamide
22	6.7	21.7	42.4	No good library match
23	8.6			Squalene
24	746.9	513.4	403.1	No good library match

Table 37 continued. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the starch containing co-extruded film (S09-012079) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
25		21.5	9.0	No good library match (similar ions to peak 21)
26	227.7	231.6	175.3	No good library match
27	64.0	103.1	96.9	No good library match
28	134.7	204.9	141.6	No good library match
29	326.3	408.6	332.9	No good library match

Table 38. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the cellulose containing film (S09-012081) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	2.2	1.4	4.7	1,6-Dioxacyclododecane-7,12-dione
2	20.6	3.9	2.4	Dehydroabietene
3	1.8	3.7	2.0	Unspecified alkane
4	9.1	4.4	3.9	Unspecified alkane
6	24.2	37.4	28.2	Unspecified alkane
7	2.5	3.8	3.9	No good library match
8	2.6	7.2	4.8	No good library match
9	46.3	56.3	29.4	Unspecified alkane
10	5.4	6.6	6.0	No good library match
11	27.9	33.4	28.5	No good library match
12	1.1	1.6	1.7	Unspecified alkane
13	68.4	77.4	43.7	Unspecified alkane
14		2.5	2.7	Di-(2-ethylhexyl) phthalate
15	74.9	90.2	49.2	Unspecified alkane

Table 38 continued. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the cellulose containing film (S09-012081) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
16	74.2	91.7	52.0	Unspecified alkane
17	67.0	85.9	48.7	Unspecified alkane
18	64.3	94.0	52.8	Unspecified alkane
19	29.1	7.3	57.2	No good library match
20	56.3	75.9	49.2	Unspecified alkane
21	68.1	96.6	57.0	Unspecified alkane
22	75.3	109.2	65.1	Unspecified alkane
23	47.0	119.7	91.0	Unspecified alkane
24	9.2			No good library match
25	46.2	80.1	43.4	Unspecified alkane
26	22.6	52.3	27.3	Unspecified alkane
27	15.3	43.0	31.5	Unspecified alkane

Table 39. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the poly(lactic acid) cups (S09-012083) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	22.6	9.6	24.2	1,6-Dioxacyclododecane-7,12-dione
2	1.6	1.6	2.5	No good library match
3		5.5	6.6	1-Propene-1,2,3-tricarboxylic acid, tributyl ester
4		2.5	4.1	No good library match
5	95.2	169.4	183.3	Acetyl tributyl citrate
6		2.1	2.0	Phosphoric acid, diphenyl 2-ethylhexyl ester
7	26.9	57.5	83.9	No good library match (2 peaks co-eluting – same mass spectra)
8	41.2	55.1	69.9	Same mass spectrum as peak 7
9	1.9	1.6	11.2	Same mass spectrum as peak 7
10	4.4	9.6	13.5	Same mass spectrum as peak 7
11	1.3			Same mass spectrum as peak 7
12		2.3	2.7	Di-(2-ethylhexyl) phthalate
13	5.9	8.3	5.5	Unspecified alkane
14	8.0			Squalene
15	4.6			Unspecified alkane
16	133.6	143.5	152.0	No good library match
17	58.8	92.6	88.4	No good library match
18		30.2	30.0	No good library match
19	11.7	26.3	20.2	No good library match

Table 40. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the cassava cups (S09-012092) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	11.2		18.8	Unspecified alkane
2	76.6	1.9	85.2	Unspecified alkane
3	251.3	2.7	229.2	Unspecified alkane
5	690.9	7.6	548.4	Unspecified alkane
6	7.4		9.1	Unspecified alkane
7	1.9		1.9	Unspecified alkane
8	1156.4	13.2	878.9	Unspecified alkane
9	17.3		20.4	Unspecified alkane
10	8.5		9.7	Unspecified alkane
11	1644.8	15.4	1242.6	Unspecified alkane
12	38.5		16.2	Unspecified alkane
13	1548.8	13.9	1196.1	Unspecified alkane
14	34.3		32.1	Unspecified alkane
15	25.4		26.7	Unspecified alkane
16	1430.2	13.2	1147.2	Unspecified alkane
17	35.6		35.6	Unspecified alkane
18	20		20.4	Unspecified alkane
19	998.2	9.4	858.9	Unspecified alkane
20	17.9		25.6	Unspecified alkane
21	24.6		17.4	Unspecified alkane
22	689.7	6.3	602.5	Unspecified alkane
23	14.7		22.8	Unspecified alkane
24	14.3		18.6	Unspecified alkane
25	11.5		10.4	Unspecified alkane

Table 40 continued. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the cassava cups (S09-012092) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
26	340.1	5.2	331.6	Unspecified alkane
27	11.3		11.8	Unspecified alkane
28			5.5	Unspecified alkane
29			8.3	Unspecified alkane
30	133.1		133.9	Unspecified alkane
31	33.4		50.0	Unspecified alkane
32			11.0	Unspecified alkane

Table 41. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the bagasse bowls (S09-012093) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1			23.1	No good library match
2			25.9	No good library match
3			1.6	No good library match
4			2.3	No good library match
5			3.7	Unspecified alkane
6			1.1	Diisobutyl phthalate
7			1.6	Di-n-butyl phthalate
9			2.8	Ethyl oleate

Table 42. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the bio hot cup lids (S09-012095) by GC-MS

Peak number	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	1.4			Dehydroabietene
2	1.7			Glycol related compound
3	2.2	7.9	27.8	Isopropyl palmitate
4	1.6	6.2	19.8	Isopropyl stearate

Table 43. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the hot-cups starch lined (S09-012096) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1	22.0	74.5	50.5	16-Hentriacontanone
2	50.7	109.8	76.3	No good library match
3	18.6	51.0	33.0	18-Pentatriacontanone

Table 44. Estimated concentrations and best library matches for the substances detected in the isooctane extracts of the poly(lactic acid) bags (S09-012097) by GC-MS

Peak number *	Concentration (mg/kg)			Best library match
	Sample as received	Sample + heat	Sample + heat + water	
1		2.7	5.7	No good library match
2		18.4	52.9	No good library match
3		4.2	5.2	No good library match
4		32.0	31.3	No good library match
5			54.0	(Z)-13-Docosenamide

Table 45. Compounds detected in the ethanol and isooctane extracts of the corn starch tray (S09-012077) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass*	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
1.3	590.3514	C26H54O14	Part of oligomer series	No	Yes	No	No	No	No
1.5-2.8	695.4303	C30H62O16	Part of oligomer series	No	Yes	No	No	No	No
3.0	766.4562	C34H70O18	Part of oligomer series	No	Yes	No	No	No	No
3.3	810.4824	C36H74O19	Part of oligomer series	No	Yes	No	No	No	No
3.6	854.5086	C38H78O20	Part of oligomer series	No	Yes	No	No	No	No
3.8	898.5349	C40H82O21	Part of oligomer series	No	Yes	No	No	No	No
4.1	942.5611	C42H86O22	Part of oligomer series	No	Yes	No	No	No	No
4.3	986.5873	C44H90O23	Part of oligomer series	No	Yes	No	No	No	No
4.4	1030.6135	C46H94O24	Part of oligomer series	No	Yes	No	No	No	No

* Only highest mass of series given

Table 46. Compounds detected in the ethanol and isooctane extracts of the corn starch tray (S09-012077) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
29.6	280.2431	C18H32O2	Linoleic acid	No	Yes	No	No	Yes	No
31.0	256.2426	C16H32O2	Palmitic acid	No	Yes	Yes	Yes	Yes	No
31.3	282.2587	C18H34O2	Oleic acid	No	Yes	Yes	Yes	Yes	No

Table 47. Compounds detected in the ethanol and isooctane extracts of the corn starch tray (S09-012078) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass*	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
1.3	634.3776	C28H58O15	Part of oligomer series	No	Yes	No	No	Yes	No
1.6-2.8	722.4300	C32H66O17	Part of oligomer series	No	Yes	No	No	Yes	No
3.1	766.4562	C34H70O18	Part of oligomer series	No	Yes	No	No	Yes	No
3.3	810.4824	C36H74O19	Part of oligomer series	No	Yes	No	No	Yes	No
3.6	854.5086	C38H78O20	Part of oligomer series	No	Yes	No	No	Yes	No
3.8	898.5349	C40H82O21	Part of oligomer series	No	Yes	No	No	Yes	No
4.1	942.5611	C42H86O22	Part of oligomer series	No	Yes	No	No	Yes	No
4.4	986.5873	C44H90O23	Part of oligomer series	No	Yes	No	No	Yes	No
4.6	1030.6135	C46H94O24	Part of oligomer series	No	Yes	No	No	Yes	No

* Only highest mass of series given

Table 48. Compounds detected in the ethanol and isooctane extracts of the corn starch tray (S09-012078) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
18.4	296.2382	C21H29N	Diisopromine #	No	Yes	No	No	Yes	No
29.4	280.2430	C18H32O2	Linoleic acid	No	Yes	Yes	No	Yes	Yes
31.0	256.2426	C16H32O2	Palmitic acid	No	Yes	No	Yes	Yes	No
31.2	282.2588	C18H34O2	Oleic acid	No	Yes	No	Yes	Yes	No

proposed from internet search of predicted formula

Table 49. Compounds detected in the ethanol and isooctane extracts of the starch containing co-extruded film (S09-012079) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
1.8-3.8	-	C29H60O15	Part of oligomer series	No	Yes	Yes	No	No	No
4.6	290.1729	C14H26O6		No	Yes	Yes	No	Yes	No
5.1	232.1331	multiple		No	Yes	Yes	No	Yes	Yes
7.4	346.1628	C16H26O8		No	Yes	Yes	Yes	No	No
7.9	348.2148	multiple		No	Yes	Yes	No	No	No
8.4	492.2571	multiple		No	Yes	Yes	No	No	No
9.3	274.1780	multiple		No	Yes	Yes	Yes	Yes	No
9.6	418.2203	multiple		No	Yes	Yes	Yes	Yes	No
11.2	360.1784	multiple		No	Yes	Yes	Yes	No	No
11.5	490.2778	multiple		No	Yes	Yes	No	No	No
12.0	346.2355	multiple		No	Yes	Yes	Yes	Yes	Yes
12.4	438.1876	multiple		No	Yes	Yes	Yes	No	No
13.3	458.2615	multiple		No	Yes	Yes	No	Yes	Yes
13.5	432.2346	multiple		No	Yes	Yes	Yes	No	Yes
14.2	510.2452	multiple		No	Yes	Yes	No	No	No
15.1	618.3238	multiple		No	Yes	Yes	Yes	No	No
16.1	400.2097	multiple		No	Yes	Yes	No	Yes	Yes
17.3	638.2925	multiple		No	Yes	Yes	No	No	No
17.7	638.2925	multiple		No	Yes	Yes	Yes	No	No
18.5	256.1675	C14H24O4		No	Yes	Yes	Yes	Yes	Yes
19.3	710.3500	multiple		No	Yes	Yes	No	No	No
20.9	420.1784	multiple		Yes	Yes	Yes	No	Yes	Yes
23.9	600.3132	multiple		No	Yes	Yes	Yes	Yes	Yes

Table 49 continued. Compounds detected in the ethanol and isooctane extracts of the starch containing co-extruded film (S09-012079) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
25.5	602.4017	C32H58O10	Octylphenol ethoxylate	No	Yes	Yes	No	No	No
26.1	620.2819	multiple		yes	Yes	Yes	Yes	Yes	Yes
26.8	800.4194	C40H64O16		No	Yes	Yes	No	Yes	Yes
27.9	476.2406	C26H36O8		No	Yes	Yes	Yes	Yes	Yes
28.9	584.3924	C32H56O9		No	Yes	Yes	No	No	Yes
29.7	512.3349	C28H48O8		No	Yes	Yes	No	Yes	Yes

Table 50. Compounds detected in the ethanol and isooctane extracts of the starch containing co-extruded film (S09-012079) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
1.1	345.1580	C19H24NO5		No	Yes	Yes	No	Yes	No
3.9	274.1807	C17H24NO2		No	Yes	Yes	No	Yes	No
4.7	418.2260	multiple		No	Yes	Yes	No	Yes	No
10.3	494.2584	multiple		No	Yes	Yes	Yes	Yes	No
14.8	494.2584	multiple		No	Yes	Yes	Yes	Yes	No
19.1	530.3528	multiple		No	Yes	Yes	Yes	No	No
26.9	750.4302	multiple		No	Yes	Yes	Yes	No	No
29.6	281.2464	C18H32O2	Linoleic acid	No	Yes	Yes	No	Yes	Yes
30.9	256.2427	C16H32O2	Palmitic acid	Yes	Yes	Yes	Yes	Yes	Yes
31.2	282.2588	C18H34O2	Oleic acid	Yes	Yes	Yes	Yes	Yes	Yes

Table 51. Compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012080) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass*	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
1.2	502.2985	C22H46O12	Part of oligomer series	No	Yes	Yes	No	Yes	Yes
1.2-6.0	722.4300	C32H66O17	Part of oligomer series	No	Yes	No	No	No	No
16.1	400.2097	multiple		No	Yes	Yes	Yes	Yes	Yes
21.0	420.1784	multiple		No	Yes	Yes	No	Yes	No
29.7	512.3352	multiple		No	Yes	Yes	No	Yes	Yes

* Only highest mass of series given

Table 52. Compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012080) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
1.0 - 3.2	Oligomer series			No	Yes	No	No	No	No
30.9	256.2426	C16H32O2	Palmitic acid	No	Yes	No	No	No	No
31.3	282.2588	C18H34O2	Oleic acid	No	Yes	No	No	No	No

Table 53. Compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012081) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
1.1	166.0841	C6H14O5		No	Yes	Yes	No	Yes	No
4.6	290.1732	C14H26O6		No	Yes	Yes	No	No	No
8.3	492.2571	multiple		No	Yes	Yes	No	Yes	No
9.6	418.2203	multiple		No	Yes	Yes	No	Yes	No
10.9	476.2621	multiple		No	Yes	Yes	No	Yes	No
11.6	490.2778	multiple		Yes	Yes	Yes	No	Yes	No
12.4	438.1890	multiple		No	Yes	No	No	No	No
14.2	510.2459	multiple		Yes	Yes	Yes	No	Yes	No
16.1	400.2097	multiple		Yes	Yes	Yes	No	Yes	Yes
16.7	690.3827	multiple		No	Yes	Yes	No	Yes	No
19.3	710.3503	multiple		No	Yes	Yes	No		No
21.0	420.1787	multiple		No	Yes	Yes	No	Yes	Yes
23.9	600.3146	multiple		No	Yes	Yes	No	Yes	Yes
25.4	771.5200	No sensible matches	Part of oligomer series*	No	Yes	Yes	No	Yes	Yes
26.1	620.2822	multiple		No	Yes	Yes	No	Yes	Yes
28.0	640.2509	multiple		No	Yes	Yes	Yes	Yes	Yes
29.1	840.3568	multiple		No	Yes	Yes	No	Yes	Yes

* Only highest mass of series given

Table 54. Compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012081) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
1.1	345.1582	C19H24NO5		No	Yes	Yes	No	Yes	No
25.7	300.2121	C20H28O2	Icosa-5,8,11-triynoic acid	Yes	Yes	Yes	Yes	Yes	Yes
30.8	256.2427	C16H32O2	Palmitic acid	No	Yes	Yes	Yes	Yes	Yes
31.2	282.2588	C18H34O2	Oleic acid	No	Yes	Yes	No	Yes	Yes

Table 55. Compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012082) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass*	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
24.1	790.5095	C41H47O14	Part of oligomer series	No	Yes	No	No	No	No
25.4	770.5494	C50H47O6	Part of oligomer series	No	Yes	No	No	No	No
28.3	740.5383	C49H72O5	Part of oligomer series	No	Yes	No	No	No	No

* Only highest mass of series given

Table 56. Compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012082) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
1.1	192.0820	multiple		No	Yes	Yes	Yes	No	No
30.6	256.2427	C16H32O2	Palmitic acid	No	Yes	Yes	No	No	No

Table 57. Compounds detected in the ethanol and isooctane extracts of the poly(lactic acid) cups (S09-012083) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
5.1	258.1580	C12H22N2O4		No	Yes	Yes	No	No	Yes
13.2	458.2628	multiple		No	Yes	Yes	No	Yes	Yes
16.1	400.2100	multiple		No	Yes	Yes	Yes	Yes	Yes
19.0	314.1157	multiple		No	Yes	Yes	No	Yes	Yes
20.9	420.1787	multiple		No	Yes	Yes	No	Yes	Yes
23.7	600.3132	multiple		Yes	Yes	Yes	No	Yes	Yes
24.1	342.1470	multiple		No	Yes	Yes	Yes	Yes	Yes
25.1	342.1470	multiple		No	Yes	Yes	Yes	Yes	Yes
25.3	342.1460	multiple		No	Yes	Yes	No	Yes	Yes
26.1	620.2822	multiple		No	Yes	Yes	Yes	Yes	Yes
28.1	402.2257	C20H34O8	Acetyl tributyl citrate	No	Yes	Yes	Yes	Yes	Yes
29.2	840.3563	multiple		Yes	Yes	Yes	Yes	Yes	Yes
29.5	660.2193	multiple		No	No	No	No	No	Yes

Table 58. Compounds detected in the ethanol and isooctane extracts of the poly(lactic acid) cups (S09-012083) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
22.6	400.2303	multiple		No	Yes	Yes	No	Yes	No
25.8	300.2122	C20H28O2	Icosa-5,8,11-triynoic acid	Yes	Yes	Yes	Yes	Yes	Yes
28.8	302.2278	C20H30O2	Eicosapentanoic acid	No	Yes	Yes	Yes	Yes	Yes
29.5	280.2430	C18H32O2	Linoleic acid	No	Yes	Yes	Yes	Yes	No
30.8	256.2427	C16H32O2	Palmitic acid	Yes	Yes	Yes	Yes	Yes	Yes
31.2	282.2587	C18H34O2	Oleic acid	No	Yes	Yes	No	Yes	Yes

Table 59. Compounds detected in the ethanol and isooctane extracts of the cassava cups (S09-012092) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
28.1	318.2770	Multiple		Yes	Yes	Yes	N/A	Yes	Yes
29.5	332.2927	Multiple		No	Yes	Yes	N/A	Yes	Yes

N/A – not analysed

Table 60. Compounds detected in the ethanol and isooctane extracts of the cassava cups (S09-012092) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
2.8	122.0368	C7H6O2	Benzoic acid	No	Yes	Yes	N/A	No	No
30.9	256.2426	C16H32O2	Palmitic acid	Yes	Yes	Yes	N/A	Yes	Yes
31.2	282.2587	C18H34O2	Oleic acid	No	Yes	Yes	N/A	Yes	Yes

N/A not analysed

Table 61. Compounds detected in the ethanol and isooctane extracts of the bagasse bowls (S09-012093) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
30.9	256.2427	C16H32O2	Palmitic acid	No	Yes	Yes	Yes	Yes	Yes
31.2	282.2588	C18H34O2	Oleic acid	No	Yes	Yes	No	Yes	Yes

Table 62. Compounds detected in the ethanol and isooctane extracts of the bio hot cup lids (S09-012095) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
16.3	432.1268	C18H24O12		No	Yes	No	No	No	No
25.4	720.2100	Multiple		No	Yes	No	No	No	No
26.1	792.2319	Multiple		No	Yes	No	No	No	No
26.9	864.2530	Multiple		No	Yes	No	No	No	No
28.9	484.3026	Multiple		No	Yes	No	No	No	No

Table 63. Compounds detected in the ethanol and isooctane extracts of the bio hot cup lids (S09-012095) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
30.9	256.2427	C16H32O2	Palmitic acid	No	Yes	No	Yes	No	Yes

Table 64. Compounds detected in the ethanol and isooctane extracts of the hot-cups starch lined (S09-012096) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
28.4	353.3294	C22H43NO2	13-hydroxy-cis-14-docosenamide	No	Yes	Yes	Yes	No	No
29.1	351.3137	Multiple		No	Yes	Yes	No	No	No

Table 65. Compounds detected in the ethanol and isooctane extracts of the hot-cups starch lined (S09-012096) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
30.8	256.2426	C16H32O2	Palmitic acid	No	No	Yes	Yes	No	Yes
31.2	281.2486	C18H34O2	Oleic acid	No	No	Yes	No	No	Yes

Table 66. Compounds detected in the ethanol and isooctane extracts of the poly(lactic acid) bags (S09-012097) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
3.1	306.0951	C12H18O9		No	Yes	No	No	No	No
5.4	378.1162	C15H22O11		No	Yes	No	No	No	No
8.2	450.1373	C18H26O13		No	Yes	No	No	No	No
10.6	522.1571	C21H30O15		No	Yes	No	Yes	No	No
12.8	594.1796	C24H34O17		No	Yes	No	No	No	No
14.4	666.2007	C27H38O19		No	Yes	No	No	No	No
15.5	432.1268	C18H24O12		No	Yes	No	Yes	No	No
16.3	738.2213	C30H42O21		No	Yes	No	No	No	No
17.6	810.2430	C33H46O23		No	Yes	No	No	No	No
18.2	504.1479	C21H28O14		No	Yes	No	No	No	No
29.2	351.3137	Multiple		No	Yes	No	No	No	No

Table 67. Compounds detected in the ethanol and isooctane extracts of the poly(lactic acid) bags (S09-012097) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Proposed identity	Present in ethanol			Present in isooctane		
				Not aged	Aged with heat	Aged with water and heat	Not aged	Aged with heat	Aged with water and heat
29.7	376.2872	C20H40O6		No	Yes	Yes	Yes	Yes	Yes
30.1	376.2870	C20H40O6		No	Yes	Yes	Yes	Yes	Yes
30.5	448.3095	C23H46NO5S		No	Yes	Yes	No	Yes	Yes
30.9	256.2425	C16H32O2	Palmitic acid	No	Yes	Yes	Yes	Yes	Yes
31.2	390.3030	C19H42N4O2S		No	Yes	Yes	No	Yes	Yes
31.7	404.3190	C18H42N7OS		Yes	Yes	Yes	Yes	Yes	Yes

Table 68. Peak areas of the compounds detected in the ethanol and isooctane extracts of the aged corn starch tray (S09-012077) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
29.6	280.2431	C18H32O2	1.2E+07	6.4E+04	1.2E+06	1.0E+05
31.0	256.2426	C16H32O2	1.9E+06	1.7E+06	3.0E+06	1.6E+05
31.3	282.2587	C18H34O2	5.1E+06	7.7E+05	2.3E+06	3.6E+05

Table 69. Peak areas of the compounds detected in the ethanol and isooctane extracts of the aged corn starch tray (S09-012078) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
18.4	296.2382	C21H29N	3.3E+06	ND	1.3E+04	ND
29.4	280.243	C18H32O2	4.4E+07	2.0E+05	2.3E+06	7.9E+04
31.0	256.2426	C16H32O2	5.2E+06	ND	2.6E+06	ND
31.2	282.2588	C18H34O2	2.0E+07	ND	3.5E+06	ND

ND – not detected

Table 70. Peak areas of the compounds detected in the ethanol and isooctane extracts of the aged starch containing co-extruded film (S09-012079) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
1.8-3.8	-	C29H60O15	6.02E+07	1.08E+08	ND	ND
4.6	290.1729	C14H26O6	2.23E+07	1.64E+06	7.85E+06	ND
5.1	232.1331	multiple	4.41E+07	4.55E+07	7.44E+06	9.85E+06
7.4	346.1628	C16H26O8	8.22E+06	6.16E+06	ND	ND
7.9	348.2148	multiple	7.53E+06	7.42E+06	ND	ND
8.4	492.2571	multiple	7.70E+06	1.37E+06	ND	ND
9.3	274.1780	multiple	4.43E+07	3.39E+07	8.33E+06	ND
9.6	418.2203	multiple	5.27E+07	3.54E+07	8.98E+06	ND
11.2	360.1784	multiple	9.15E+06	3.65E+06	ND	ND
11.5	490.2778	multiple	4.10E+07	2.92E+07	ND	ND
12.0	346.2355	multiple	1.06E+08	1.26E+08	3.81E+07	2.77E+06
12.4	438.1876	multiple	5.92E+07	4.27E+07	ND	ND

Table 70 continued. Peak areas of the compounds detected in the ethanol and isooctane extracts of the aged starch containing co-extruded film (S09-012079) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
13.3	458.2615	multiple	4.78E+07	5.20E+07	9.18E+06	8.71E+06
13.5	432.2346	multiple	5.92E+07	3.25E+07	ND	8.71E+06
14.2	510.2452	multiple	4.38E+07	2.58E+07	ND	ND
15.1	618.3238	multiple	2.50E+07	1.41E+07	ND	ND
16.1	400.2097	multiple	6.41E+08	5.31E+08	6.64E+08	6.85E+08
17.3	638.2925	multiple	6.42E+06	4.03E+06	ND	ND
17.7	638.2925	multiple	4.92E+06	2.83E+06	ND	ND
18.5	256.1675	C14H24O4	1.83E+08	1.41E+08	1.86E+08	1.27E+08
19.3	710.3500	multiple	3.79E+07	2.83E+07	ND	ND
20.9	420.1784	multiple	7.05E+08	6.36E+08	4.51E+08	3.81E+08
23.8	600.3132	multiple	3.34E+08	3.11E+08	2.47E+08	2.58E+08
25.5	602.4017	multiple	2.67E+07	3.11E+08	ND	ND
26.1	620.2819	multiple	2.35E+08	2.16E+08	1.78E+08	1.51E+08
26.8	800.4194	C40H64O16	8.88E+07	8.60E+07	3.38E+07	2.91E+07
27.9	476.2406	C26H36O8	1.65E+08	3.20E+08	2.95E+08	2.47E+08
28.9	584.3924	C32H56O9	8.05E+07	5.50E+07	ND	1.03E+07
29.7	512.3349	C28H48O8	3.58E+08	3.24E+08	3.37E+08	3.29E+08

ND – not detected

Table 71. Peak areas of the compounds detected in the ethanol and isooctane extracts of the aged starch containing co-extruded film (S09-012079) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
1.1	345.1580	C19H24NO5	2.4E+06	3.1E+06	2.4E+05	ND
3.9	274.1807	C17H24NO2	4.3E+06	3.6E+06	9.3E+05	ND
4.7	418.2260	multiple	2.1E+06	1.7E+06	5.3E+05	ND
10.4	494.2584	multiple	1.2E+06	9.5E+05	1.2E+05	ND
14.8	494.2584	multiple	3.7E+06	3.2E+06	1.0E+05	ND
19.2	530.3528	multiple	3.7E+06	3.5E+06	ND	ND
27.0	750.4302	multiple	3.5E+06	3.5E+06	ND	ND
29.6	281.2464	C18H32O2	5.5E+07	5.2E+07	4.3E+07	5.4E+07
30.9	256.2427	C16H32O2	1.1E+07	6.1E+06	1.2E+07	1.2E+07
31.2	282.2588	C18H34O2	1.0E+07	7.5E+06	1.5E+07	1.7E+07

ND – not detected

Table 72. Peak areas of the compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012080) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
1.2	502.2985	C22H46O12	4.73E+08	5.66E+08	2.38E+08	9.64E+06
1.2-6.0	722.4300	C32H66O17	8.55E+09	ND	ND	ND
16.1	400.2097	multiple	2.60E+07	6.84E+06	1.47E+07	2.40E+06
21.0	420.1784	multiple	8.97E+06	2.09E+06	3.85E+06	ND
29.7	512.3352	multiple	2.40E+07	7.96E+06	1.37E+07	2.26E+06

ND – Not detected

Table 73. Peak areas of the compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012080) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
1.0 - 3.2	range	multiple	4.3E+07	ND	ND	ND
30.9	256.2426	C16H32O2	2.6E+06	ND	ND	ND
31.3	282.2588	C18H34O2	1.4E+06	ND	ND	ND

ND – not detected

Table 74. Peak areas of the compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012081) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
1.1	166.0841	C6H14O5	6.42E+07	5.10E+07	1.84E+07	ND
4.6	290.1732	C14H26O6	1.37E+07	4.42E+06	ND	ND
8.3	492.2571	multiple	5.31E+06	5.96E+05	8.18E+05	ND
9.6	418.2203	multiple	7.13E+06	4.74E+05	2.10E+06	ND
10.9	476.2621	multiple	3.26E+06	1.07E+06	1.29E+06	ND
11.6	490.2778	multiple	9.18E+06	2.72E+06	1.57E+06	ND
12.4	438.1890	multiple	3.61E+06	ND	ND	ND
14.2	510.2459	multiple	1.28E+07	3.75E+06	5.38E+05	ND
16.1	400.2097	multiple	1.30E+08	6.74E+07	2.10E+08	2.42E+07
16.7	690.3827	multiple	4.37E+06	1.69E+06	5.84E+05	ND
19.3	710.3503	multiple	9.20E+06	5.09E+06	ND	ND
21.0	420.1787	multiple	7.36E+07	5.46E+07	9.07E+07	2.19E+07
23.9	600.3146	multiple	1.25E+08	2.87E+07	3.89E+07	7.79E+06

Table 74 continued. Peak areas of the compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012081) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
25.4	771.5200	No sensible matches	1.58E+08	5.62E+07	4.51E+07	1.88E+07
26.1	620.2822	multiple	3.09E+07	6.05E+06	2.25E+06	2.16E+06
28.0	640.2509	multiple	3.73E+07	5.08E+07	3.49E+07	1.64E+07
29.1	840.3568	multiple	3.92E+06	1.53E+07	4.64E+06	2.03E+06

Table 75. Peak areas of the compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012081) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
1.1	192.0813	multiple	1.8E+07	1.0E+07	2.6E+06	ND
25.7	300.2121	C17H32O2S	6.1E+06	1.0E+07	1.0E+07	2.4E+06
30.8	256.2427	C16H32O2	3.0E+05	2.0E+05	5.9E+05	1.3E+05
31.2	282.2588	C18H34O2	1.3E+05	3.4E+05	3.5E+05	1.0E+05

Table 76. Peak areas of the compounds detected in the ethanol and isooctane extracts of the cellulose containing film (S09-012082) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
1.1	192.0820	multiple	1.0E+07	2.6E+04	ND	ND
30.6	256.2427	C16H32O2	4.0E+05	1.5E+05	ND	ND

ND – not detected

Table 77. Peak areas of the compounds detected in the ethanol and isooctane extracts of the poly(lactic acid) cups (S09-012083) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
5.1	258.158	C12H22N2O4	6.59E+06	7.19E+06	ND	6.70E+05
13.2	458.2628	multiple	1.94E+07	1.93E+07	2.96E+06	3.90E+06
16.1	400.2100	multiple	4.58E+08	4.54E+08	3.97E+08	4.10E+08
19.0	314.1157	multiple	1.13E+08	1.27E+08	1.29E+08	3.48E+07
20.9	420.1787	multiple	3.03E+08	3.06E+08	2.47E+08	2.74E+08
23.7	600.3132	multiple	1.72E+08	1.77E+08	1.05E+08	1.30E+08
24.1	342.1470	multiple	1.28E+06	2.51E+07	1.74E+07	2.27E+07
25.1	342.1470	multiple	2.14E+08	2.31E+08	2.19E+08	2.12E+08
25.3	342.1460	multiple	2.31E+08	3.35E+08	2.74E+08	2.45E+08
26.1	620.2822	multiple	1.08E+08	1.02E+08	7.98E+07	7.79E+07
28.1	402.2257	C20H34O8	2.77E+08	3.00E+08	2.59E+08	2.73E+08
29.2	840.3563	multiple	2.88E+07	1.92E+07	7.57E+06	7.57E+06
29.5	660.2193	multiple	ND	ND	ND	9.97E+05

ND – not detected

Table 78. Peak areas of the compounds detected in the ethanol and isooctane extracts of the poly(lactic acid) cups (S09-012083) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
22.6	400.2303	multiple	6.4E+06	3.2E+06	3.8E+05	ND
25.8	300.2122	C20H28O2	6.0E+06	4.8E+06	3.7E+06	1.5E+06
28.8	302.2278	C20H30O2	8.8E+05	1.3E+06	7.8E+05	5.4E+05
29.6	280.243	C18H32O2	3.0E+05	2.4E+05	2.9E+05	ND
30.9	256.2427	C16H32O2	2.7E+06	2.3E+06	2.5E+06	1.1E+06
31.2	282.2587	C18H34O2	1.2E+06	1.4E+06	1.5E+06	9.2E+05

ND – not detected

Table 79. Peak areas of the compounds detected in the ethanol and isooctane extracts of the cassava cups (S09-012092) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
28.1	318.2770	Multiple	3.37E+07	4.20E+07	3.12E+06	3.17E+07
29.5	332.2927	Multiple	2.12E+07	2.34E+07	4.99E+06	1.49E+07

Table 80. Peak areas of the compounds detected in the ethanol and isooctane extracts of the cassava cups (S09-012092) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
2.8	122.0368	C7H6O2	1.1E+06	1.0E+05	ND	ND
30.9	256.2426	C16H32O2	2.6E+07	3.3E+07	4.8E+07	2.6E+07
31.2	282.2587	C18H34O2	6.2E+06	5.8E+06	7.3E+05	3.4E+06

ND – not detected

Table 81. Peak areas of the compounds detected in the ethanol and isooctane extracts of the bagasse bowls (S09-012093) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
30.9	256.2427	C16H32O2	5.4E+05	3.1E+06	5.5E+05	3.5E+04
31.2	282.2588	C18H34O2	4.6E+05	2.0E+06	4.6E+05	5.3E+05

Table 82. Peak areas of the compounds detected in the ethanol and isooctane extracts of the bio hot cup lids (S09-012095) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
30.8	256.2425	C16H32O2	3.0E+06	ND	ND	9.6E+06

ND – not detected

Table 83. Peak areas of the compounds detected in the ethanol and isooctane extracts of the hot-cups starch lined (S09-012096) by LC-TOF-MS analysis (positive mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
28.4	353.3294	Multiple	1.58E+06	3.05E+06	ND	ND
29.1	351.3137	Multiple	2.19E+06	4.64E+06	ND	ND

ND – not detected

Table 84. Peak areas of the compounds detected in the ethanol and isooctane extracts of the hot-cups starch lined (S09-012096) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
30.8	256.2426	C16H32O2	ND	1528723	ND	1424968
31.2	281.2486	C18H34O2	ND	902188	ND	744015

ND – not detected

Table 85. Peak areas of the compounds detected in the ethanol and isooctane extracts of the poly(lactic acid) bags (S09-012097) by LC-TOF-MS analysis (negative mode electrospray)

Retention time (minutes)	Mass	Predicted formula	Peak area in ethanol		Peak area in isooctane	
			Aged with heat	Aged with water and heat	Aged with heat	Aged with water and heat
29.7	376.2872	C18H39N4O2S	1.1E+07	4.6E+06	9.6E+06	2.5E+05
30.1	376.2870	C18H39N4O2S	4.0E+07	3.4E+07	2.8E+07	1.5E+07
30.5	448.3095	C23H46NO5S	2.1E+06	1.1E+06	1.3E+06	3.9E+05
30.9	256.2425	C16H32O2	2.2E+06	1.1E+06	2.4E+06	3.5E+05
31.2	390.3030	C19H42N4O2S	1.5E+06	1.0E+06	6.6E+05	5.1E+05
31.7	404.3190	C18H42N7OS	8.7E+06	4.5E+06	8.6E+06	1.6E+06

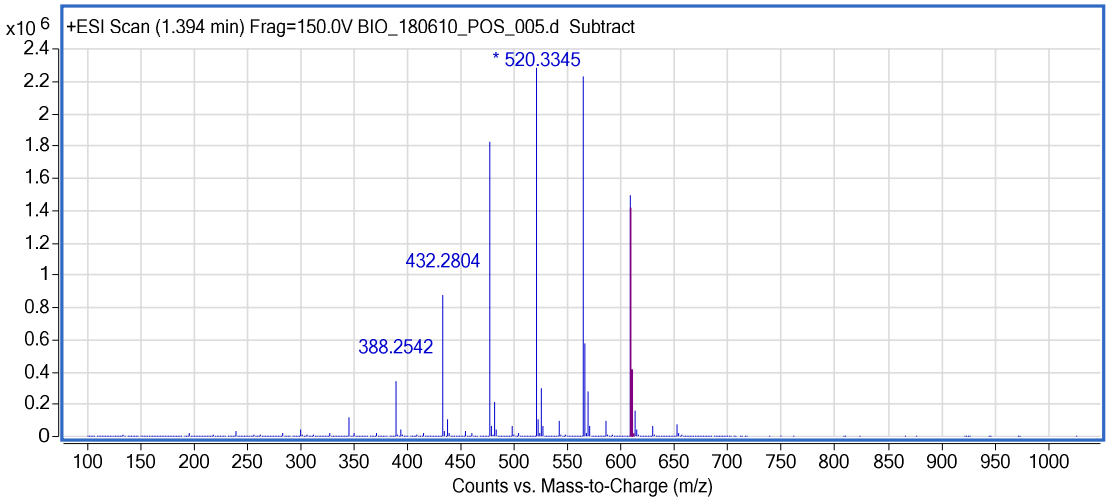
Table 86. Migration studies

LIMS code	Migrant(s)	Simulants / Foodstuff	Test conditions
S09-012077	N,N-Dimethyl-1-tetradecanamine [#] N,N-Dimethyl-1-hexadecanamine [#] N,N-Dimethyl-1-octadecanamine 9-Octadecenoic acid (Z)-, 2-hydroxy-1-(hydroxymethyl)ethyl ester [#]	Olive oil / Chocolate	10 days at 40℃
S09-012079	Glycerin 1-Phenylmethoxynaphthalene [#] Di-(2-ethylhexyl) phthalate (Z)-13-Docosenamide	Tenax / Cereal	10 days at 40℃
S09-012081	Glycerin	Tenax / Cereal	10 days at 40℃
S09-012092	2,3-Dihydrobenzofuran [#] Total alkanes	3% Acetic acid / 10% Ethanol / Apple juice	2 hours at 70℃
S09-012093	Unspecified perfluorinated compounds [#]	3% Acetic acid / Olive oil / Soup	2 hours at 70℃
S09-012097	(Z)-13-Docosenamide	Tenax / Bread	24 hours at 40℃

Analytical standards were not obtained

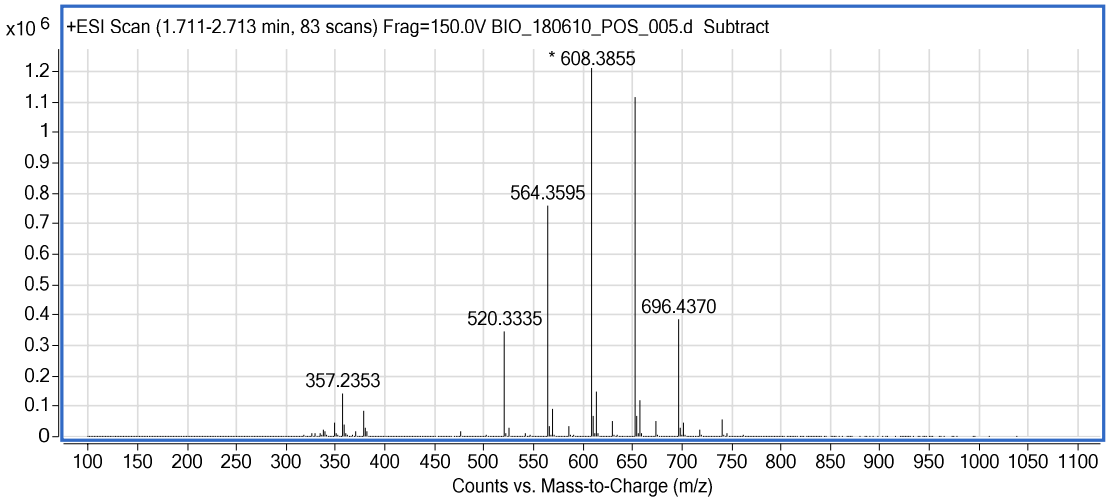
ANNEX 1

S09-012077 positive mode ESI, peak at 1.3 minutes



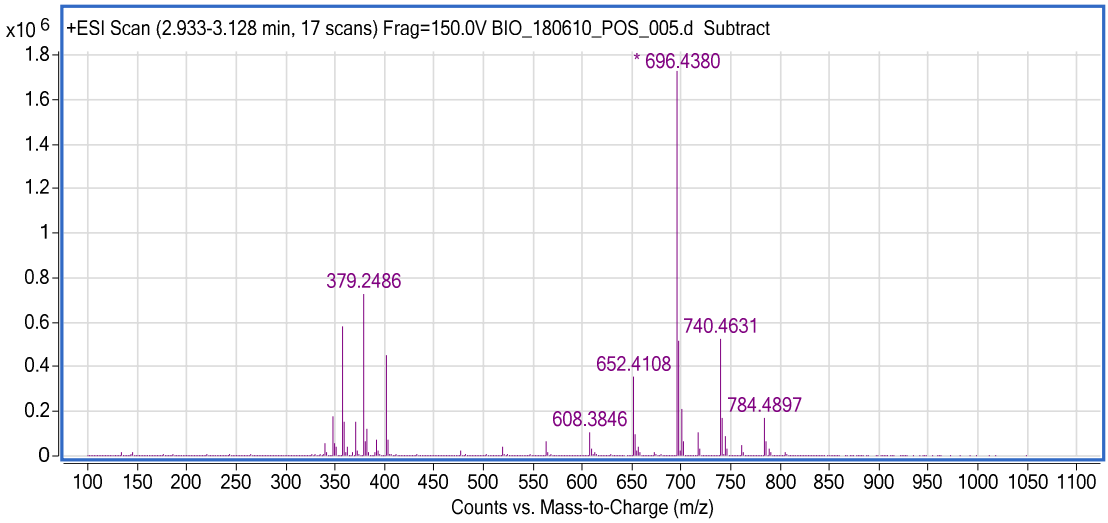
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C26H54O14	590.3514	0.03	

S09-012077 positive mode ESI, peak at 1.5-2.8 minutes



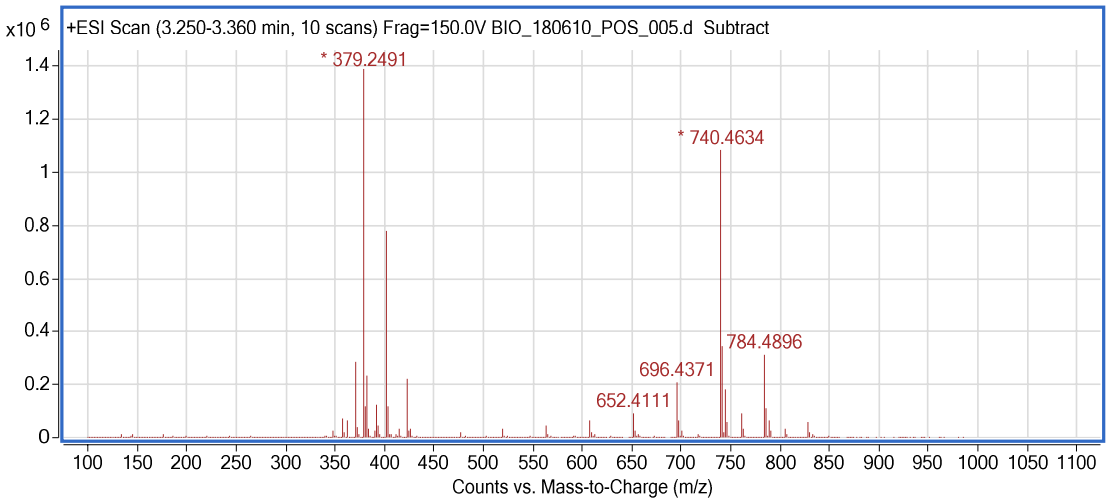
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C30H62O16	678.4024	-1.1	

S09-012077 positive mode ESI, peak at 3.0 minutes



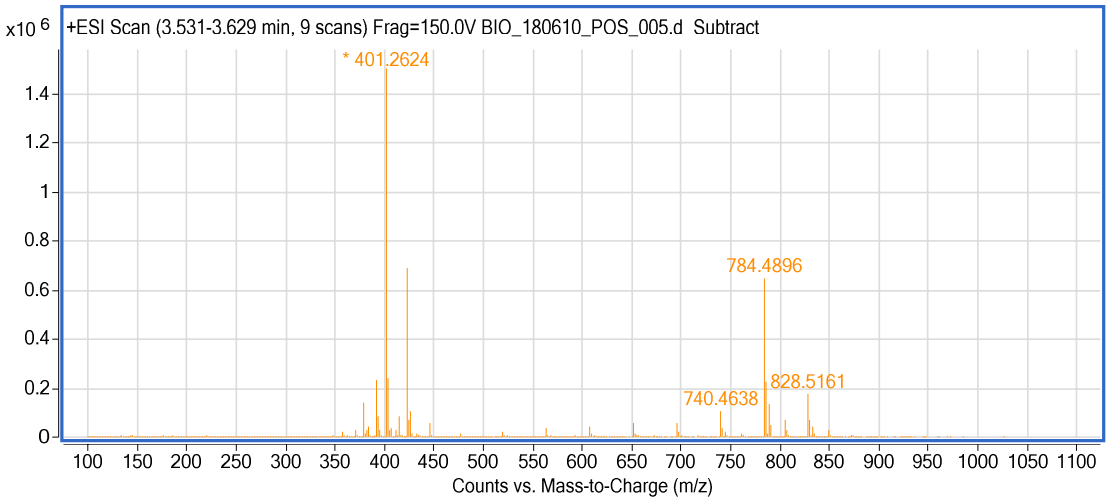
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C34H70O18	766.4562	0.92	

S09-012077 positive mode ESI, peak at 3.3 minutes



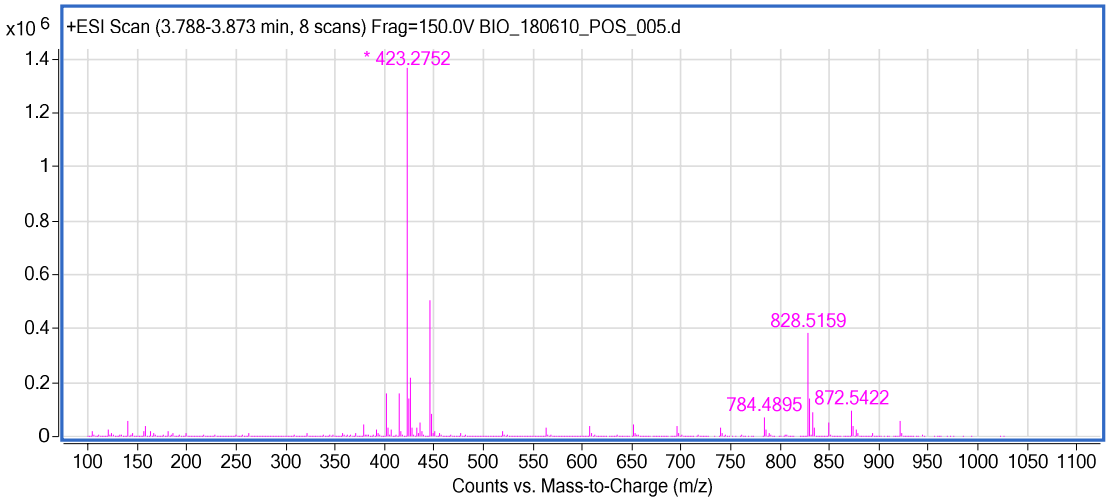
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012077 positive mode ESI, peak at 3.6 minutes



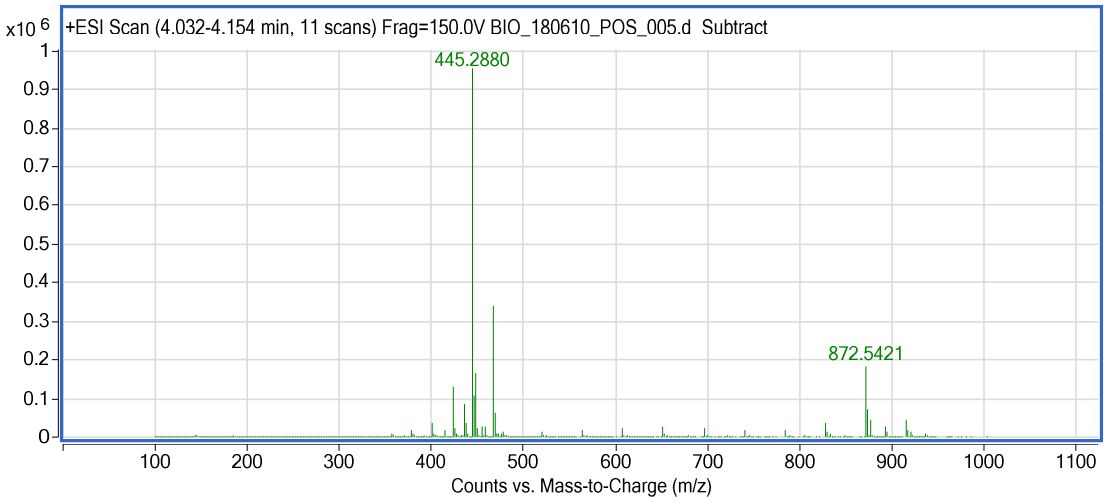
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012077 positive mode ESI, peak at 3.8 minutes



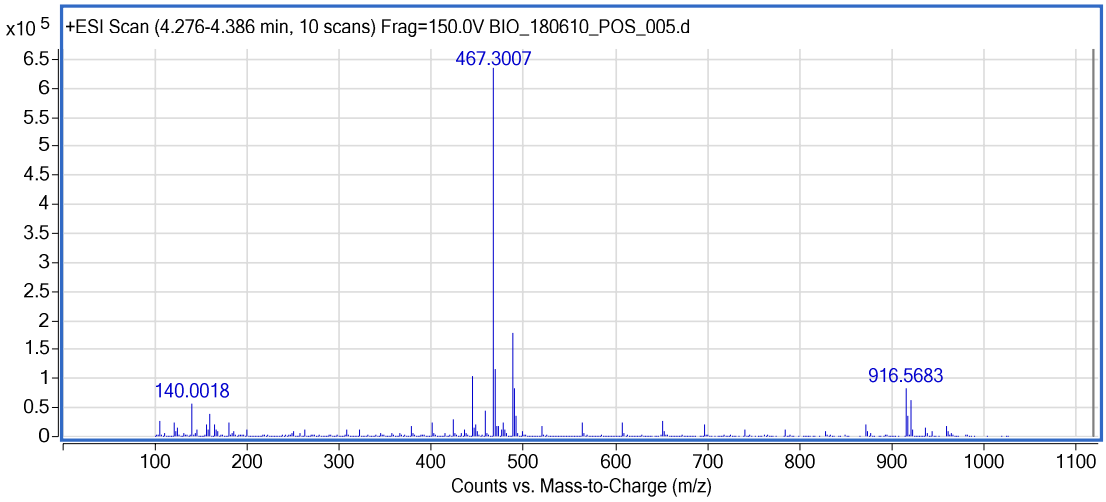
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012077 positive mode ESI, peak at 4.1 minutes



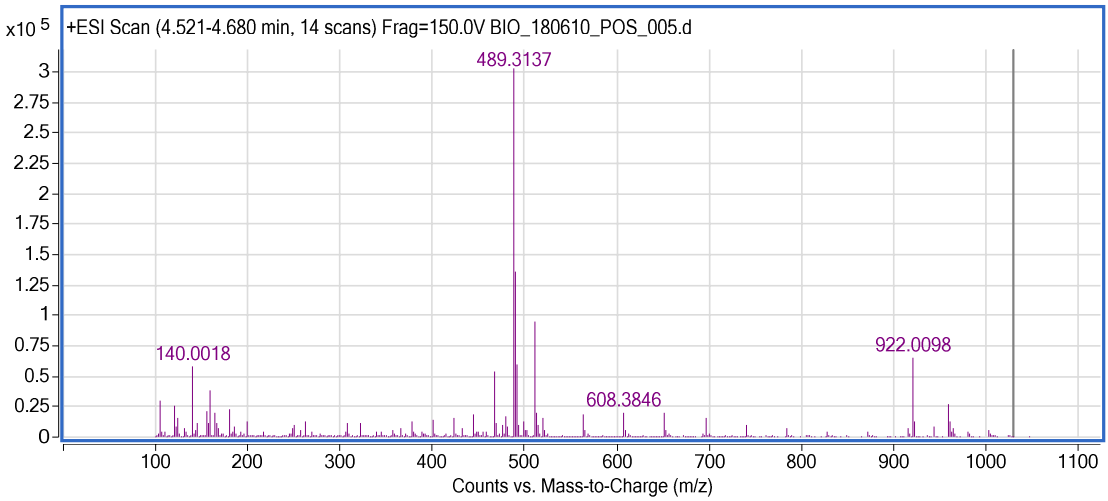
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012077 positive mode ESI, peak at 4.3 minutes



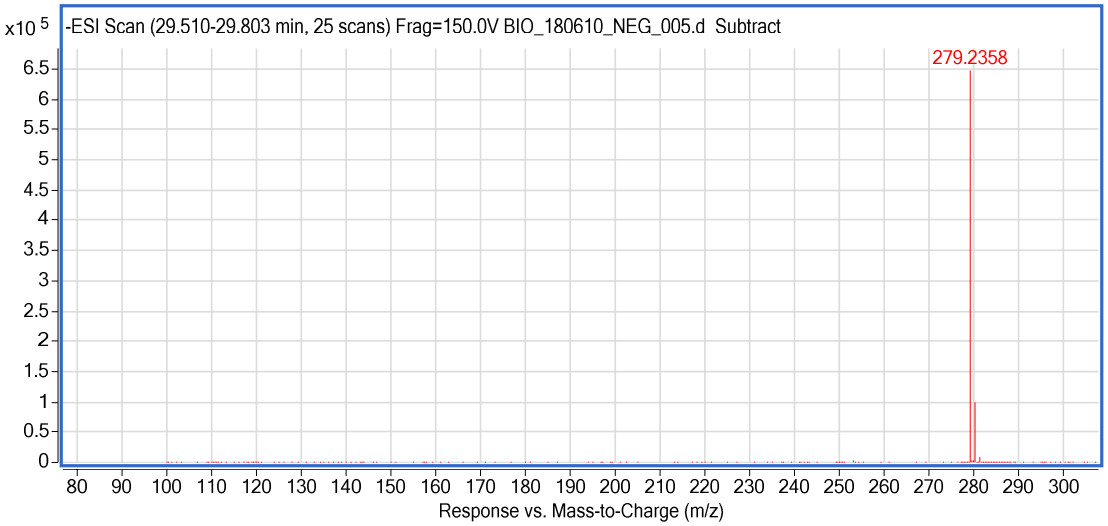
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012077 positive mode ESI, peak at 4.4 minutes



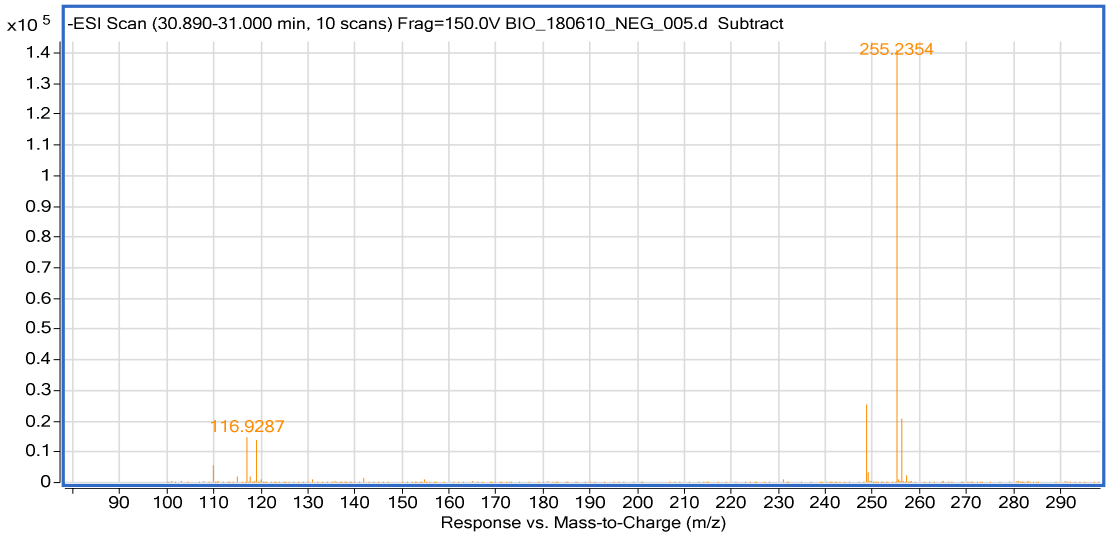
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012077 negative mode ESI, peak at 29.6 minutes



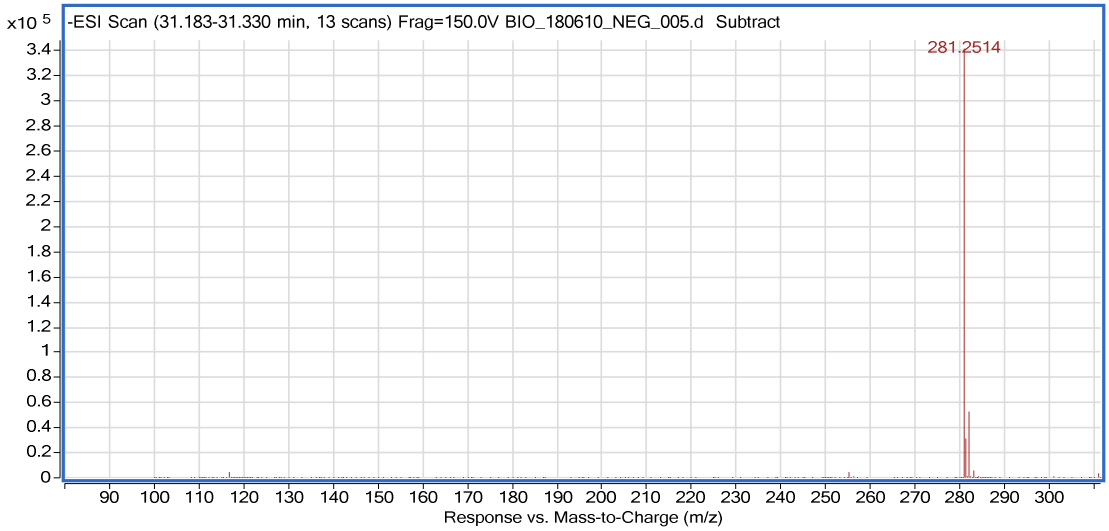
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H32O2	280.2431	-10	

S09-012077 negative mode ESI, peak at 31.0 minutes



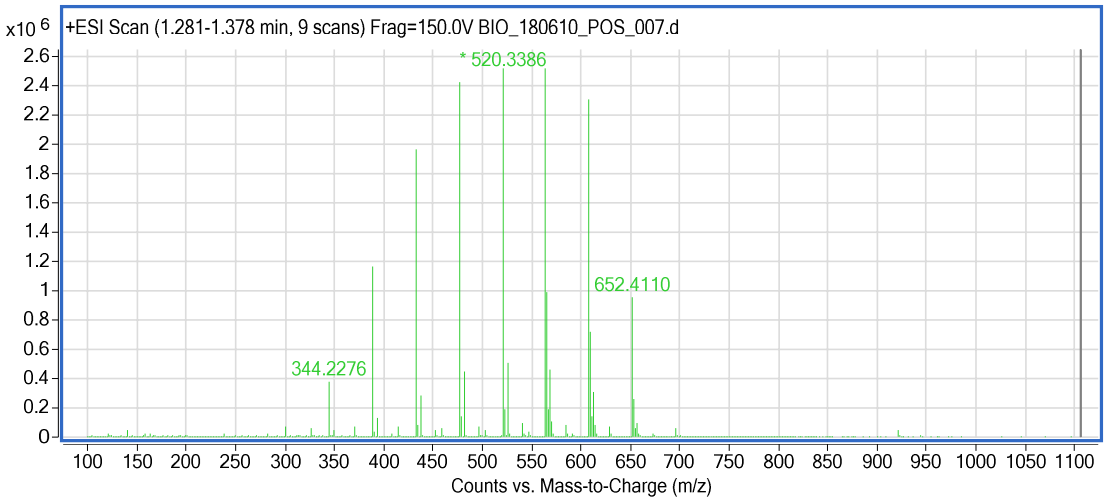
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2426	-0.94	

S09-012077 negative mode ESI, peak at 31.3 minutes



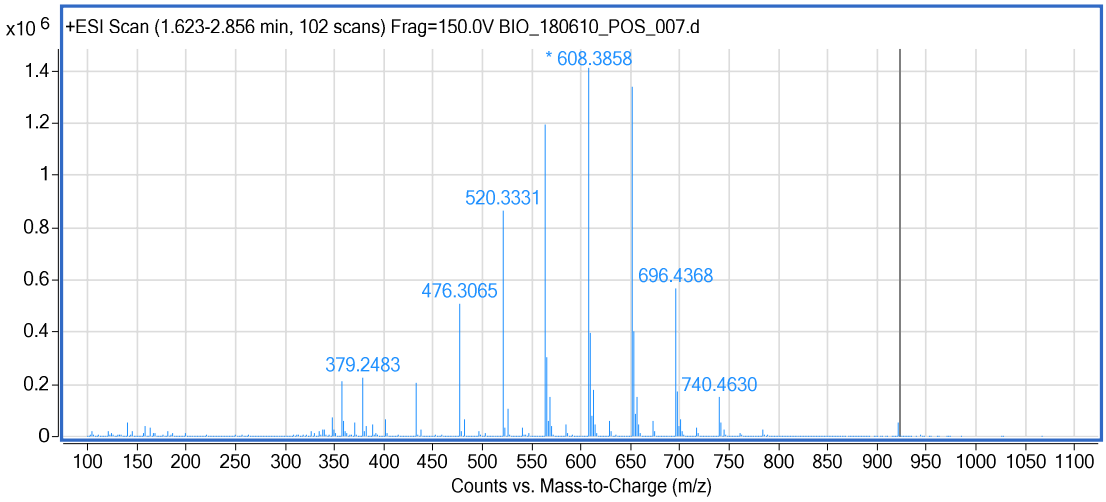
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O2	378.2485	-0.98	

S09-012078 positive mode ESI, peak at 1.3 minutes



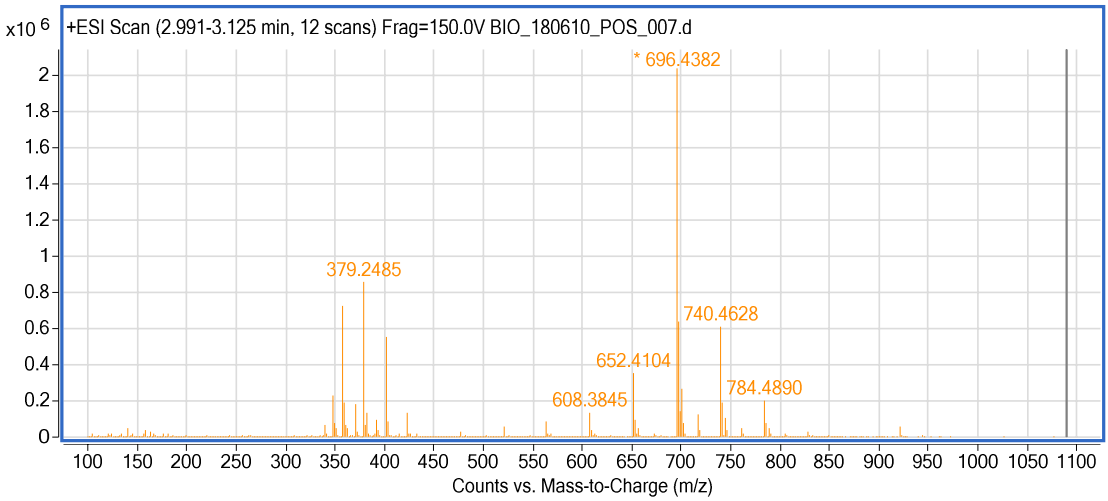
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C28H58O15	634.3776	0.66	

S09-012078 positive mode ESI, peak at 1.6-2.8 minutes



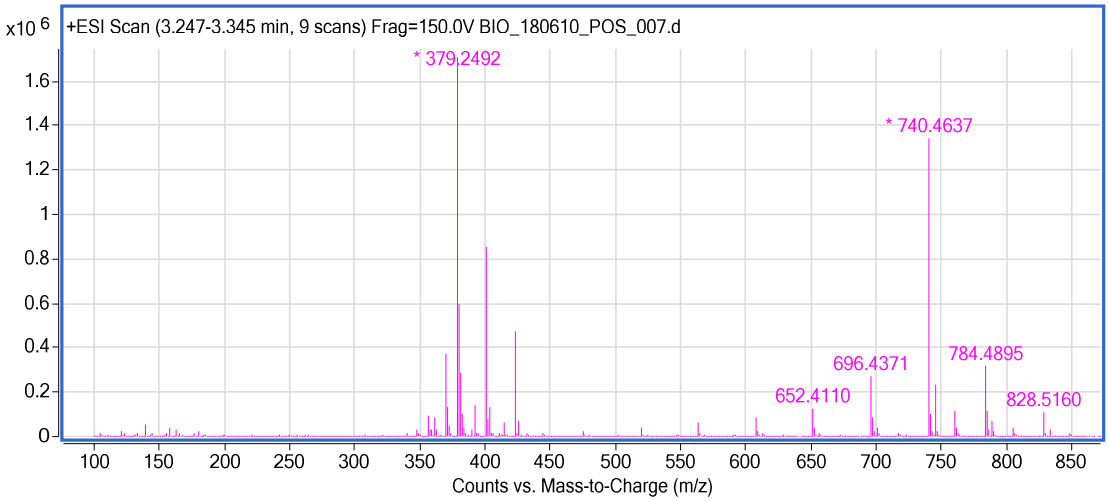
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C32H66O17	722.4300	1.2	

S09-012078 positive mode ESI, peak at 3.1 minutes



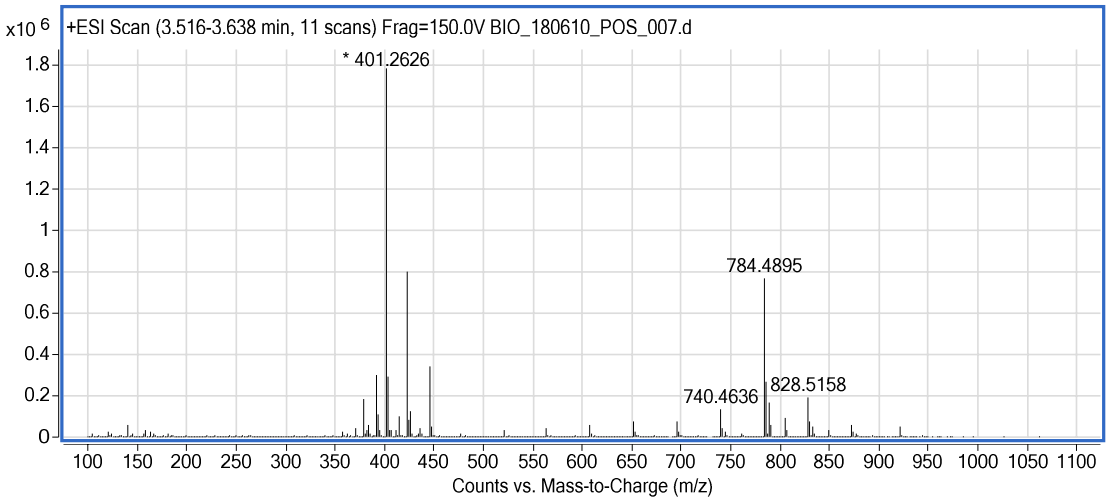
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012078 positive mode ESI, peak at 3.3 minutes



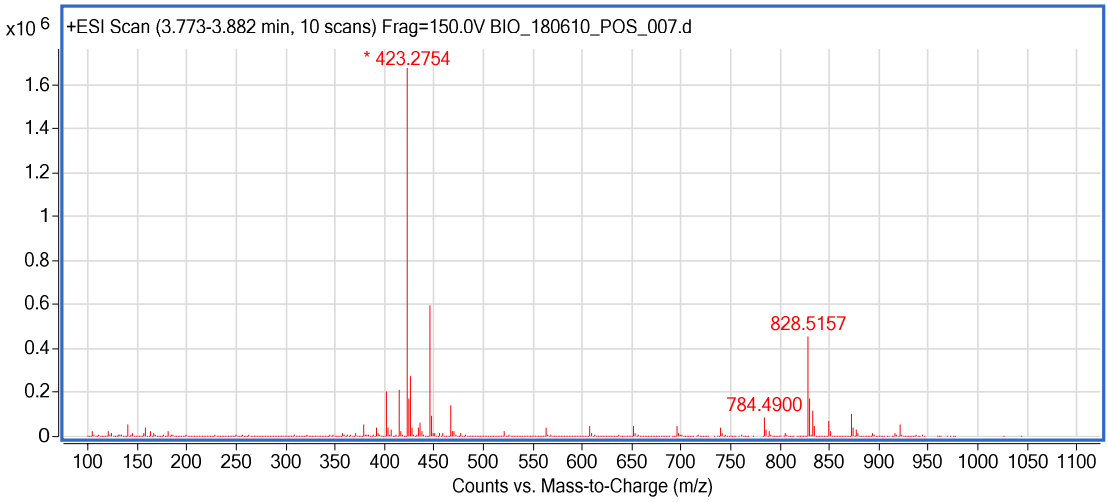
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012078 positive mode ESI, peak at 3.6 minutes



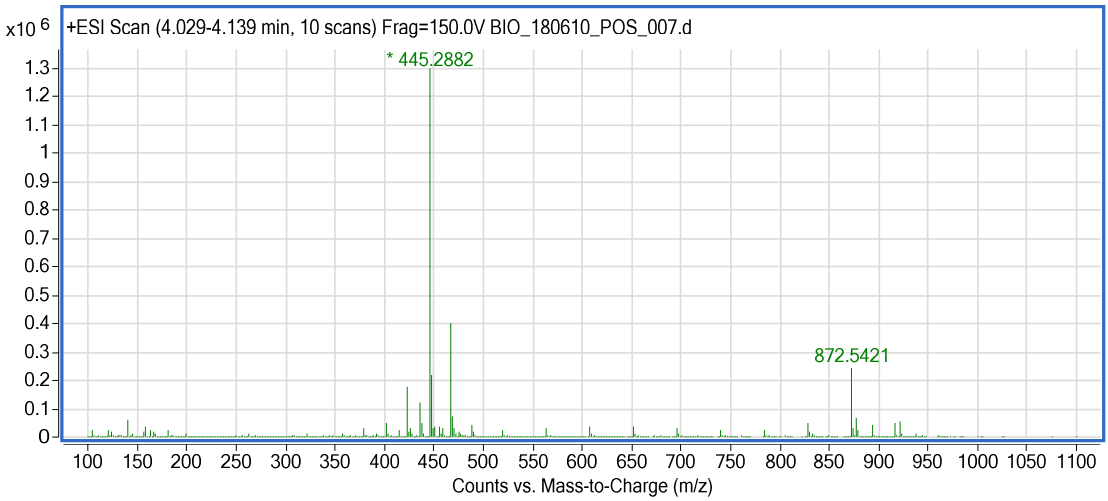
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012078 positive mode ESI, peak at 3.8 minutes



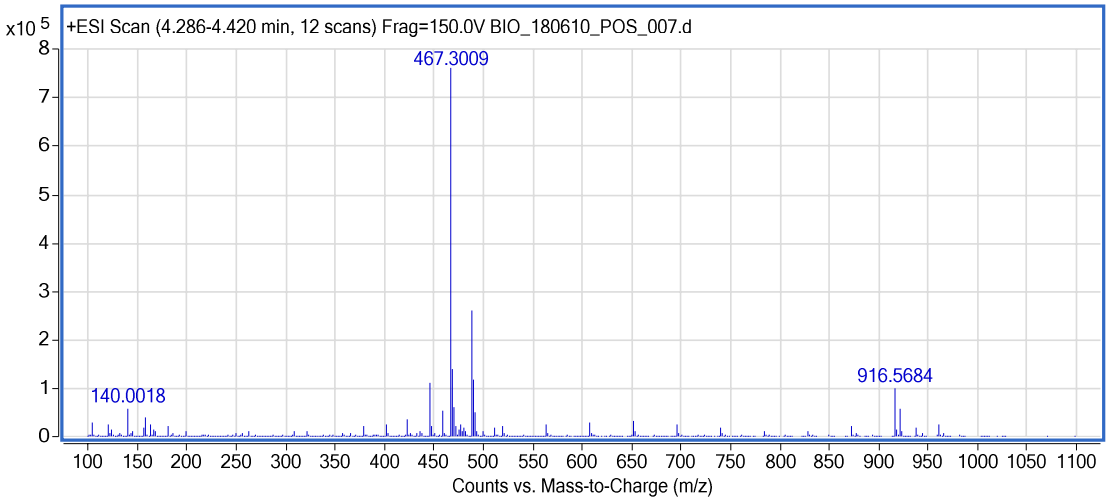
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012078 positive mode ESI, peak at 4.1 minutes



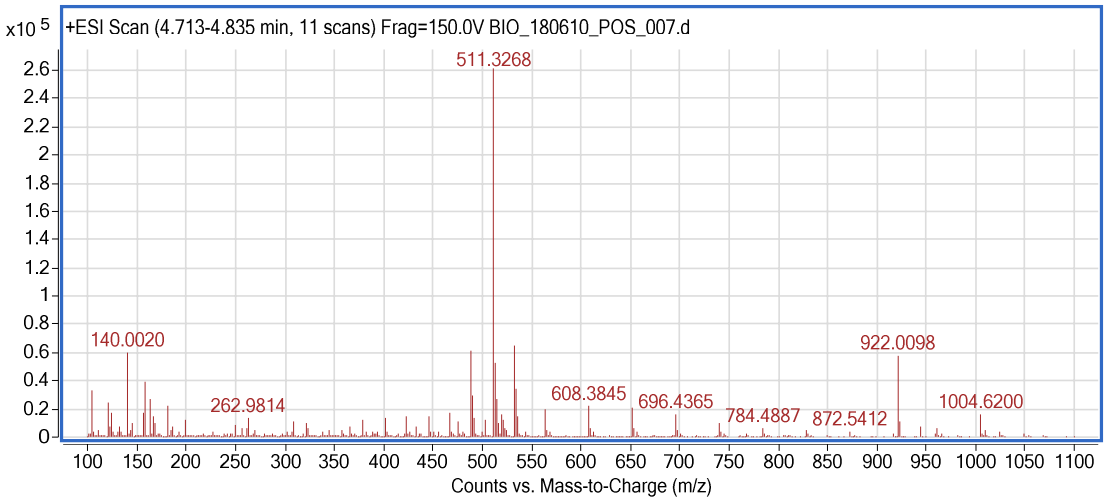
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012078 positive mode ESI, peak at 4.4 minutes



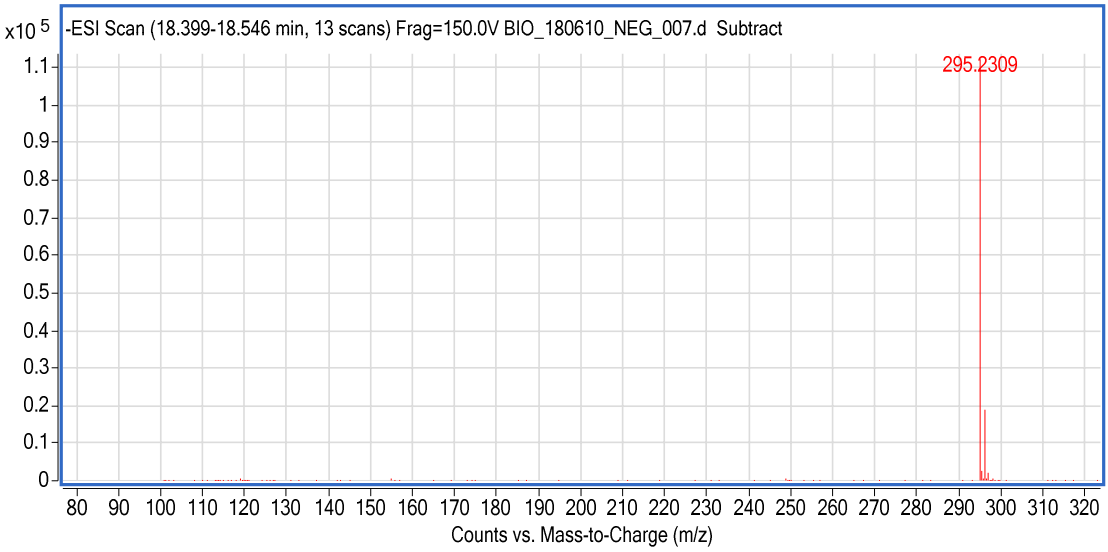
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012078 positive mode ESI, peak at 4.6 minutes



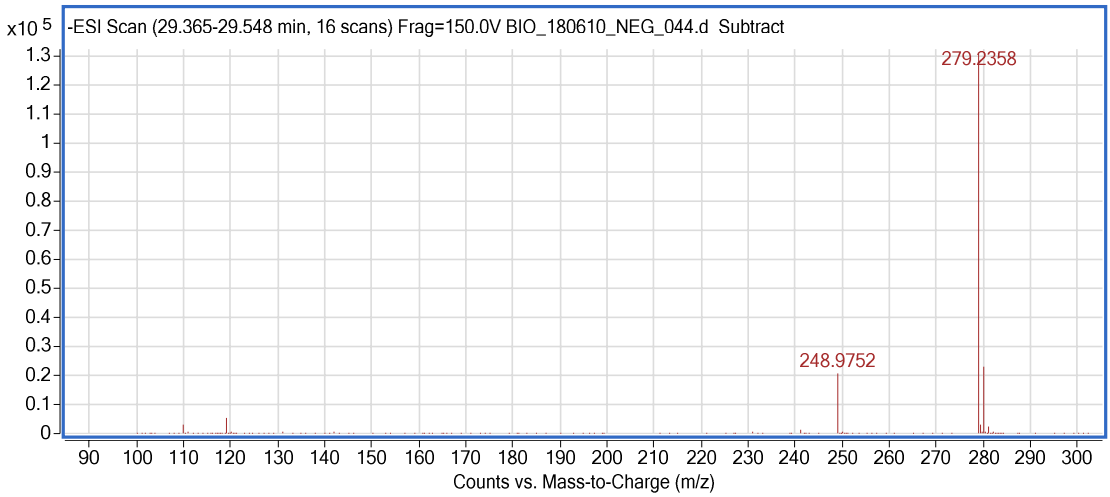
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	No good match on peak with highest m/z – part of oligomer series			

S09-012078 negative mode ESI, peak at 18.4 minutes



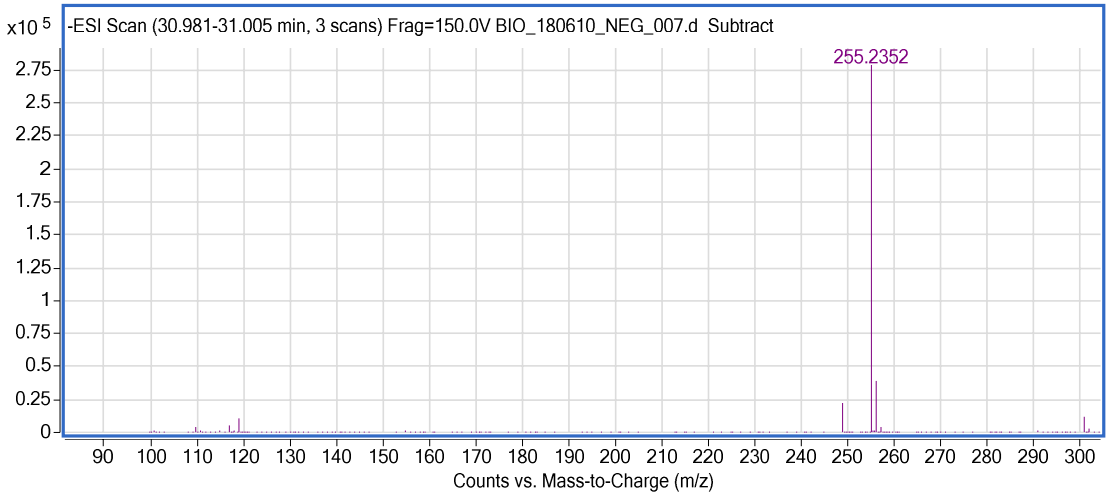
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C21H29N	296.2382	-2.5	

S09-012078 negative mode ESI, peak at 29.4 minutes



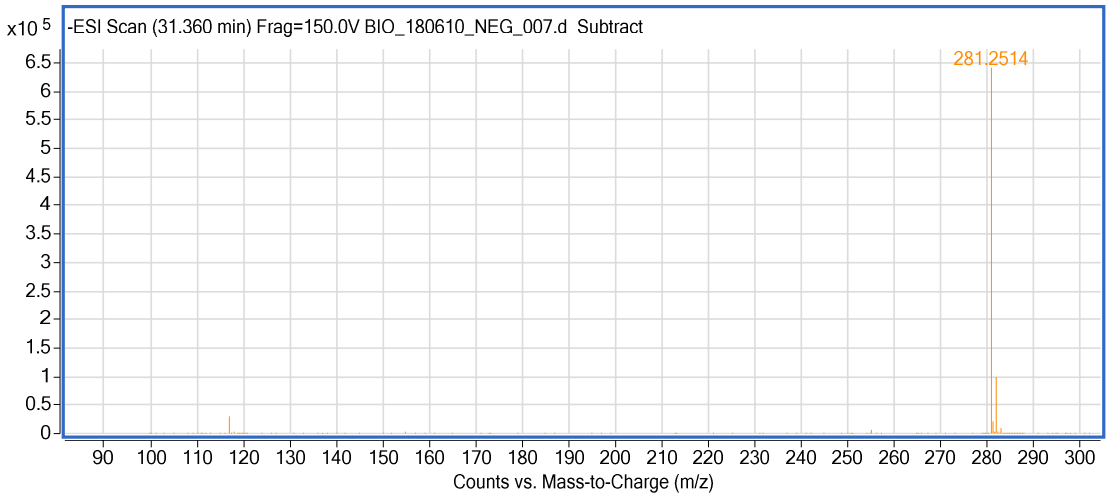
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H32O2	280.2430	-10.0	

S09-012078 negative mode ESI, peak at 31.0 minutes



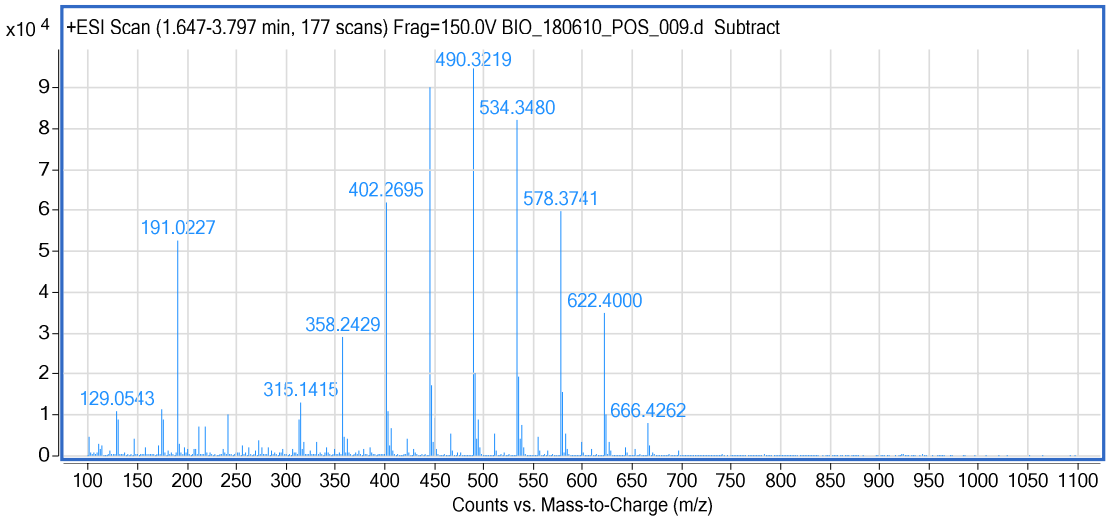
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2426	-9.4	

S09-012078 negative mode ESI, peak at 31.2 minutes



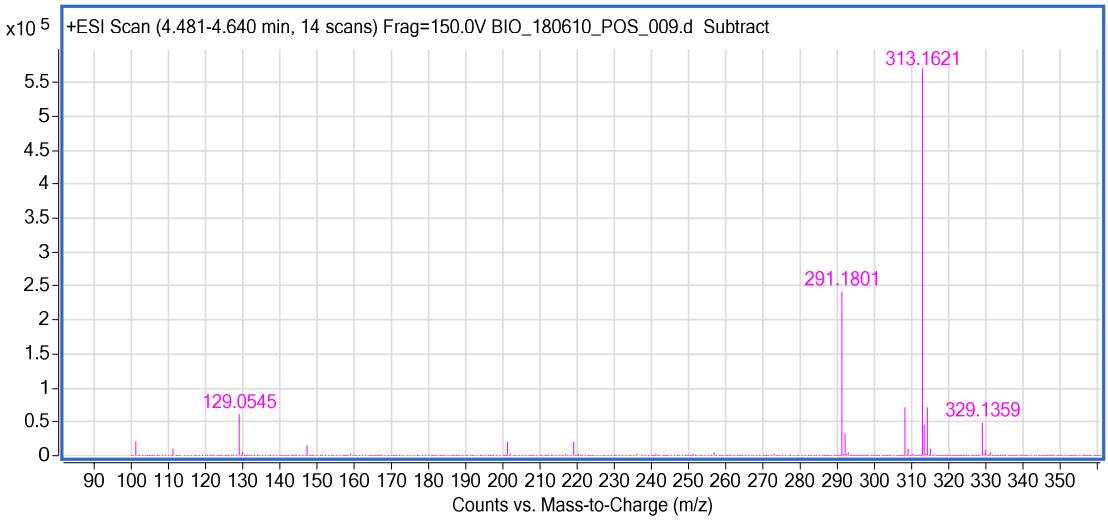
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O2	282.2588	-10.4	

S09-012079 positive mode ESI, peak at 1.8-3.8 minutes



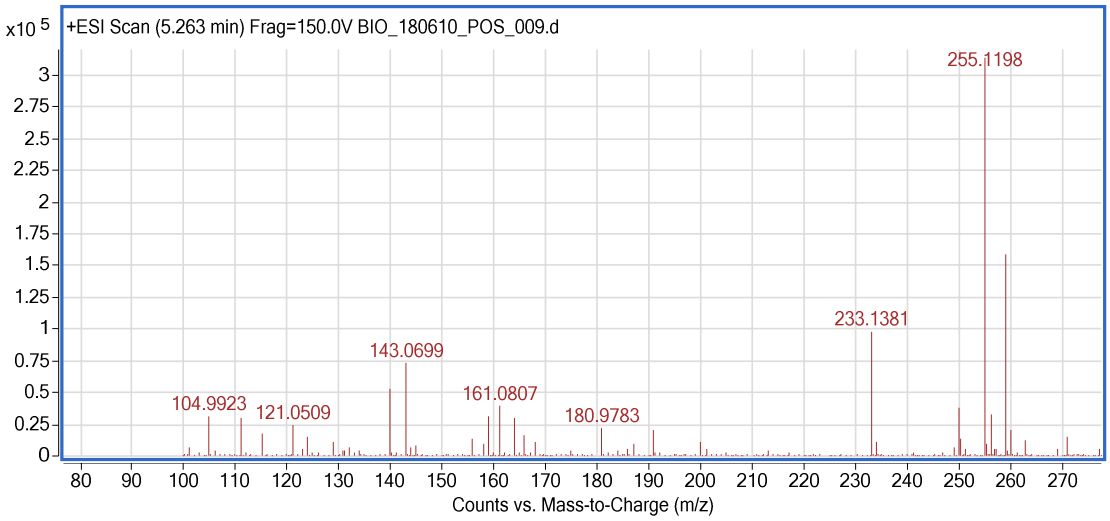
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	Part of oligomer series			

S09-012079 positive mode ESI, peak at 4.6 minutes



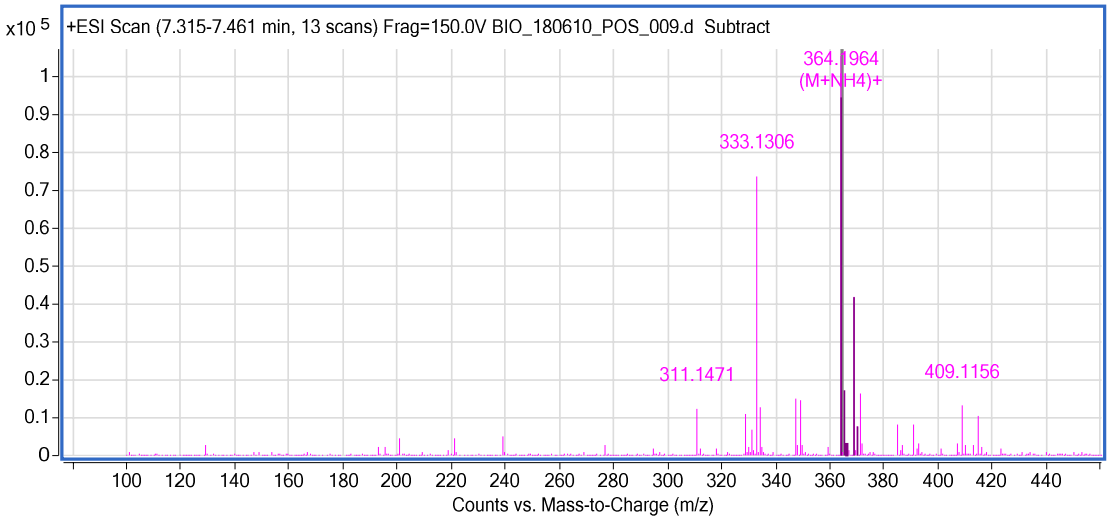
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C14H26O6	290.1729	0.36	

S09-012079 positive mode ESI, peak at 5.1 minutes



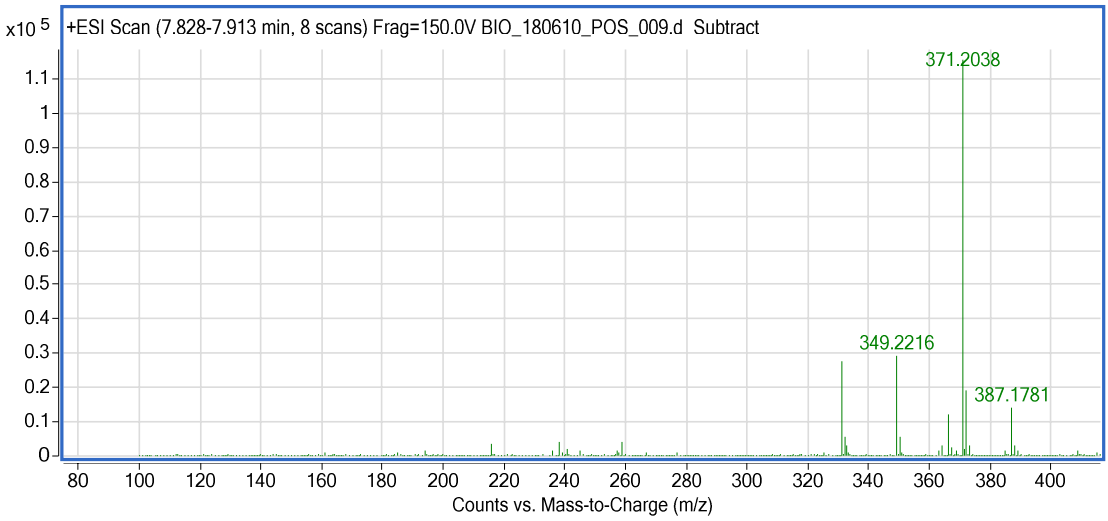
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C11H20O5	232.1331	1.9	
2	C9H18N3O4	232.1297	-3.8	

S09-012079 positive mode ESI, peak at 7.4 minutes



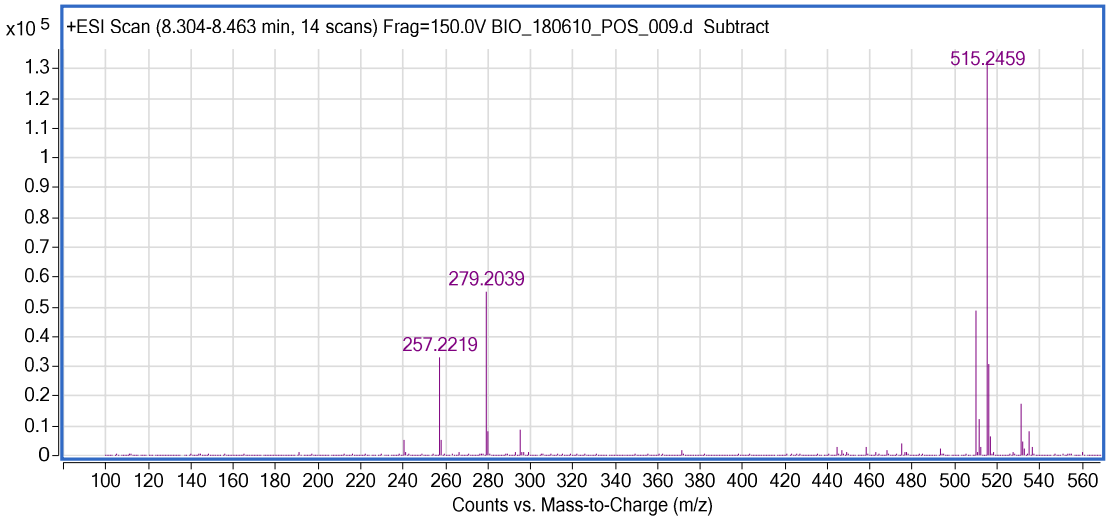
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H26O8	346.1628	0.55	

S09-012079 positive mode ESI, peak at 7.9 minutes



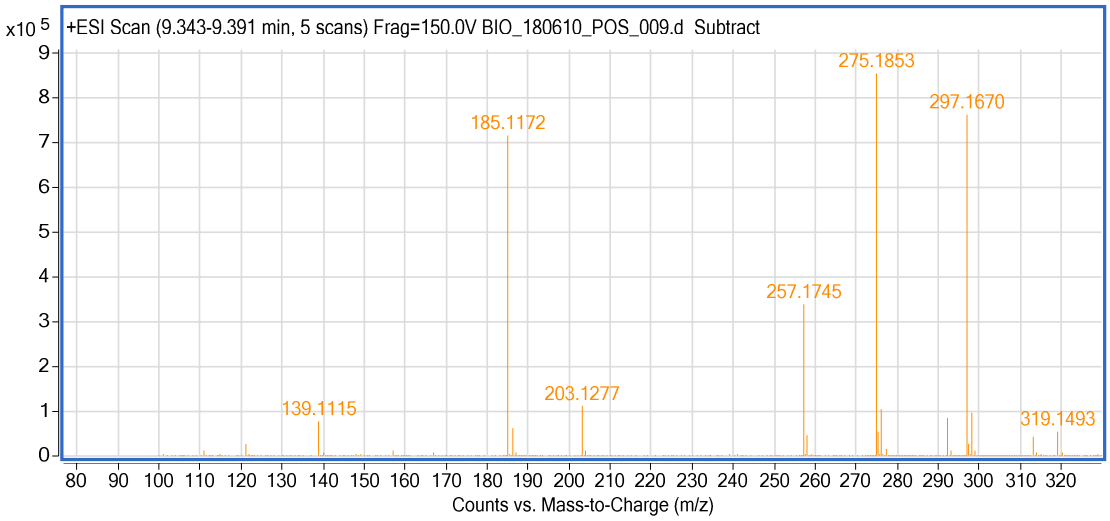
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C17H32O7	348.2148	0.55	
2	C15H30N3O6	348.2135	-3.3	

S09-012079 positive mode ESI, peak at 8.4 minutes



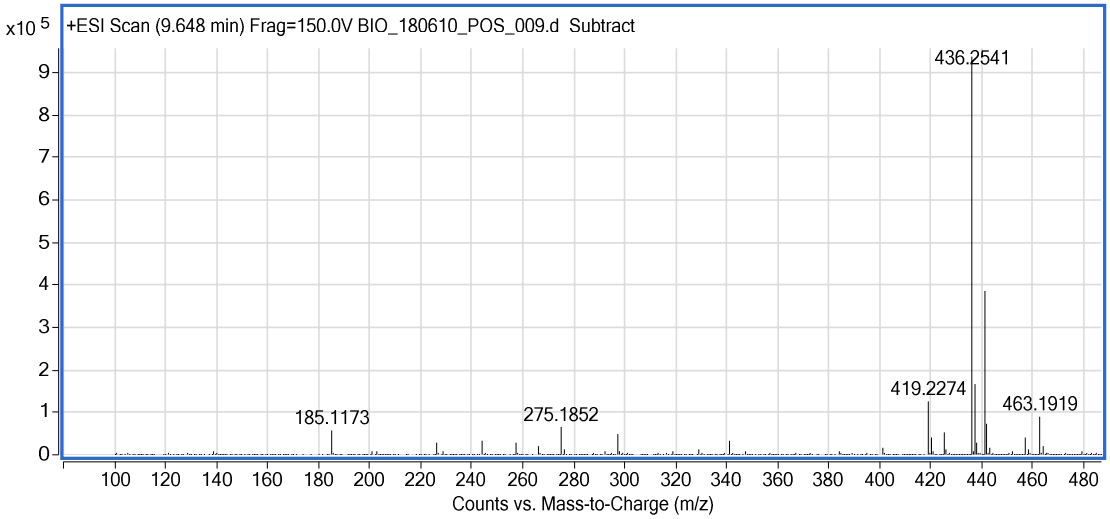
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C23H40O11	492.2571	0.79	
2	C21H38N3O10	492.2557	-1.9	
3	C22H34N7O6	492.2571	0.76	

S09-012079 positive mode ESI, peak at 9.3 minutes



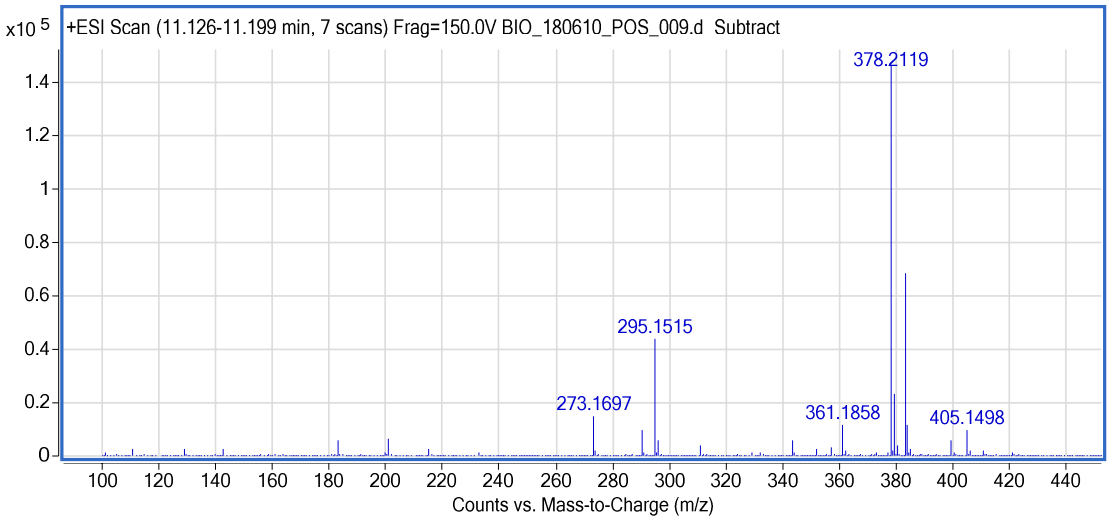
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C14H26O5	274.1780	0.94	
2	C12H24N3O4	274.1767	-4.0	
3	C13H20N7	274.1780	0.91	

S09-012079 positive mode ESI, peak at 9.6 minutes



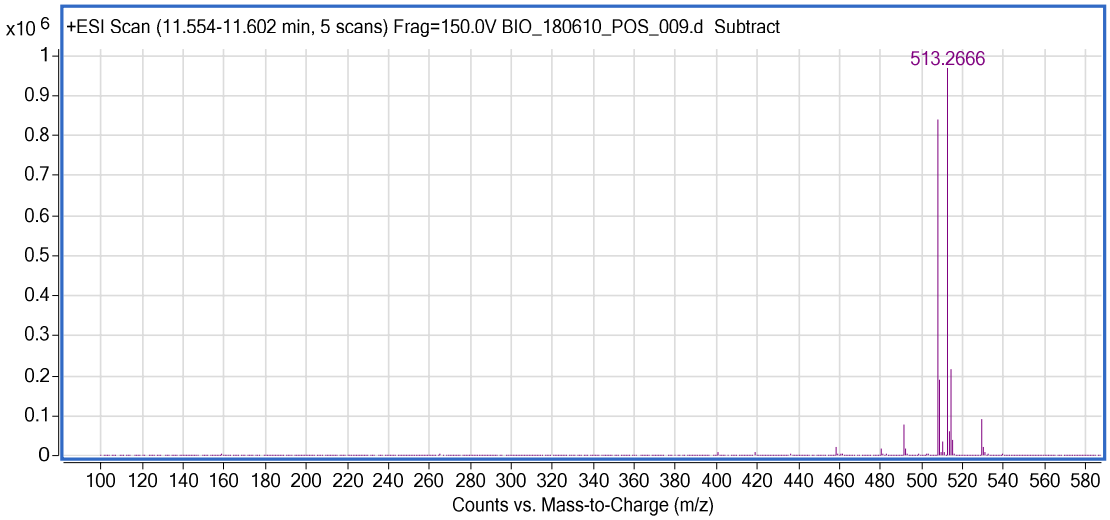
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H34O9	418.2203	0.64	
2	C18H32N3O8	418.2189	-2.6	
3	C19H28N7O4	418.2203	0.61	

S09-012079 positive mode ESI, peak at 11.2 minutes



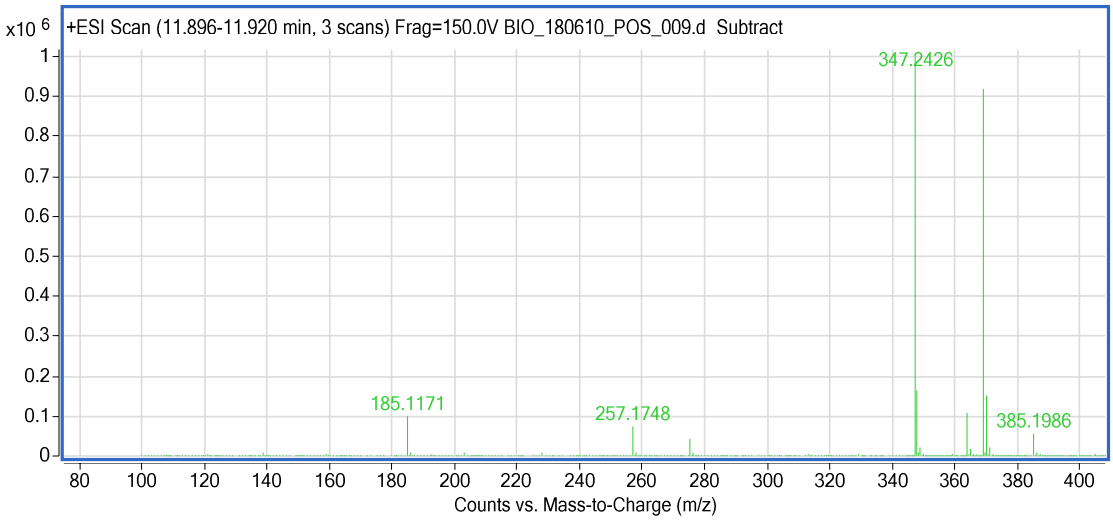
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C17H28O8	360.1784	0.42	
2	C15H26N3O7	360.1771	-3.3	

S09-012079 positive mode ESI, peak at 11.5 minutes



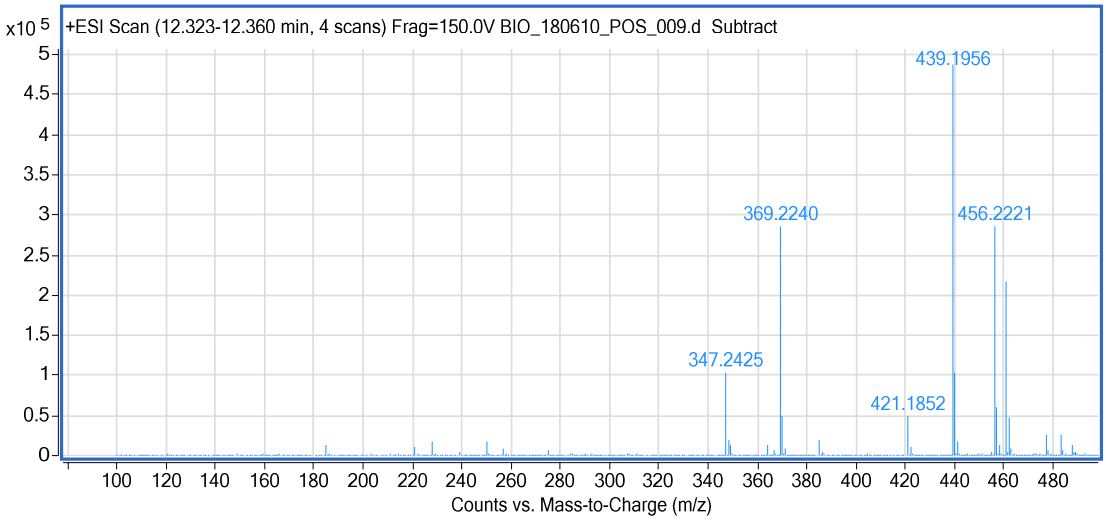
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C24H42O10	490.2778	0.76	
2	C22H40N3O9	490.2765	-1.9	

S09-012079 positive mode ESI, peak at 12.0 minutes



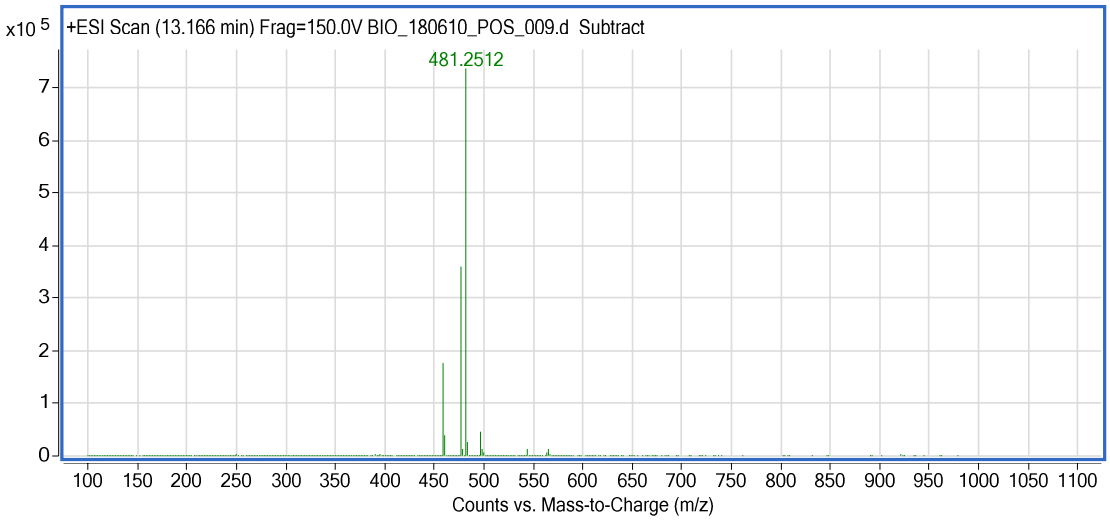
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O6	346.2355	0.31	
2	C16H32N3O5	346.2355	-3.6	

S09-012079 positive mode ESI, peak at 12.4 minutes



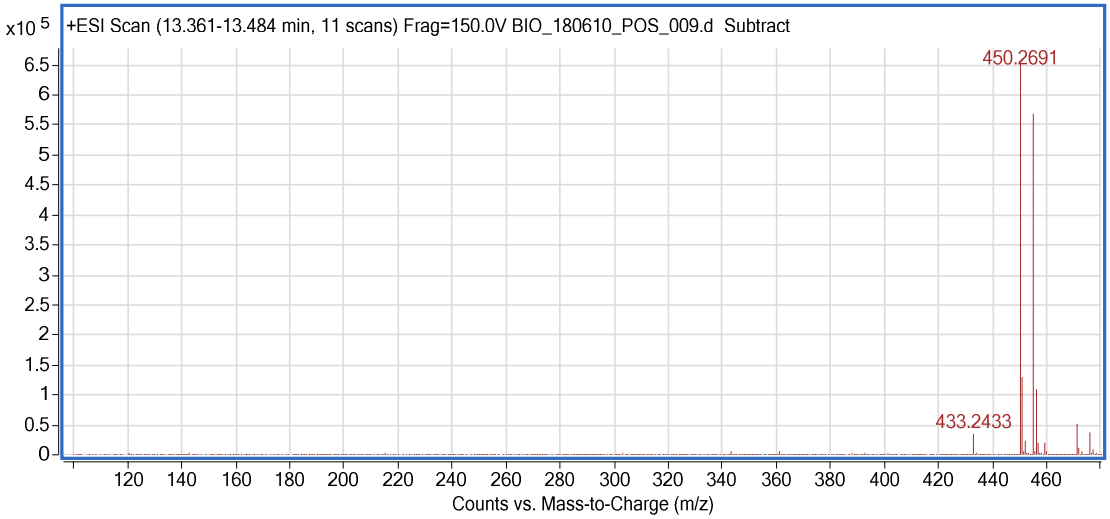
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H28N3O8	438.1876	-0.78	
2	C22H30O9	438.1890	2.3	

S09-012079 positive mode ESI, peak at 13.3 minutes



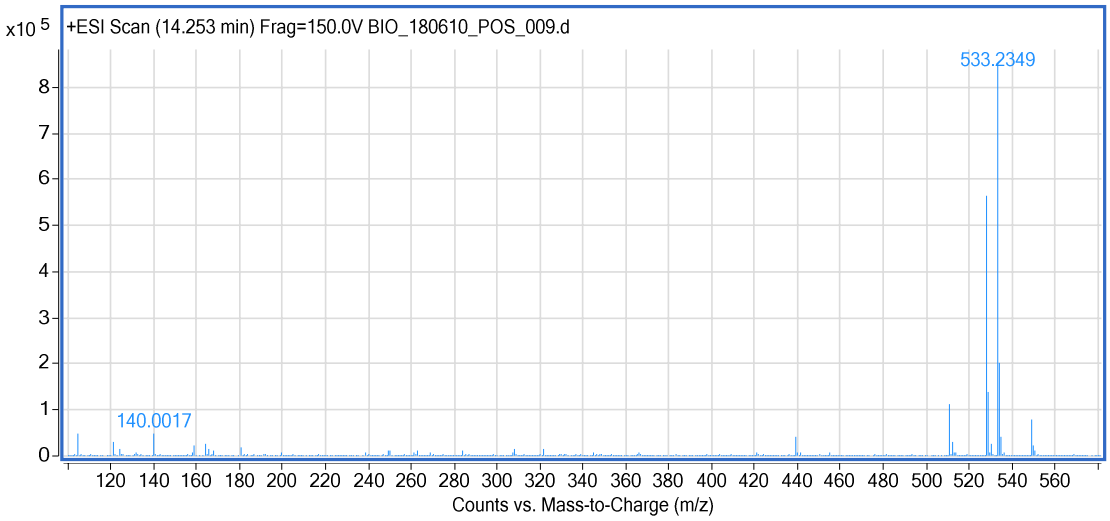
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H36N5O7	458.2615	-1.0	
2	C22H38N2O8	458.2628	1.9	
3	C19H40NO11	458.2601	-3.9	

S09-012079 positive mode ESI, peak at 13.5 minutes



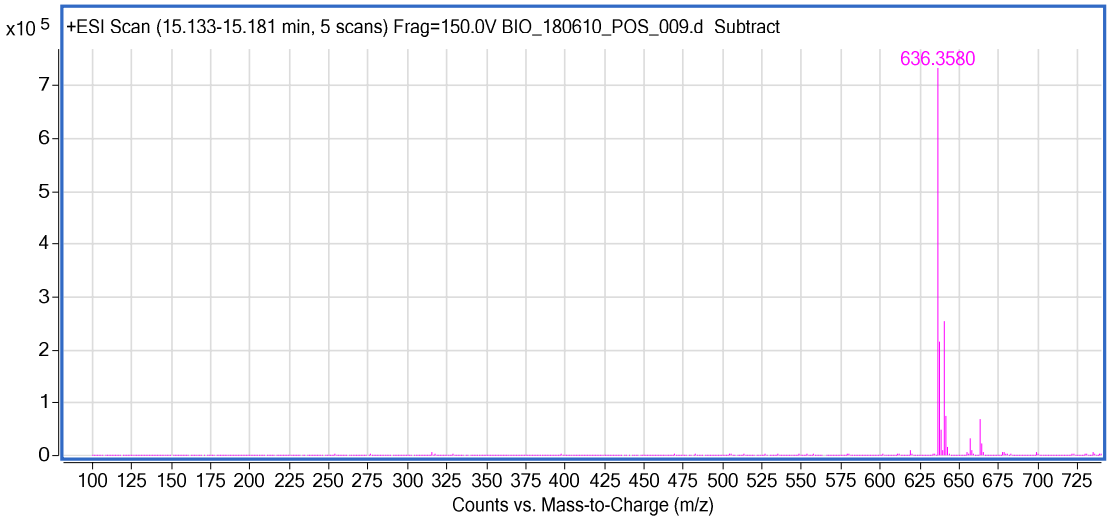
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C19H34N3O8	432.2346	-1.5	
2	C21H36O9	432.2359	1.6	

S09-012079 positive mode ESI, peak at 14.2 minutes



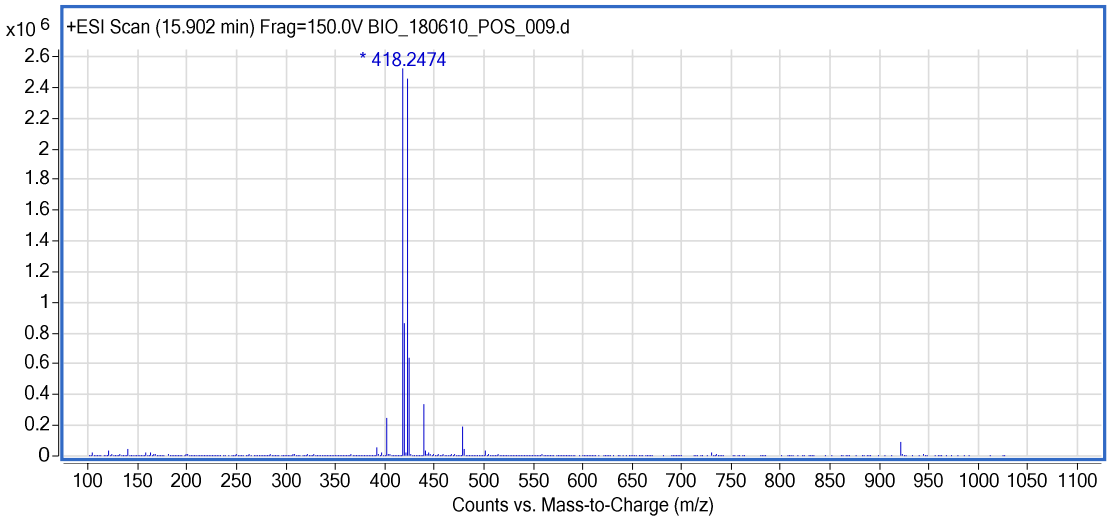
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C24H36N3O9	510.2452	-1.1	
2	C26H38O10	510.2465	1.6	

S09-012079 positive mode ESI, peak at 15.1 minutes



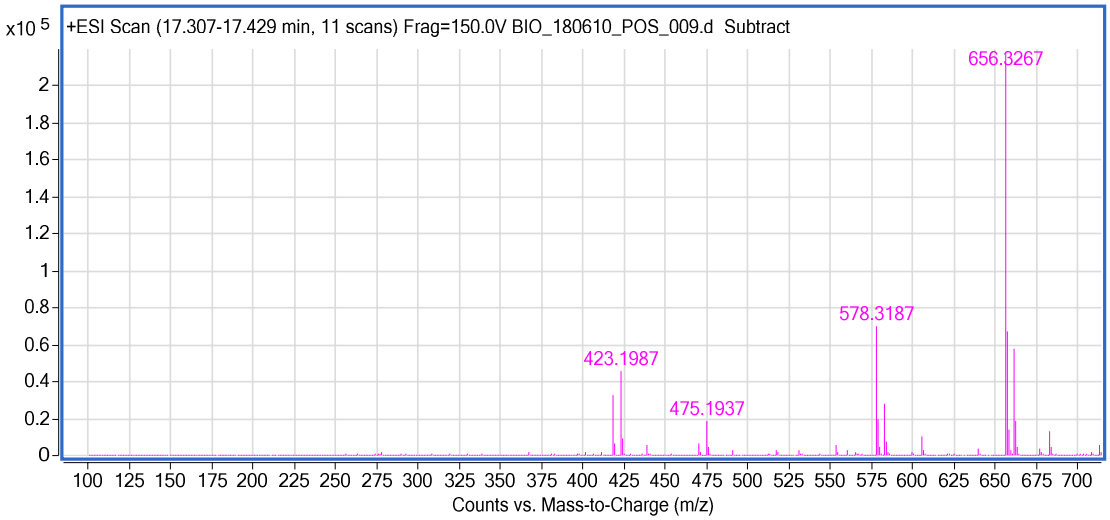
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C28H48N3O12	618.3238	-0.47	
2	C30H50O13	618.3251	1.7	

S09-012079 positive mode ESI, peak at 16.1 minutes



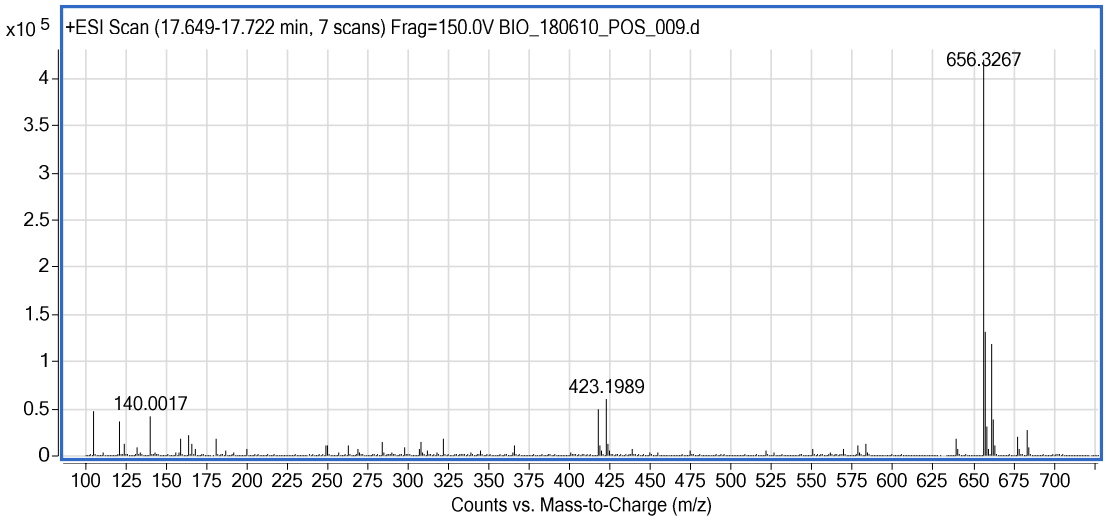
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C30H32O8	400.2097	1.2	
2	C18H30N3O7	400.2084	-2.2	

S09-012079 positive mode ESI, peak at 17.3 minutes



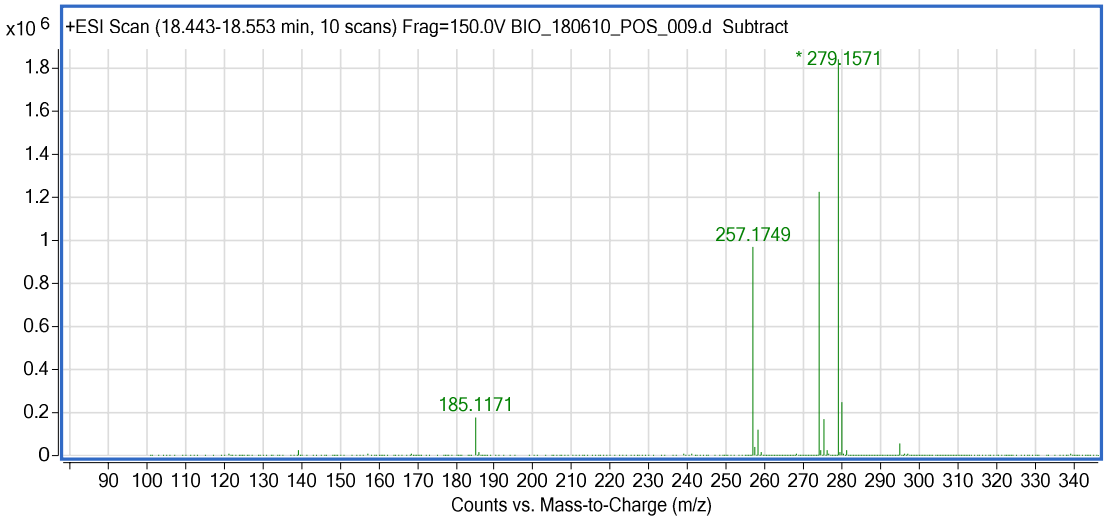
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C30H44N3O12	638.2925	-0.8	
2	C32H46O13	638.2938	1.3	
3	C24H50N2O15S	638.2932	0.28	

S09-012079 positive mode ESI, peak at 17.7 minutes



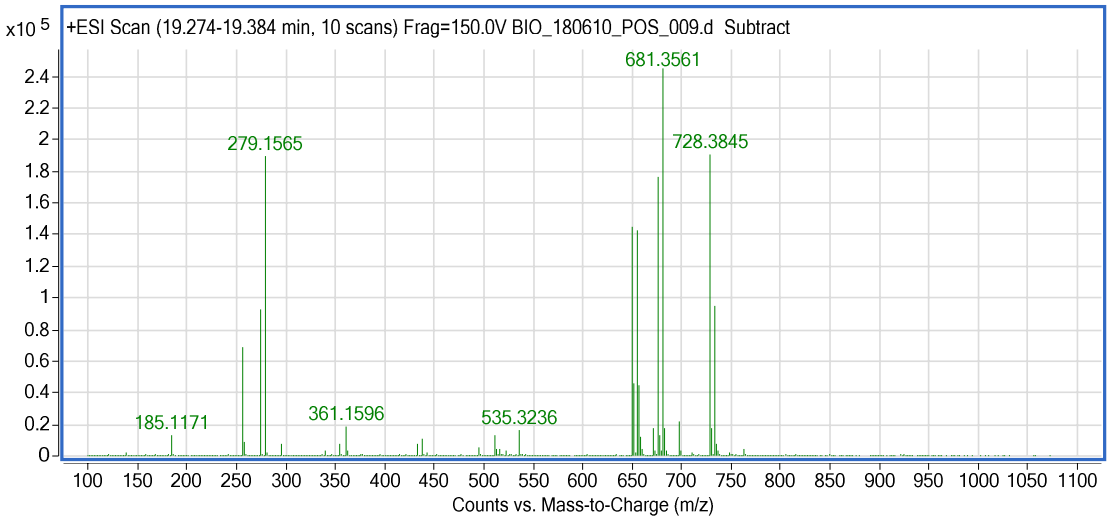
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C30H44N3O12	638.2925	-0.77	
2	C32H46O13	638.2938	1.4	

S09-012079 positive mode ESI, peak at 18.5 minutes



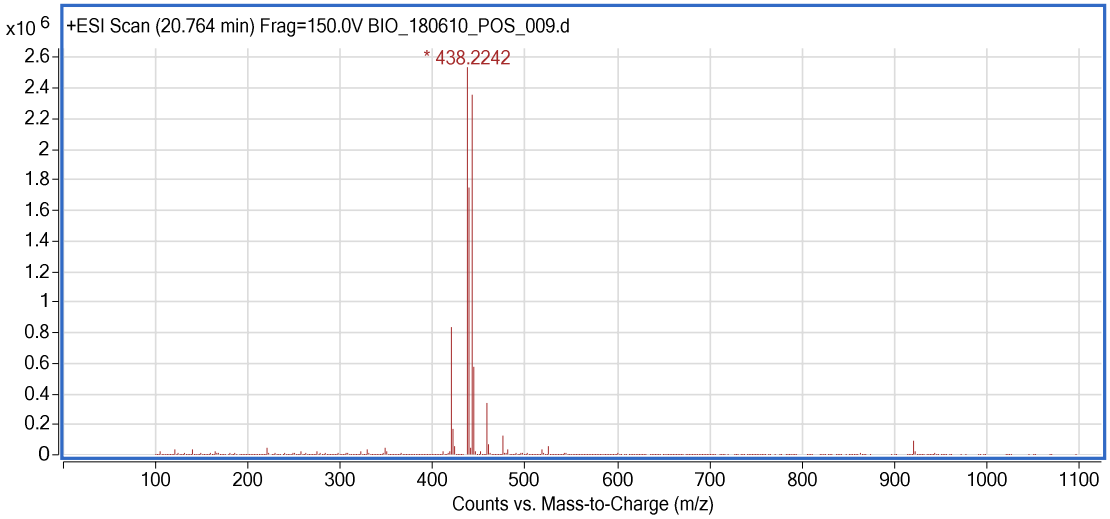
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C14H24O4	256.1675	-0.02	

S09-012079 positive mode ESI, peak at 19.3 minutes



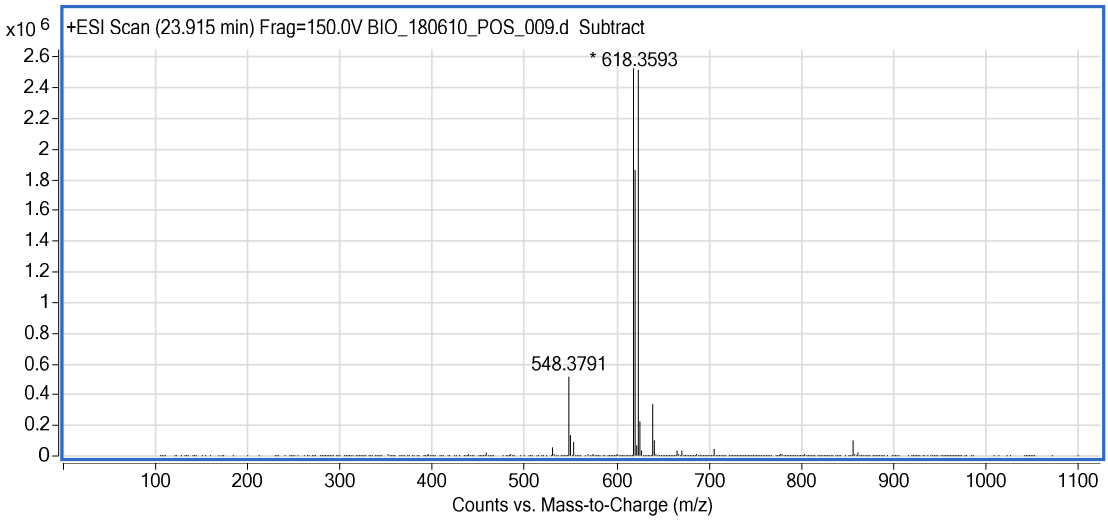
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C34H52N3O13	710.3500	-0.92	
2	C36H54O14	710.3514	0.98	
3	C28H58N2O16S	710.3507	0.05	

S09-012079 positive mode ESI, peak at 20.9 minutes



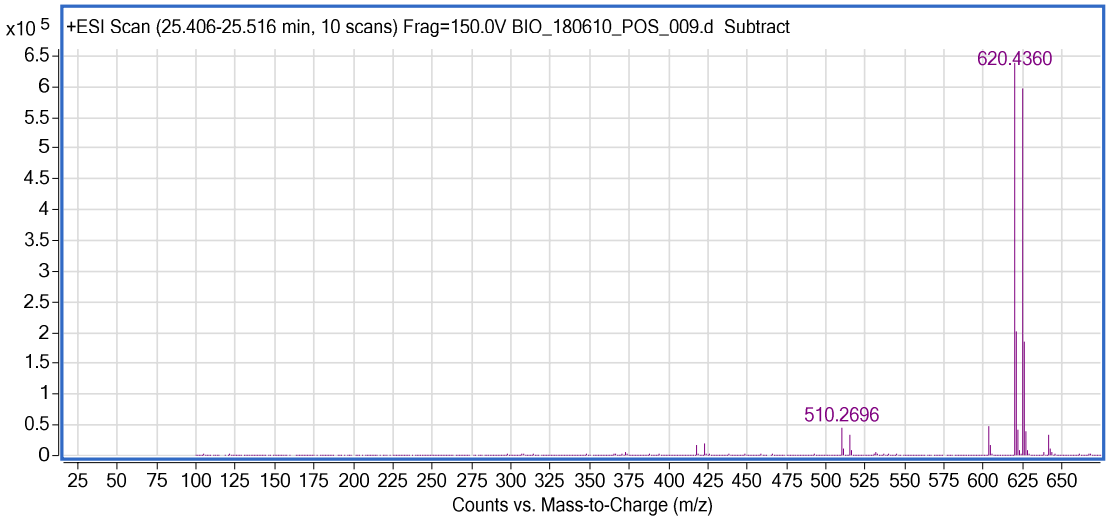
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H28O8	420.1784	0.62	
2	C20H26N3O7	420.1771	-2.6	
3	C23H24N4O4	420.1798	3.8	

S09-012079 positive mode ESI, peak at 23.9 minutes



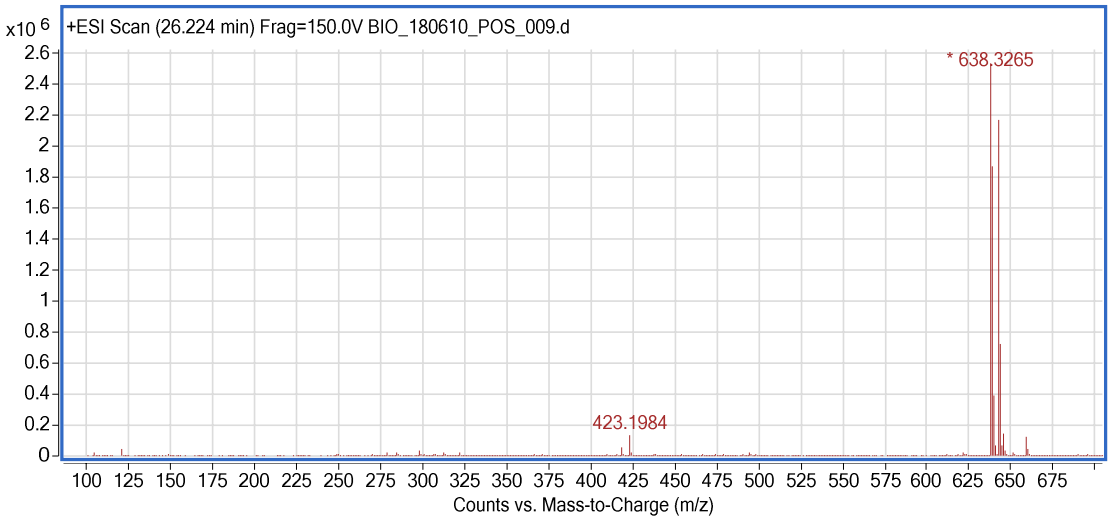
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C28H46N3O11	600.3132	-0.75	
2	C30H48O12	600.3146	1.5	

S09-012079 positive mode ESI, peak at 25.5 minutes



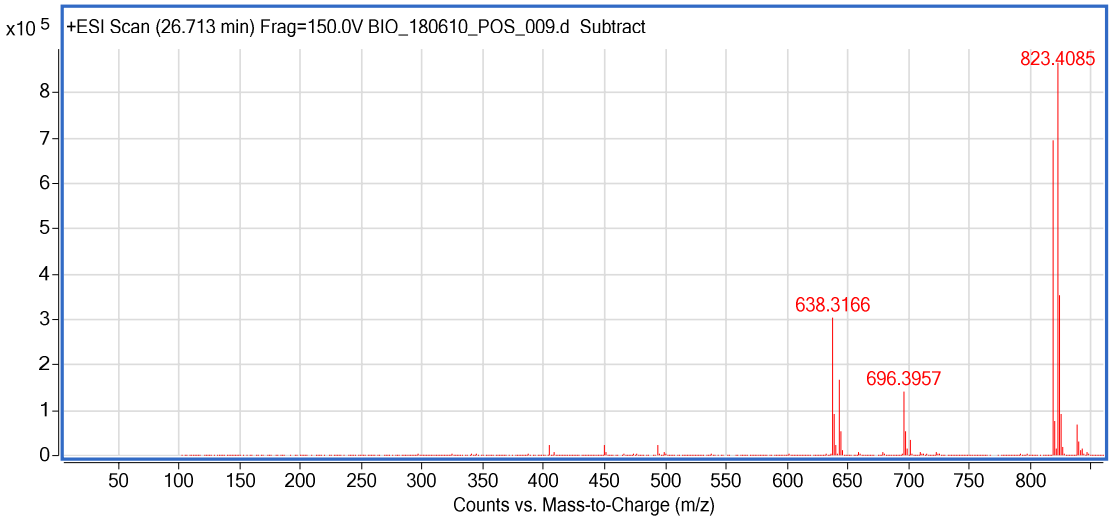
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C32H58O10	602.4030	1.4	

S09-012079 positive mode ESI, peak at 26.1 minutes



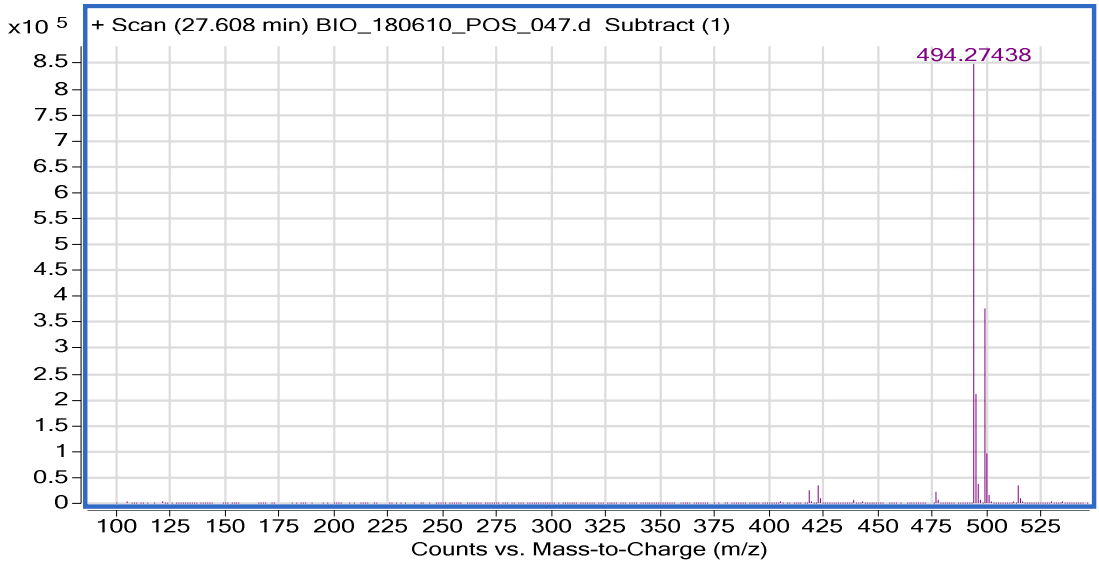
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C30H42N3O11	620.2819	-0.51	
2	C32H44O12	620.2833	1.7	

S09-012079 positive mode ESI, peak at 26.8 minutes



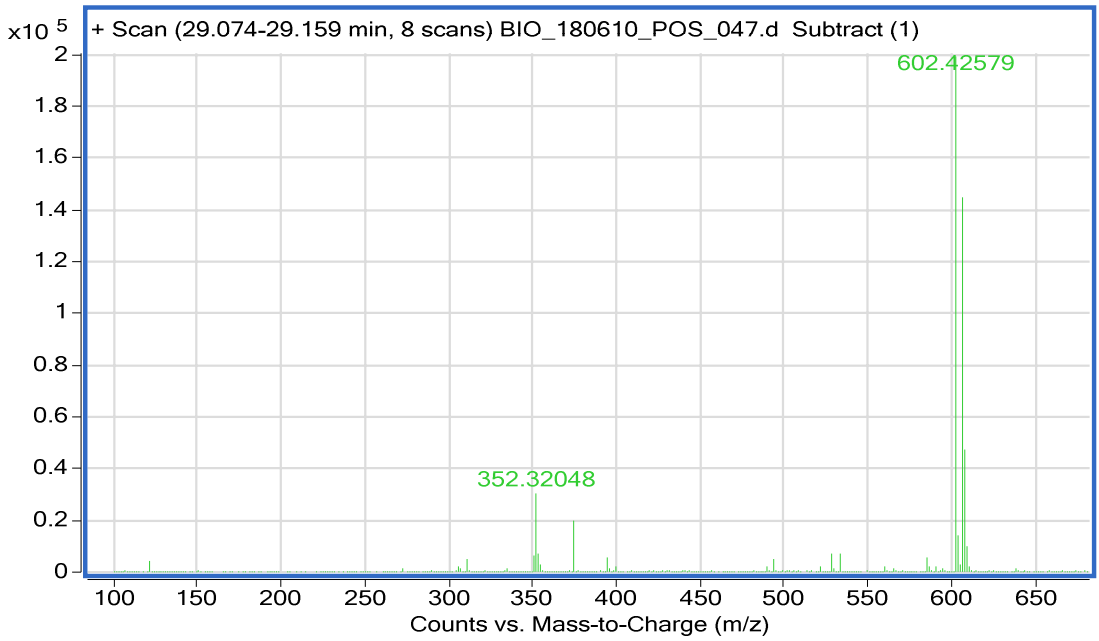
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C40H64O16	800.4194	0.2	

S09-012079 positive mode ESI, peak at 27.9 minutes



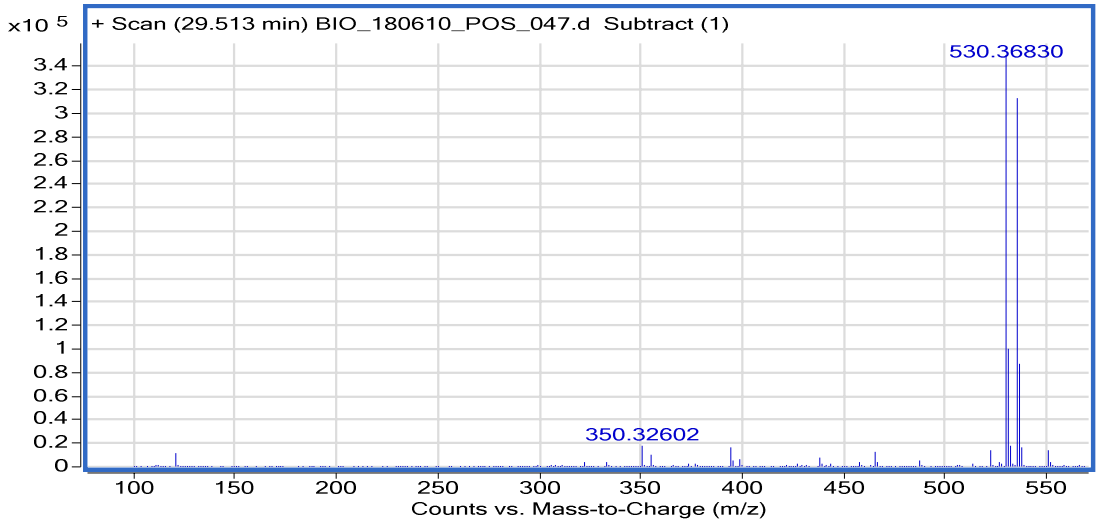
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C26H36O8	476.2406	0.98	

S09-012079 positive mode ESI, peak at 28.9 minutes



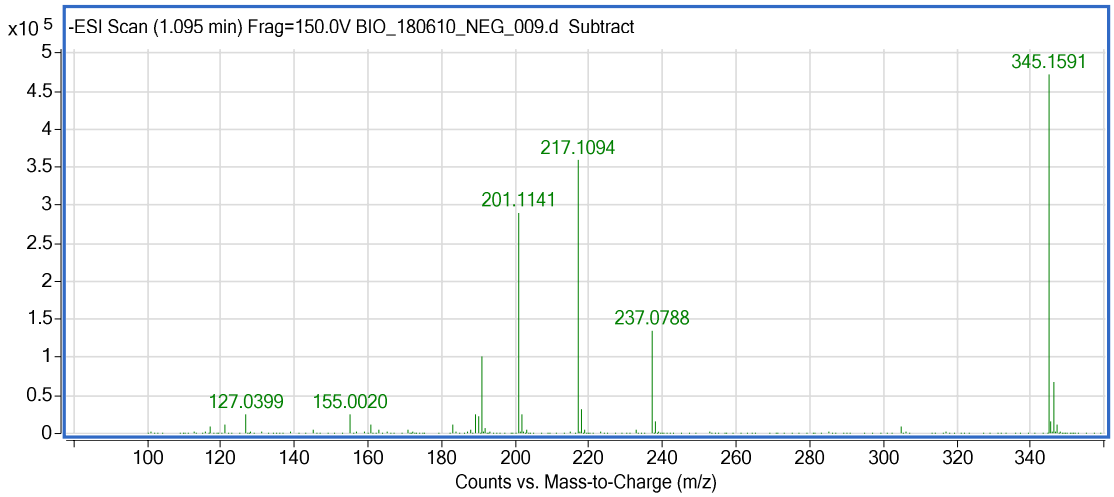
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C32H56O9	584.3924	0.8	

S09-012079 positive mode ESI, peak at 29.7 minutes



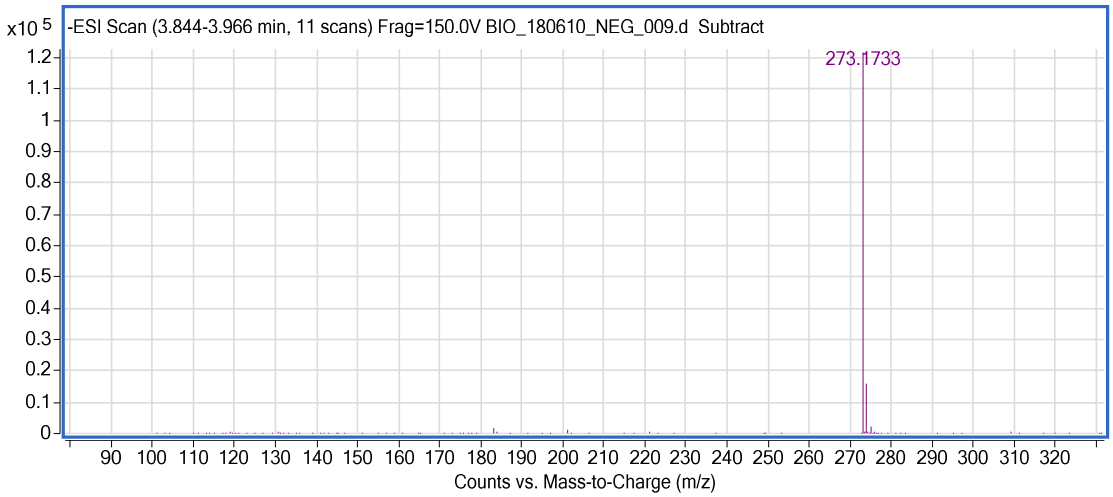
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C28H48O8	512.3349	0.89	

S09-012079 negative mode ESI, peak at 1.1 minutes



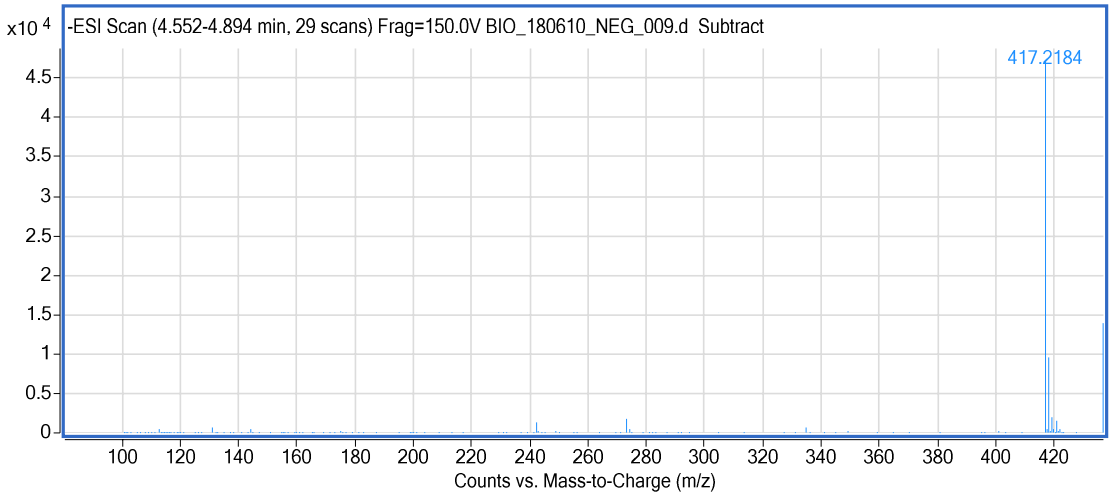
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C19H24NO5	345.1582	-2.5	

S09-012079 negative mode ESI, peak at 3.9 minutes



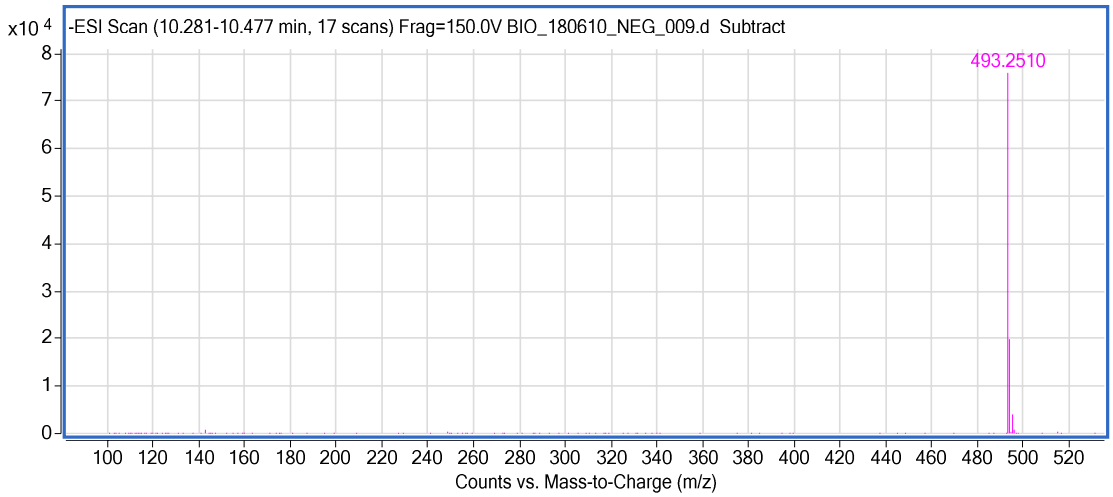
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C17H24NO2	274.1807	0.44	

S09-012079 negative mode ESI, peak at 4.7 minutes



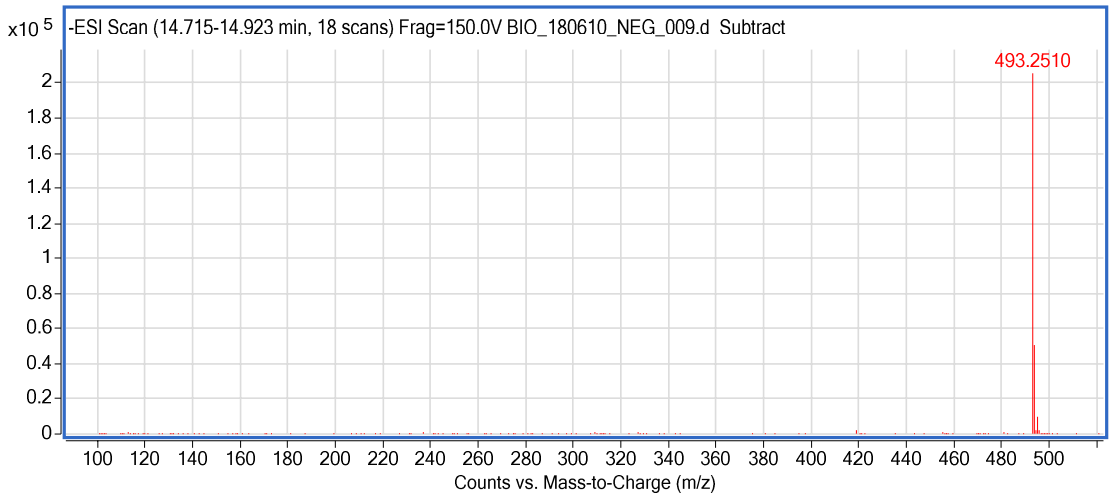
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C12H32N7O9	418.2262	1.03	
2	C18H34N4O5S	418.2250	-1.7	
3	C20H35NO6S	418.2262	1.5	

S09-012079 negative mode ESI, peak at 10.3 minutes



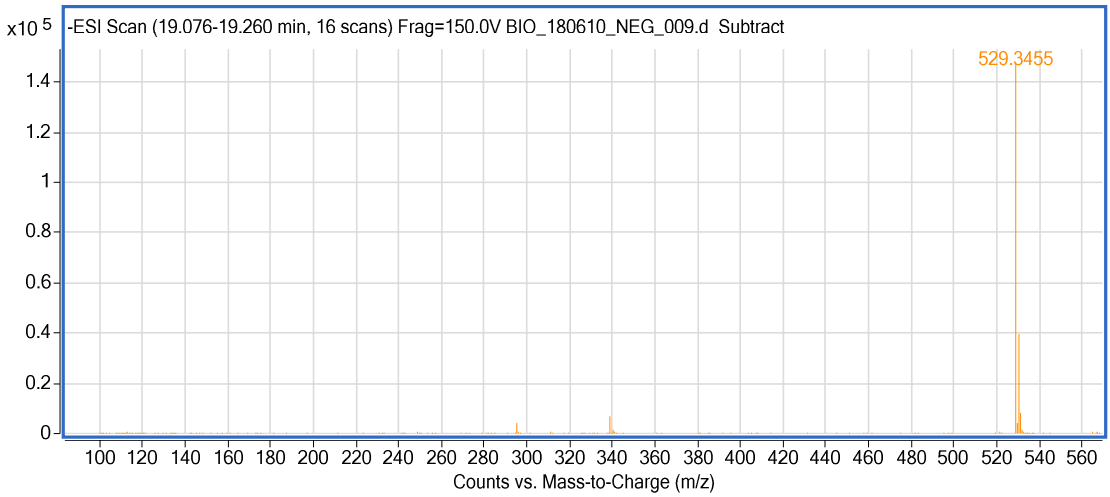
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H38N4O10	494.2588	1.1	
2	C18H36N7O9	494.2575	-1.7	
3	C22H40NO11	494.2601	3.8	

S09-012079 negative mode ESI, peak at 14.8 minutes



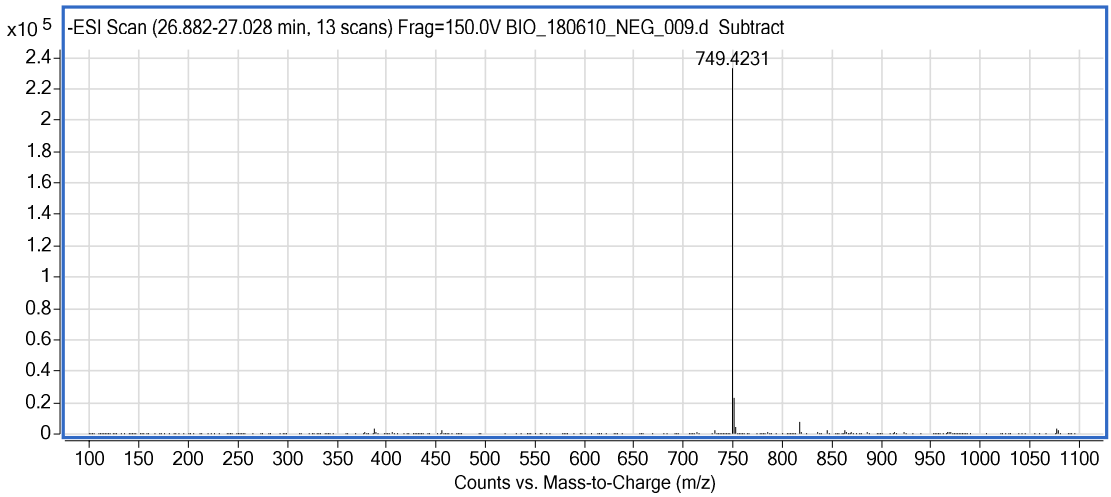
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H38N4O10	494.2588	0.98	
2	C18H36N7O9	494.2575	-1.8	
3	C22H40NO11	494.2601	3.7	

S09-012079 negative mode ESI, peak at 19.1 minutes



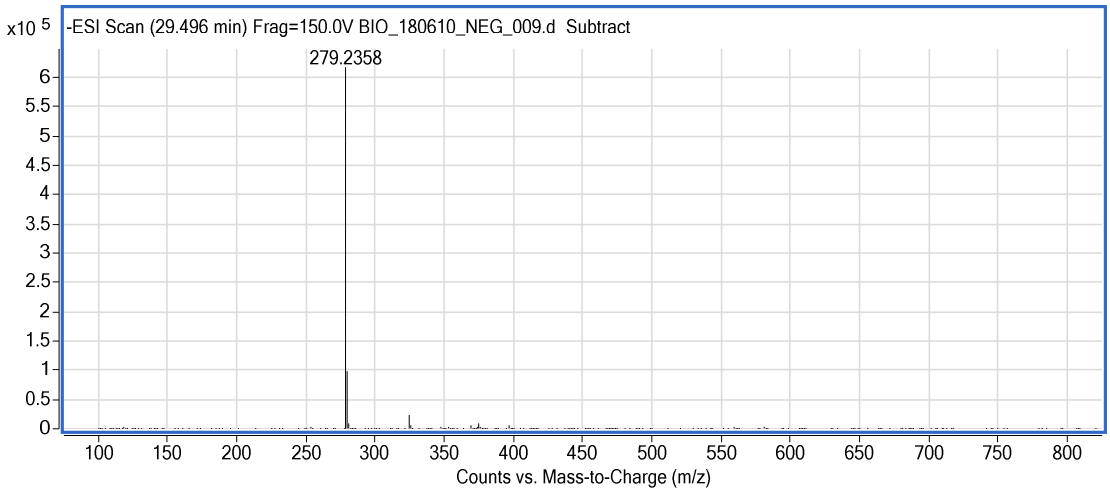
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H50N4O10	530.3527	-0.19	
2	C23H46N8O6	530.3540	2.3	
3	C20H48N7O9	530.3514	-2.7	

S09-012079 negative mode ESI, peak at 26.9 minutes



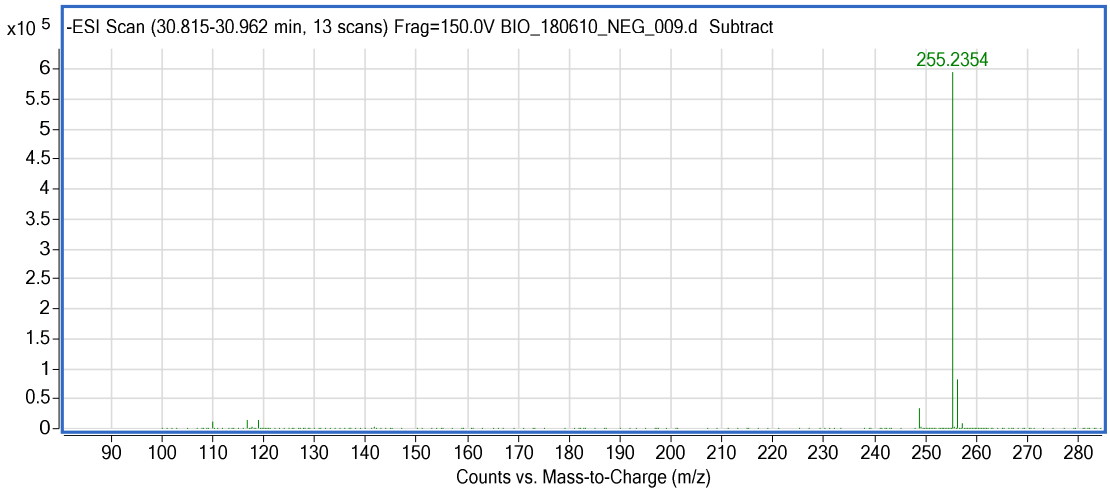
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C39H62N2O12	750.4230	-0.16	
2	C38H56N9O7	750.4230	-0.20	
3	C37H60N5O11	750.4217	-2.0	

S09-012079 negative mode ESI, peak at 29.6 minutes



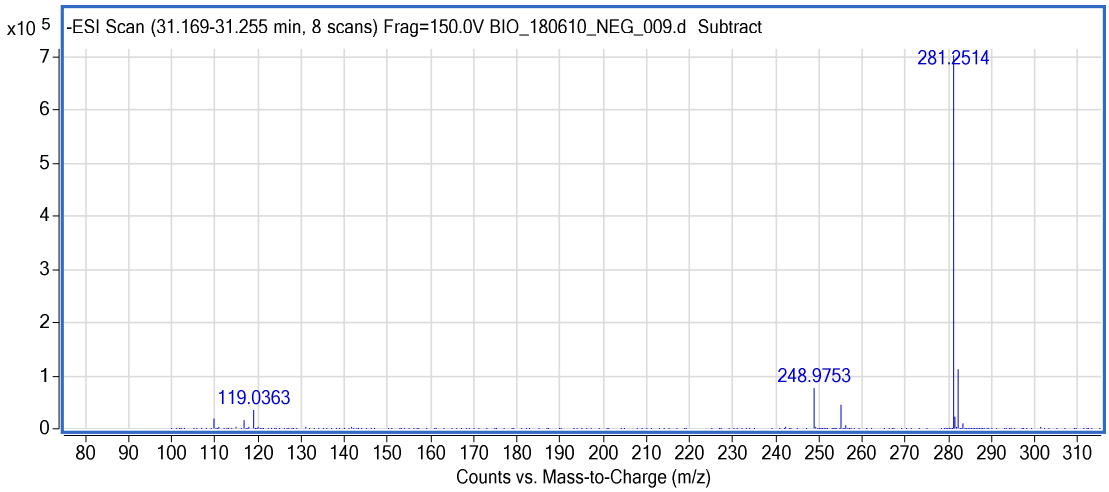
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H32O2	280.2402	-10.0	

S09-012079 negative mode ESI, peak at 30.9 minutes



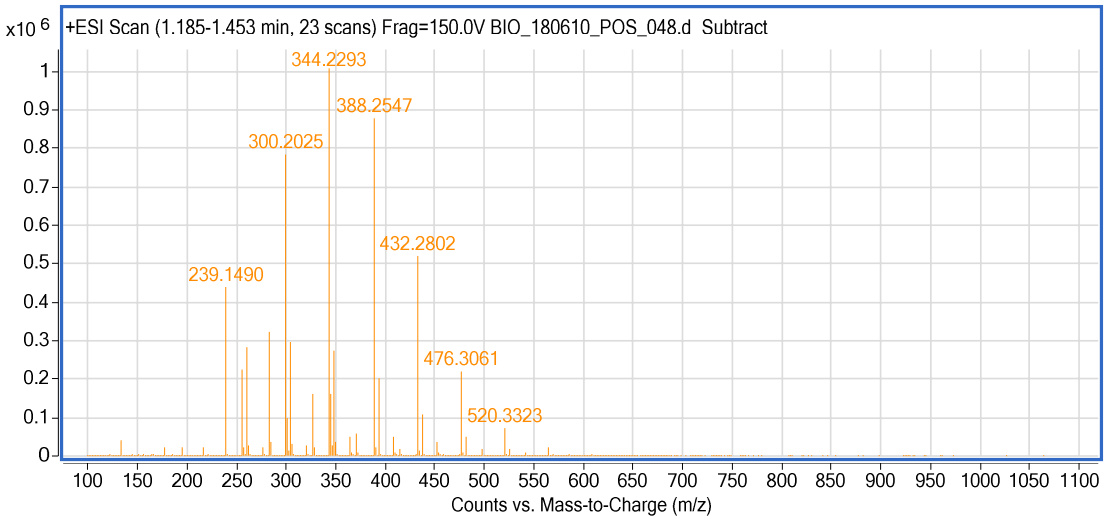
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2402	-9.4	

S09-012079 negative mode ESI, peak at 31.2 minutes



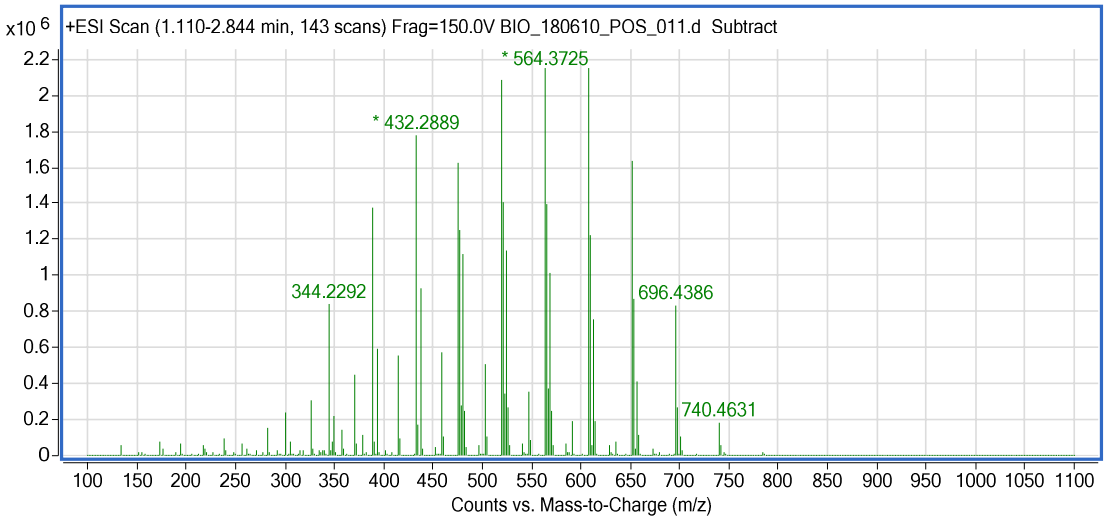
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O2	282.2559	-10.0	

S09-012080 positive mode ESI, peak at 1.2 minutes



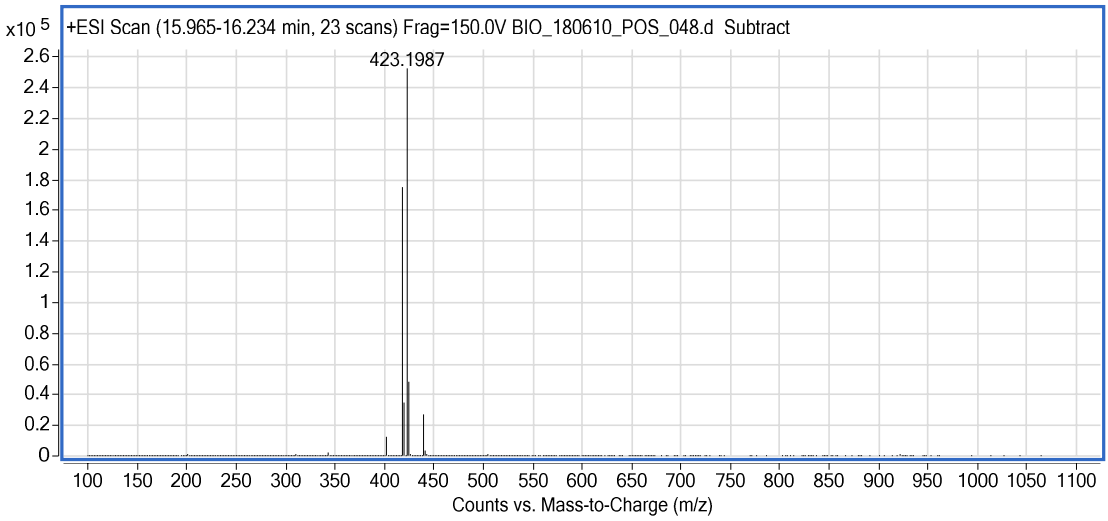
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	Oligomer series			

S09-012080 positive mode ESI, peak at 1.2-6.0 minutes



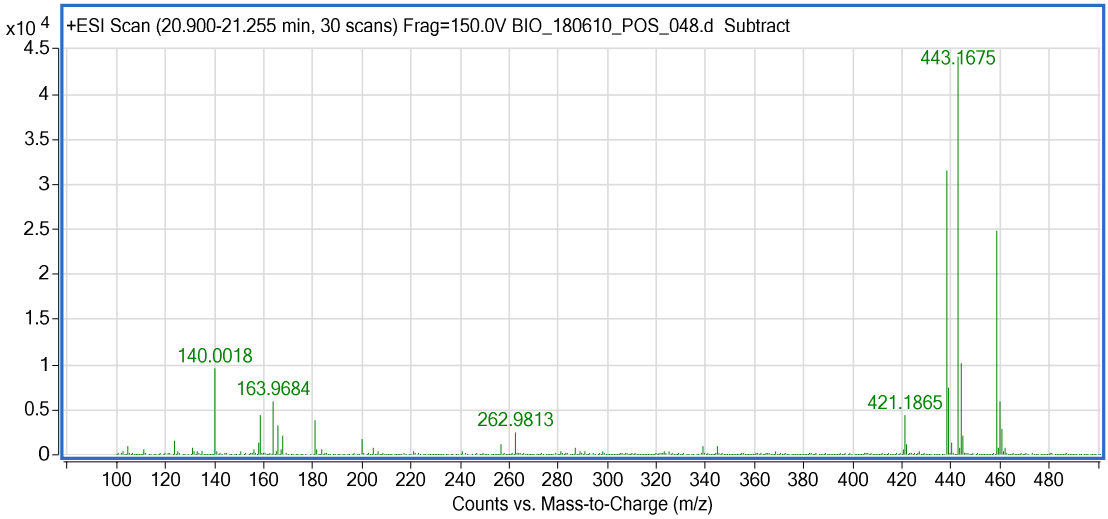
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	Oligomer series			

S09-012080 positive mode ESI, peak at 16.1 minutes



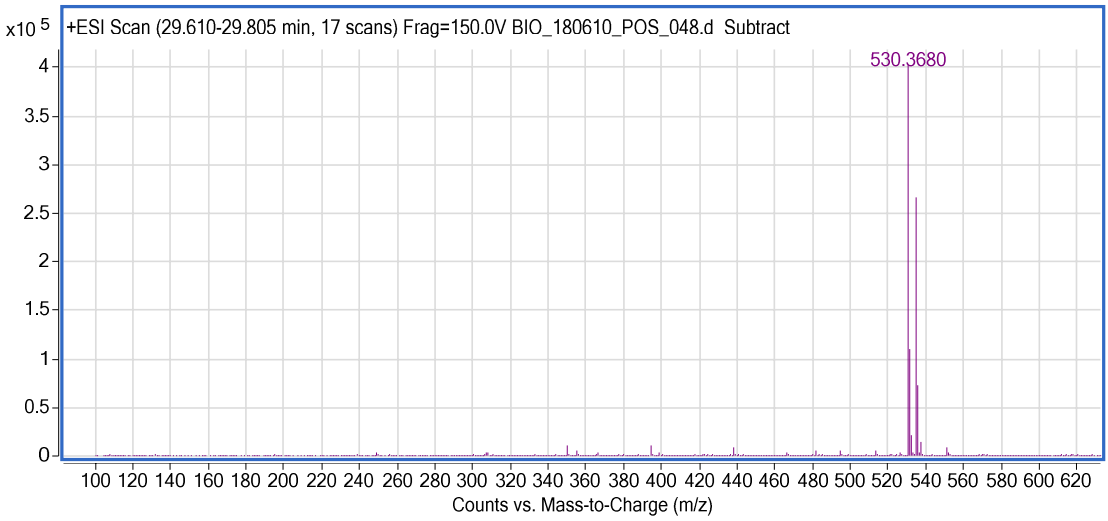
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H35NO8P	400.2100	1.6	
2	C16H40N2OP4	400.2091	-0.72	
3	C20H32O8	400.2097	0.80	

S09-012080 positive mode ESI, peak at 21.0 minutes



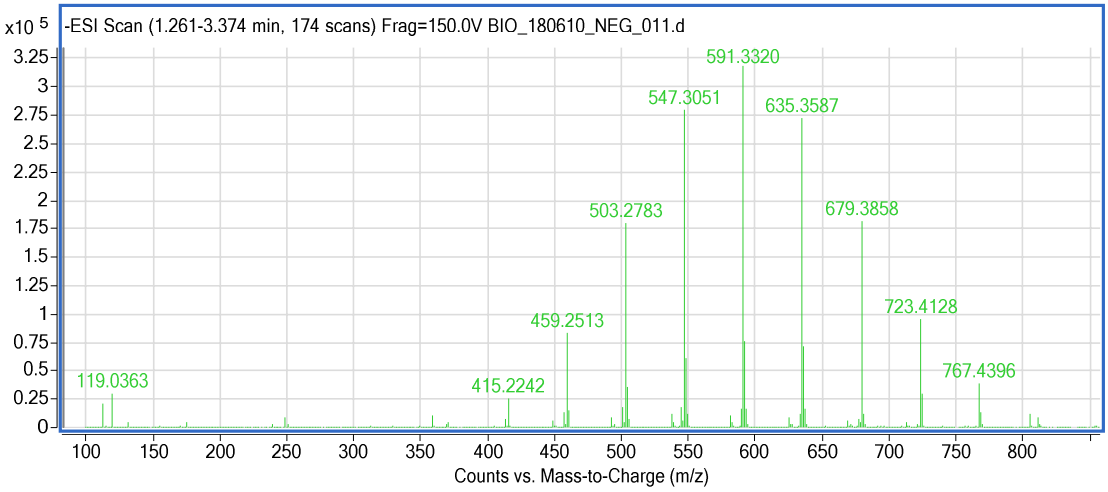
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H28O8	420.1784	1.12	
2	C20H26N3O7	420.1771	-2.08	
3	C22H33NOP3	420.1775	-1.07	

S09-012080 positive mode ESI, peak at 29.7 minutes



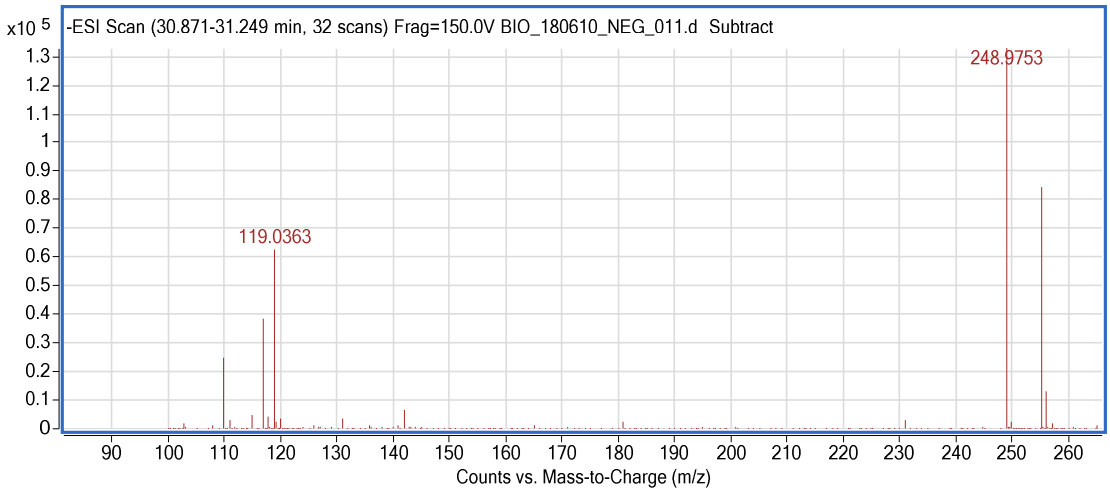
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H49N4O7P	512.3339	-0.65	
2	C24H51NO8P	512.3352	2.0	
3	C28H48O8	512.3349	1.4	

S09-012080 negative mode ESI, peak at 1.0 – 3.2 minutes



	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H48N8O12	592.3392	-0.12	
2	C22H50N5O13	592.3405	2.2	

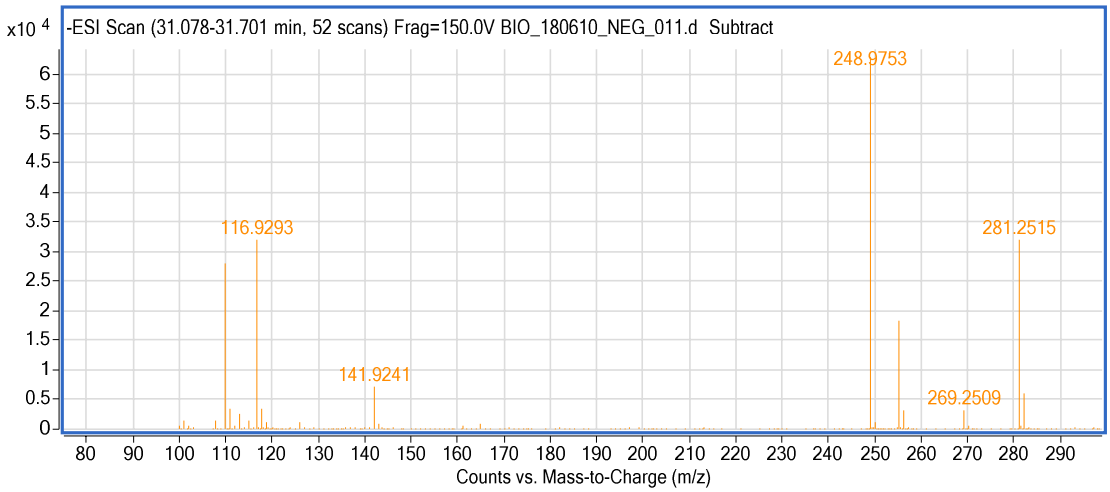
S09-012080 negative mode ESI, peak at 30.9 minutes



NOTE: 248.9753 co-eluting interference

	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2426	-9.6	

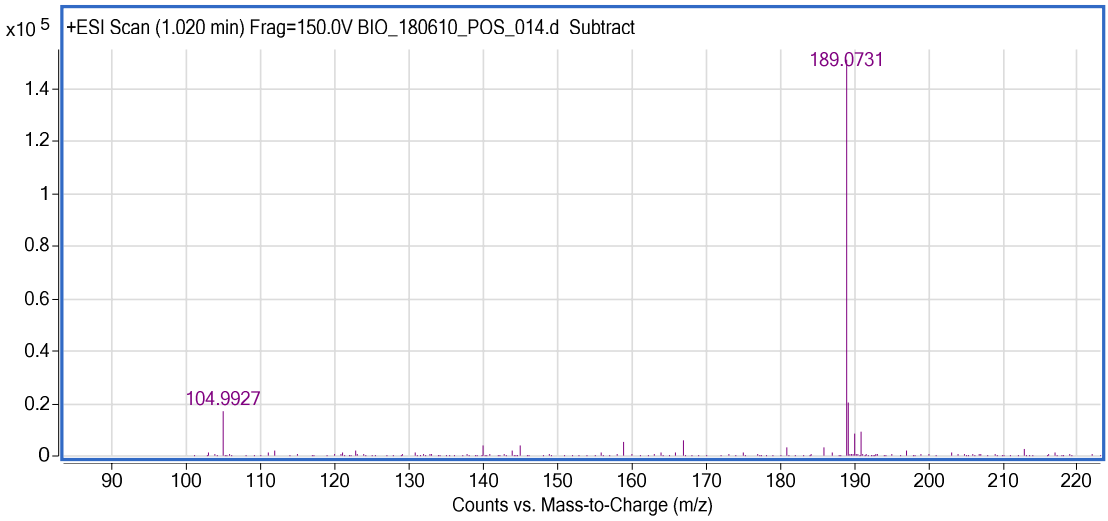
S09-012080 negative mode ESI, peak at 31.3 minutes



NOTE: 248.9753 co-eluting interference

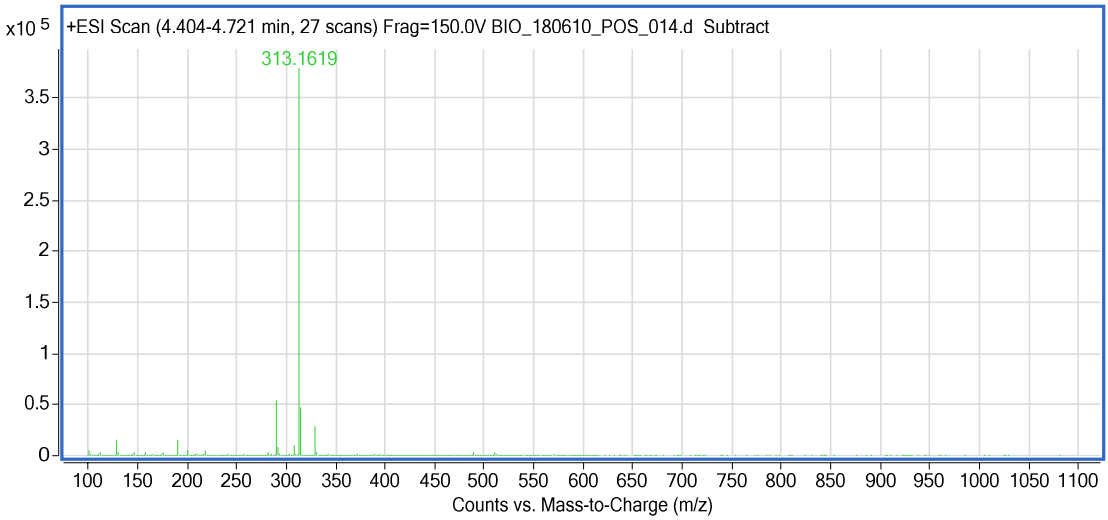
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O2	282.2588	-10.0	

S09-012081 positive mode ESI, peak at 1.1 minutes



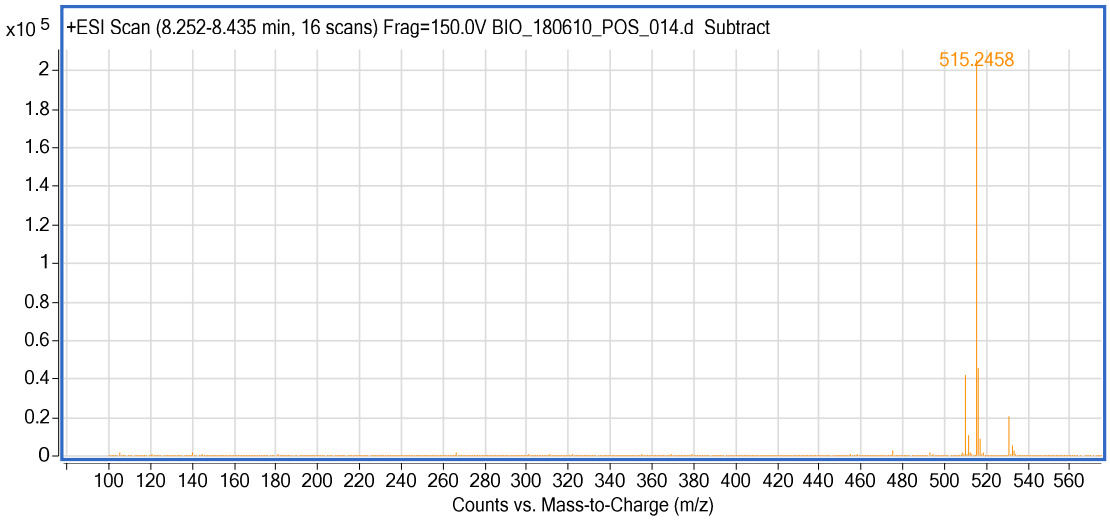
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C6H14O5	166.0841	1.3	

S09-012081 positive mode ESI, peak at 4.6 minutes



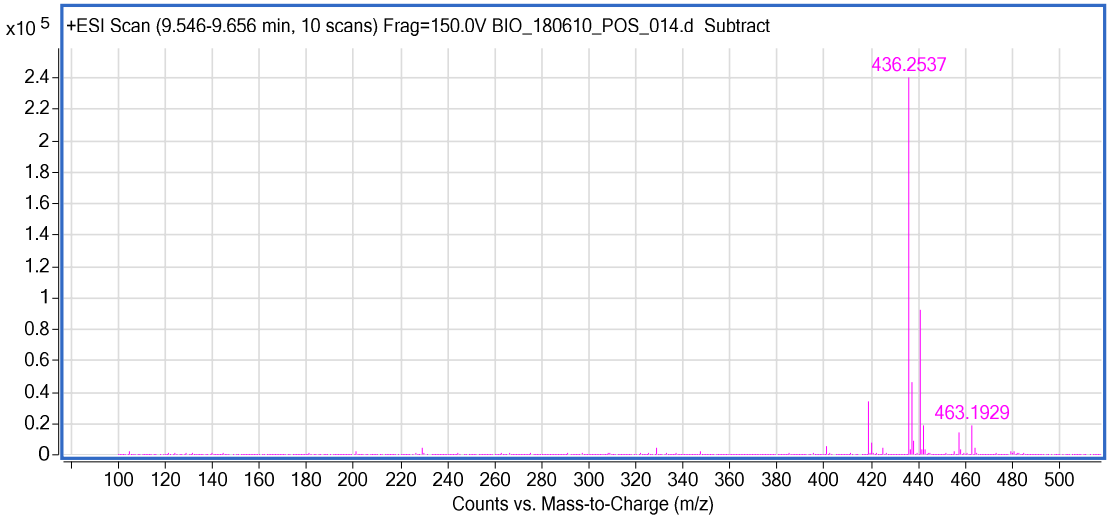
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C14H26O6	290.1732	0.82	

S09-012081 positive mode ESI, peak at 8.3 minutes



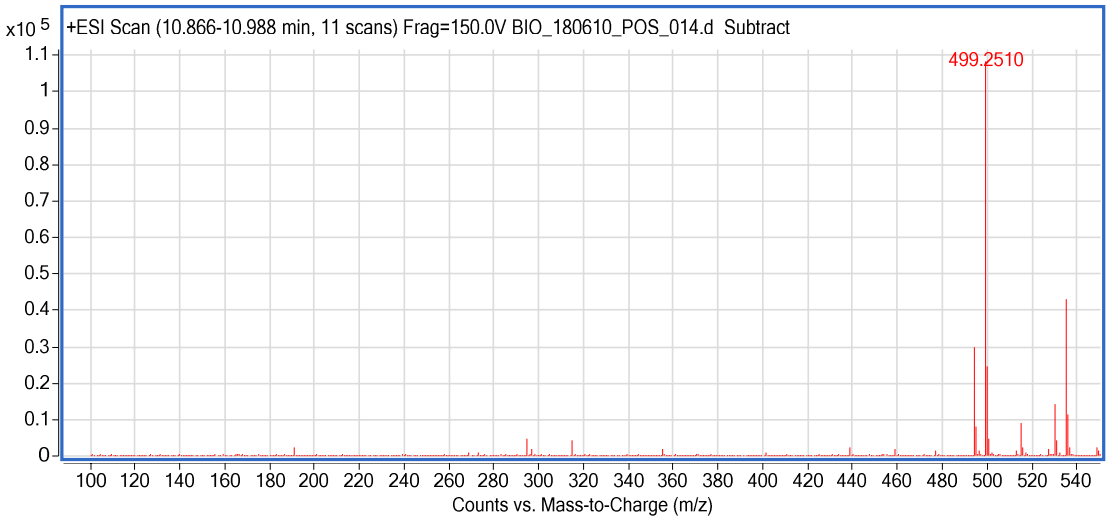
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C17H41N4O10P	492.2560	-1.1	
2	C21H38N3O10	492.2557	-1.7	
3	C23H40O11	492.2571	1.0	

S09-012081 positive mode ESI, peak at 9.6 minutes



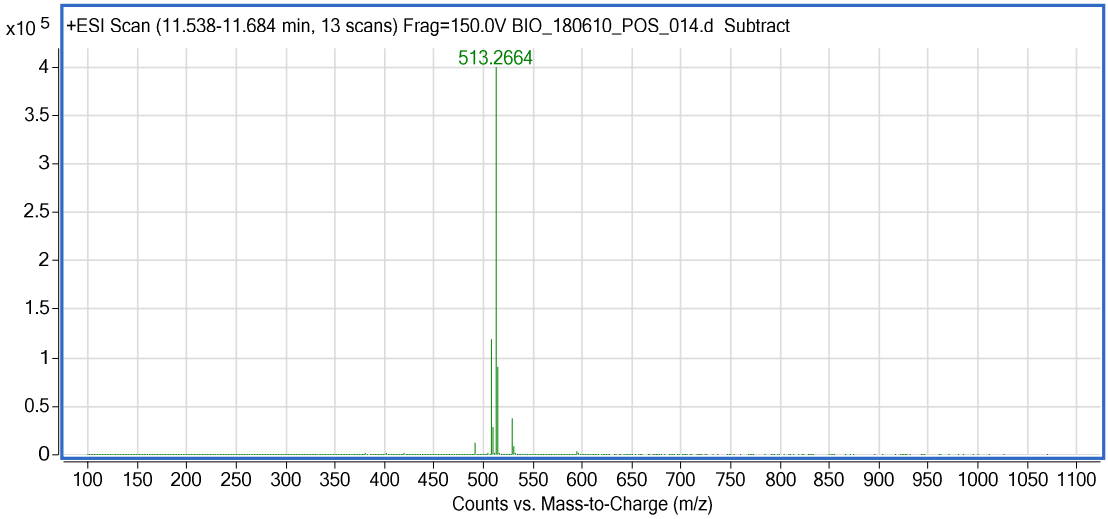
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H34O9	418.2203	0.22	
2	C15H31N8O4P	418.2206	0.93	
3	C16H37NO9P	418.2206	0.96	

S09-012081 positive mode ESI, peak at 10.9 minutes



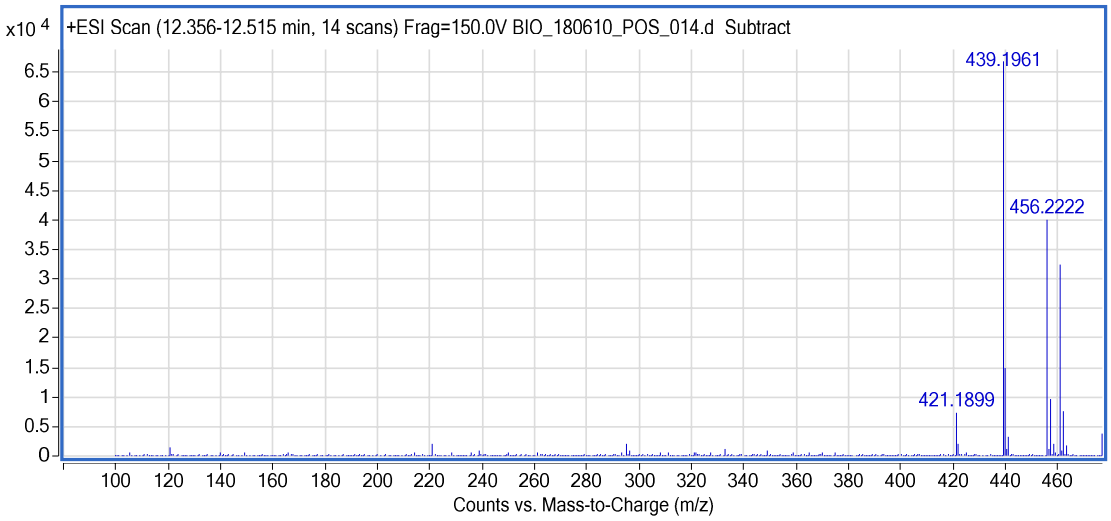
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C17H41N4O9P	476.2611	-1.3	
2	C18H37N8O5P	476.2625	1.5	
3	C23H40O10	476.2621	0.84	

S09-012081 positive mode ESI, peak at 11.6 minutes



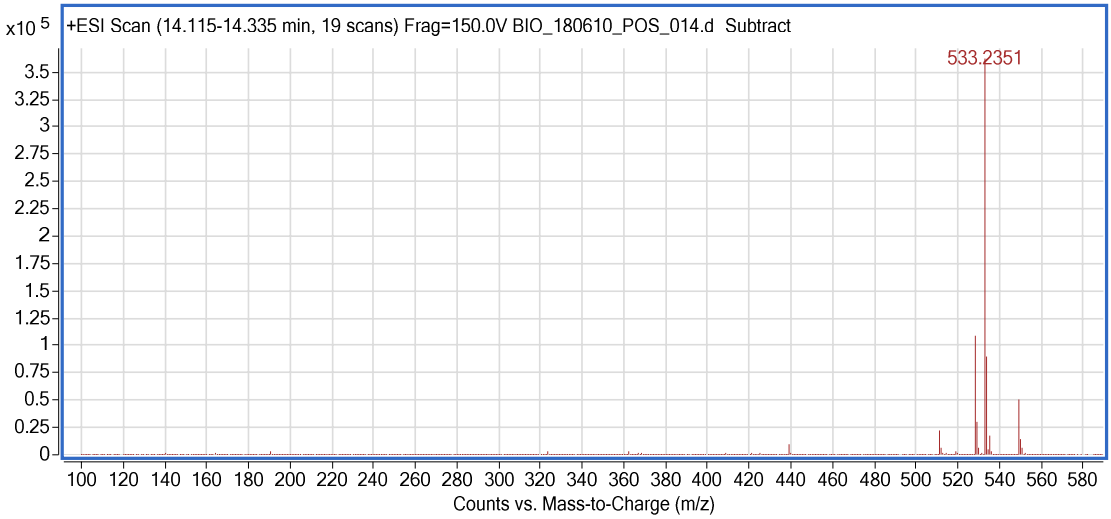
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C24H42O10	490.2778	1.2	
2	C18H43N4O9P	490.2768	-0.89	
3	C20H45NO10P	490.2781	1.9	

S09-012081 positive mode ESI, peak at 12.4 minutes



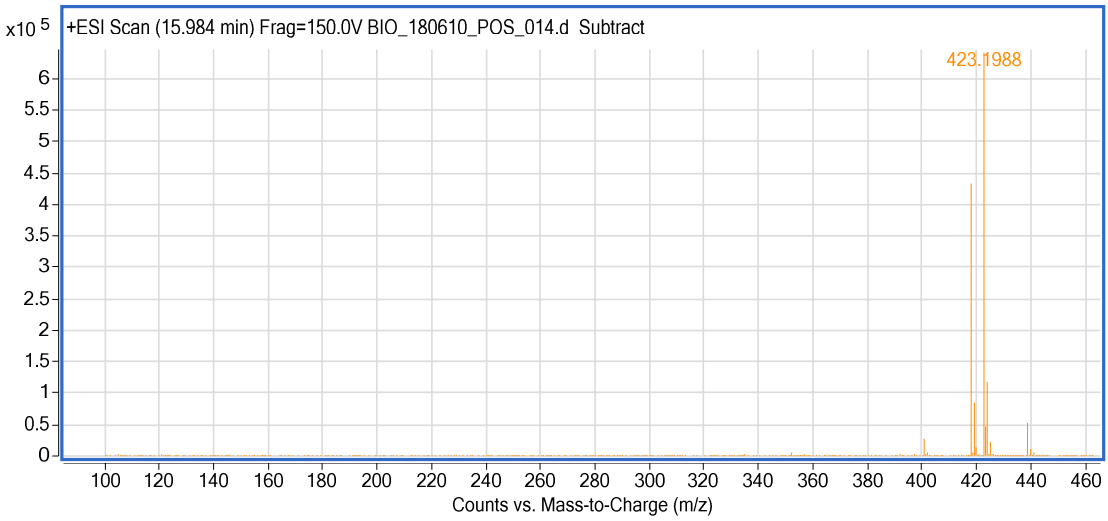
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H30O9	438.1890	0.85	
2	C22H35NO2P3	438.1861	-1.3	
3	C21H24N7O4	438.1890	0.82	

S09-012081 positive mode ESI, peak at 14.2 minutes



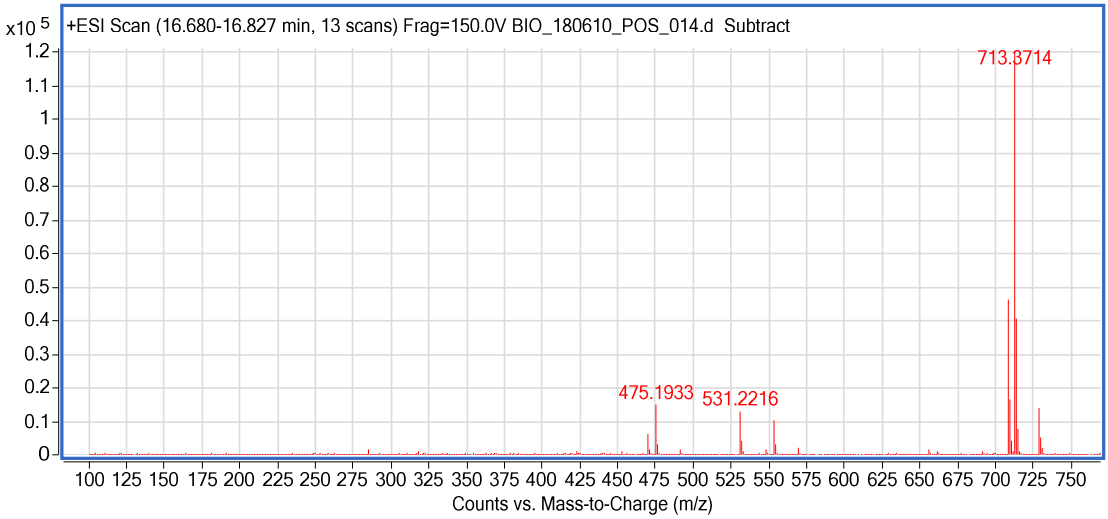
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H46N2O3P4	510.2459	0.11	
2	C20H39N4O9P	510.2455	-0.73	
3	C26H38O10	510.2465	1.3	

S09-012081 positive mode ESI, peak at 16.1 minutes



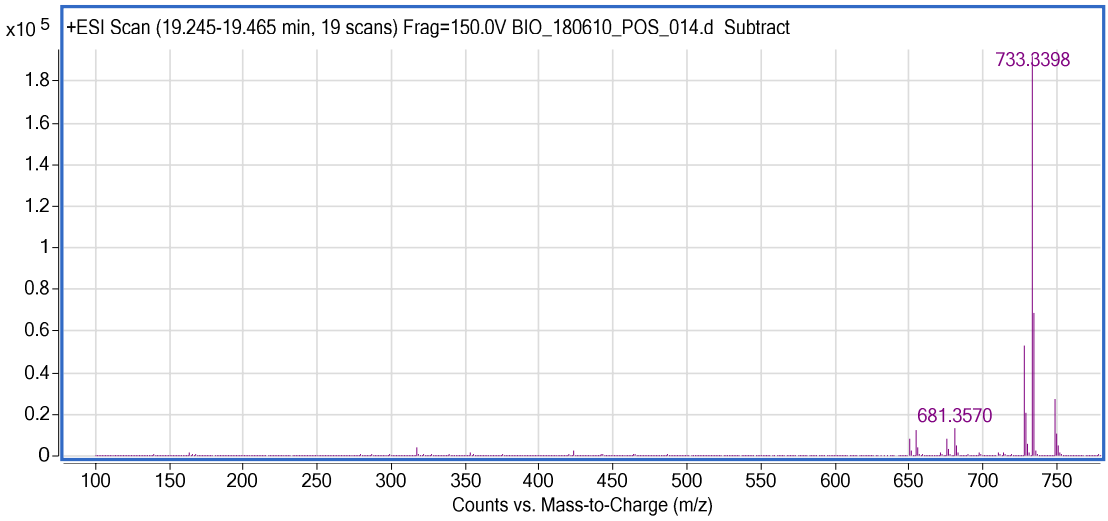
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H32O8	400.2097	0.97	
2	C16H35NO8P	400.2100	1.0	
3	C15H29N8O3P	400.2091	-1.3	

S09-012081 positive mode ESI, peak at 16.7 minutes



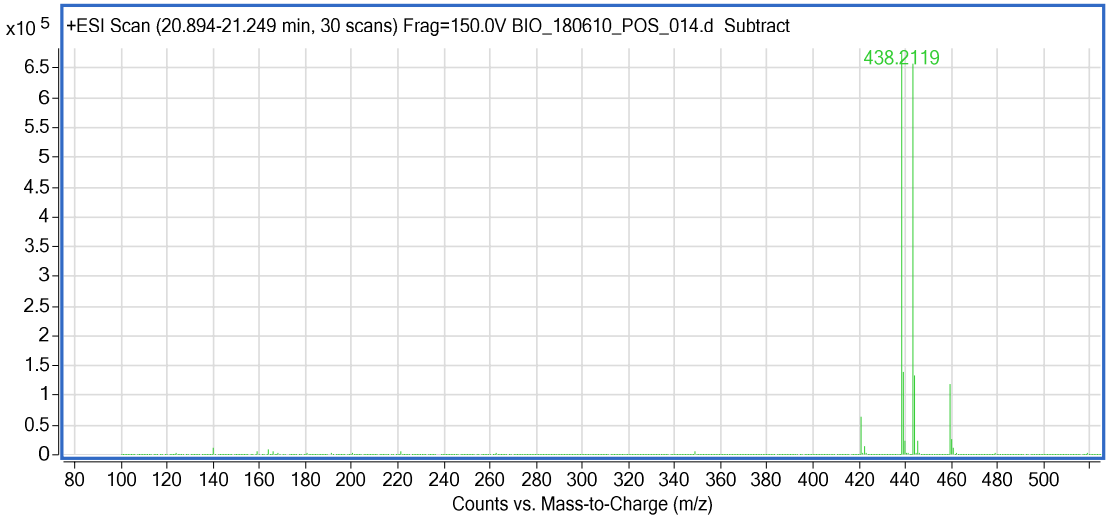
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C29H60N9O2P4	690.3820	-0.22	
2	C30H66N2O7P4	690.3820	-0.19	
3	C34H58O14	690.3827	0.67	

S09-012081 positive mode ESI, peak at 19.3 minutes



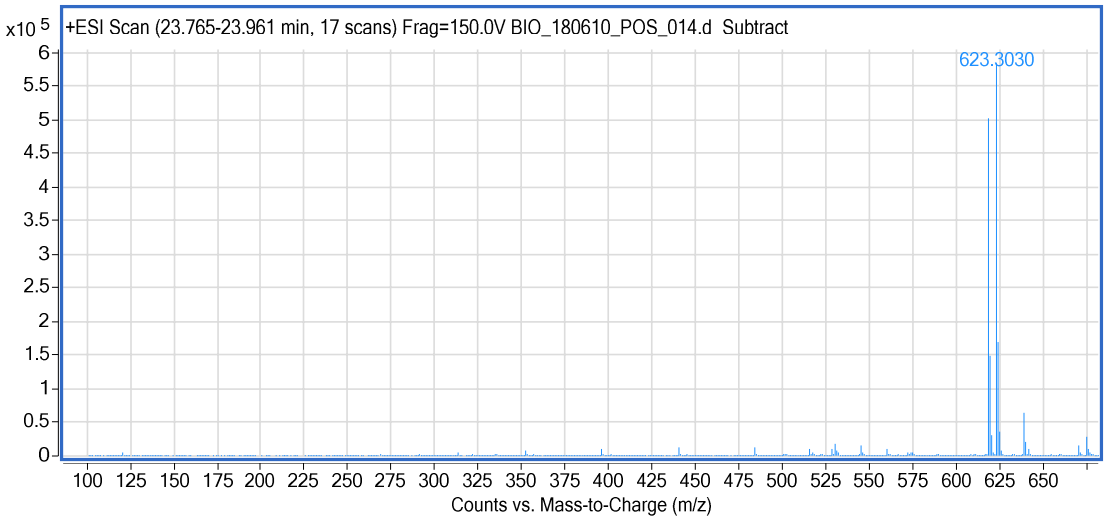
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C30H55N4O13P	710.3503	-0.36	
2	C36H54O14	710.3514	1.1	
3	C32H57NO14P	710.3517	1.6	

S09-012081 positive mode ESI, peak at 21.0 minutes



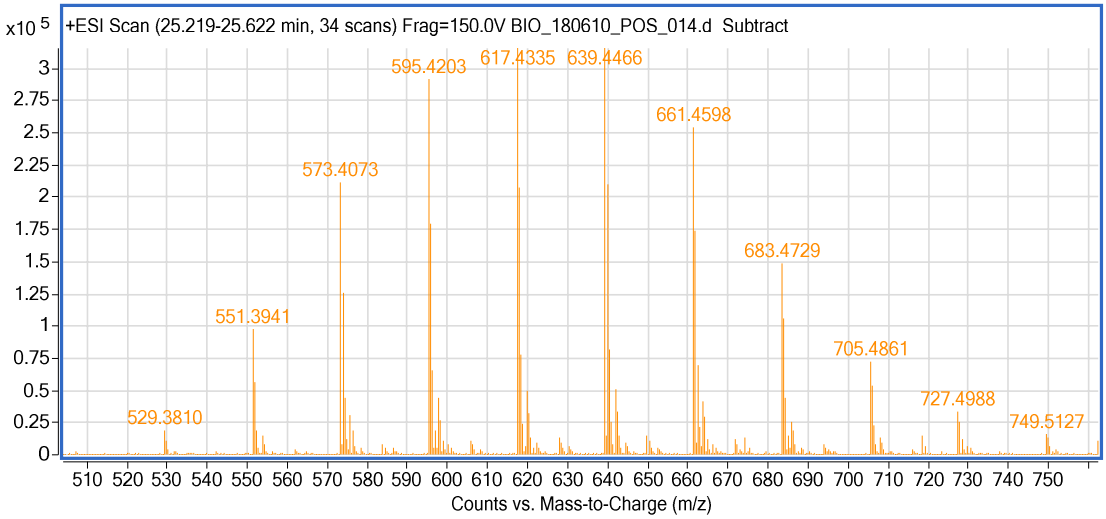
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H31NO8P	420.1787	1.6	
2	C16H29N4O7P	420.1774	-1.6	
3	C22H28O8	420.1784	0.86	

S09-012081 positive mode ESI, peak at 23.9 minutes



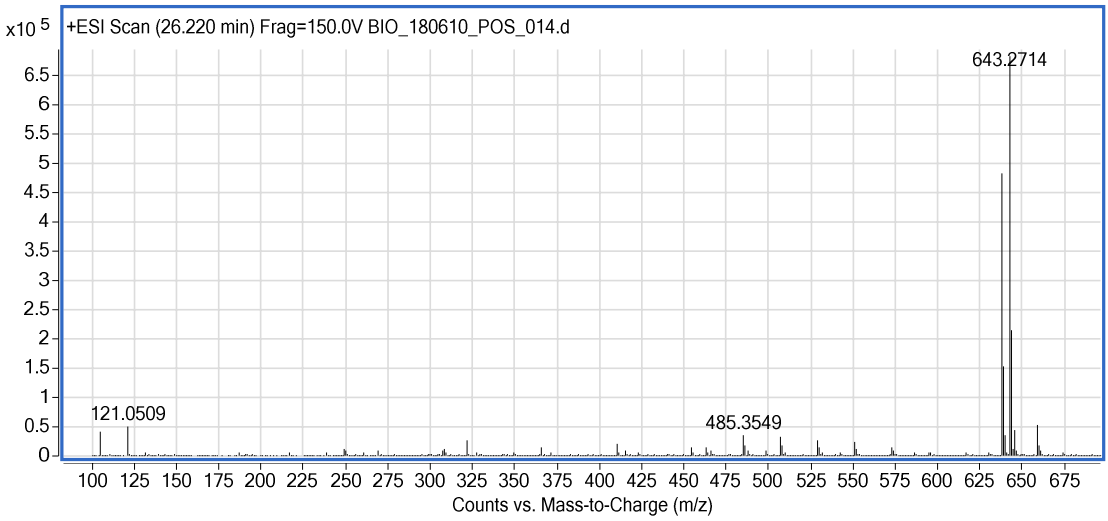
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C26H51NO12P	600.3149	1.8	
2	C28H46N3O11	600.3132	-0.94	
3	C30H48O12	600.3146	1.3	

S09-012081 positive mode ESI, peak at 25.4 minutes



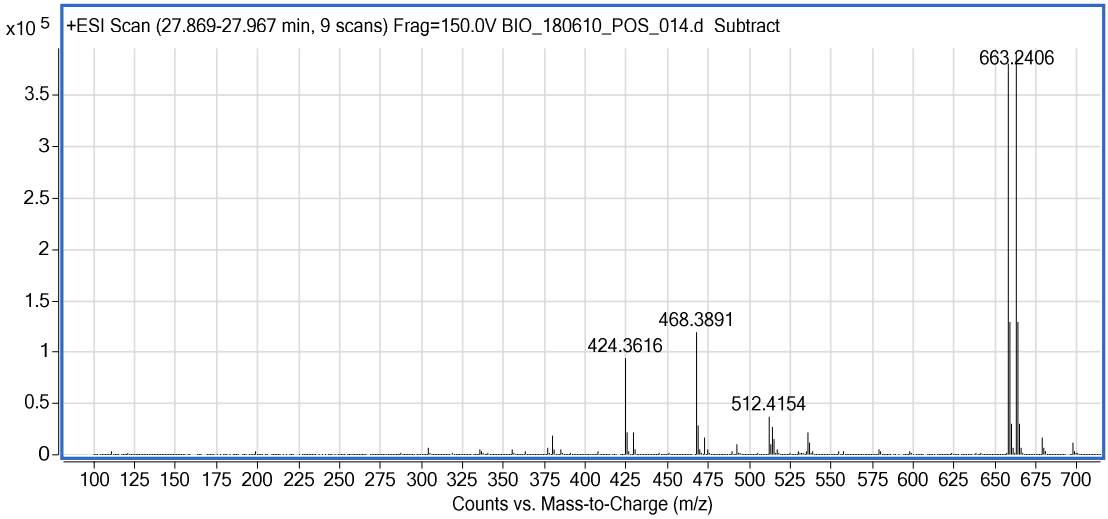
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	OLIGOMER SERIES			

S09-012081 positive mode ESI, peak at 26.1 minutes



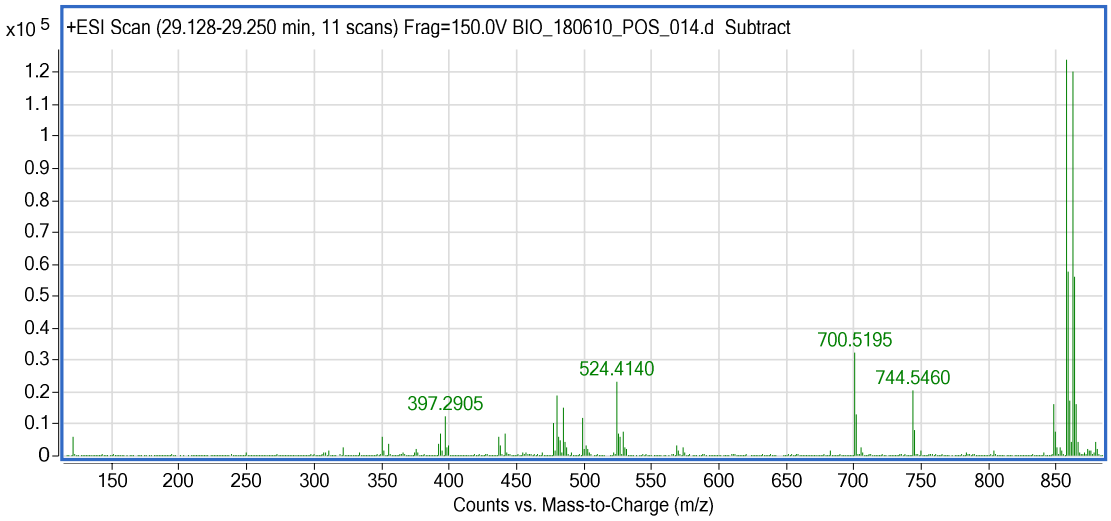
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C30H42N3O11	620.2819	-0.45	
2	C28H47NO12P	620.2836	2.2	
3	C32H44O12	620.2833	1.7	

S09-012081 positive mode ESI, peak at 28.0 minutes



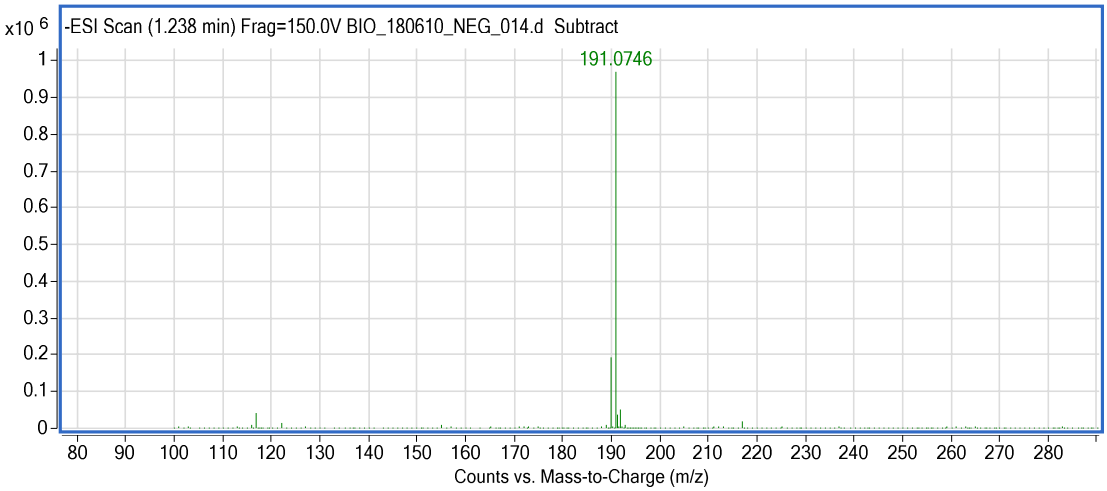
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C32H38N3O11	640.2506	-1.2	
2	C34H40O12	640.2520	0.90	

S09-012081 positive mode ESI, peak at 29.1 minutes



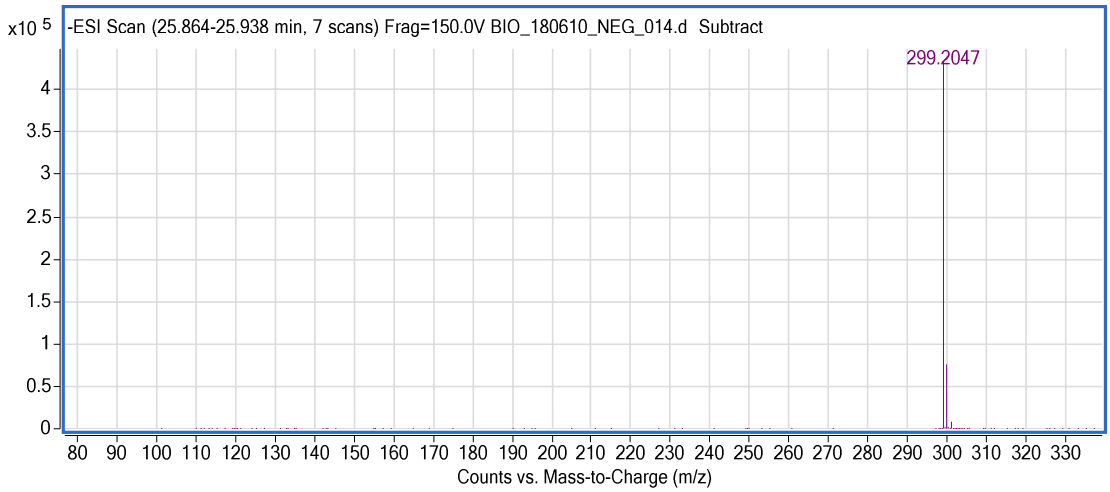
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C57H52N3O5	840.3563	0.18	
2	C44H56O16	840.3568	0.80	

S09-012081 negative mode ESI, peak at 1.1 minutes



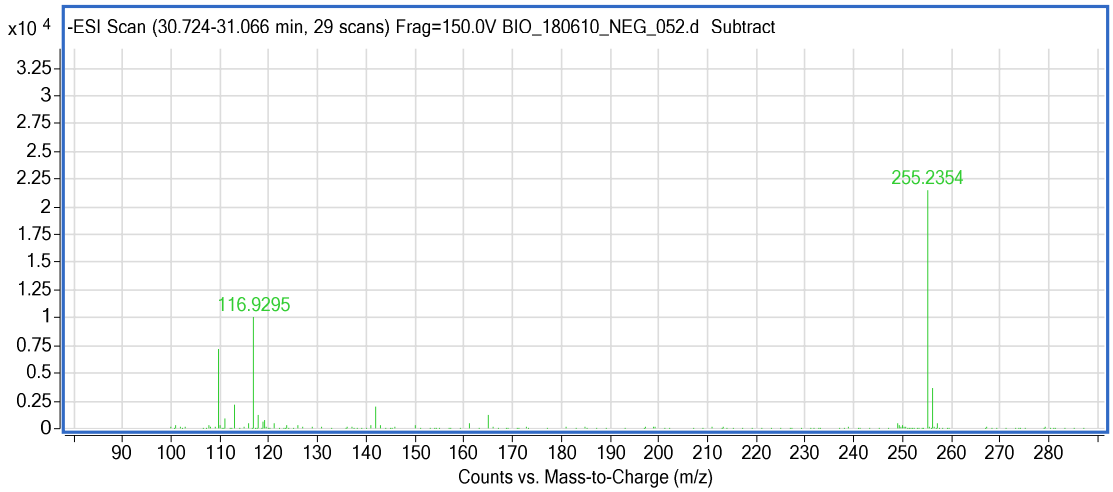
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C8H16O3S	192.0820	0.89	
2	C14H10N	192.0813	-2.7	

S09-012081 negative mode ESI, peak at 25.7 minutes



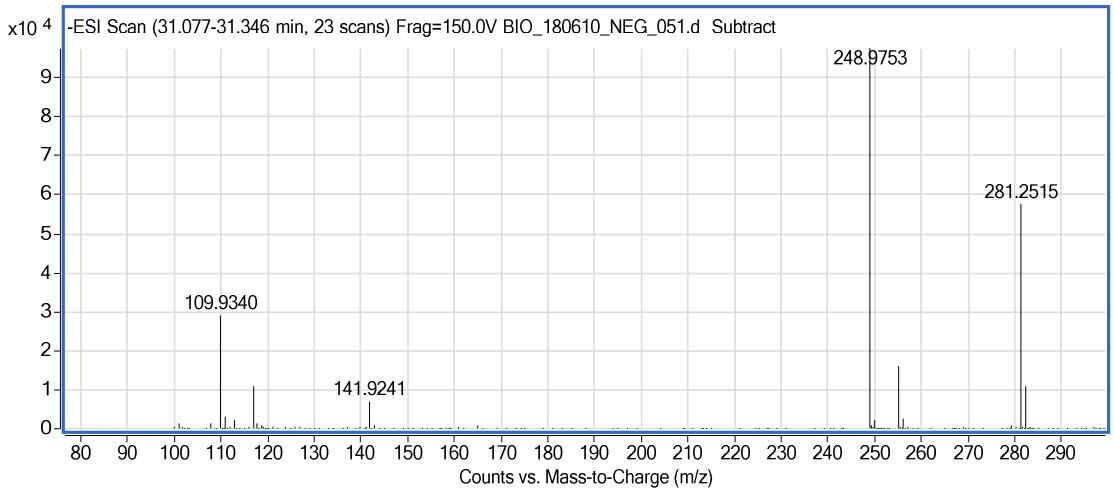
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C17H32O2S	300.2123	0.93	

S09-012081 negative mode ESI, peak at 30.8 minutes



	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2402	-9.5	

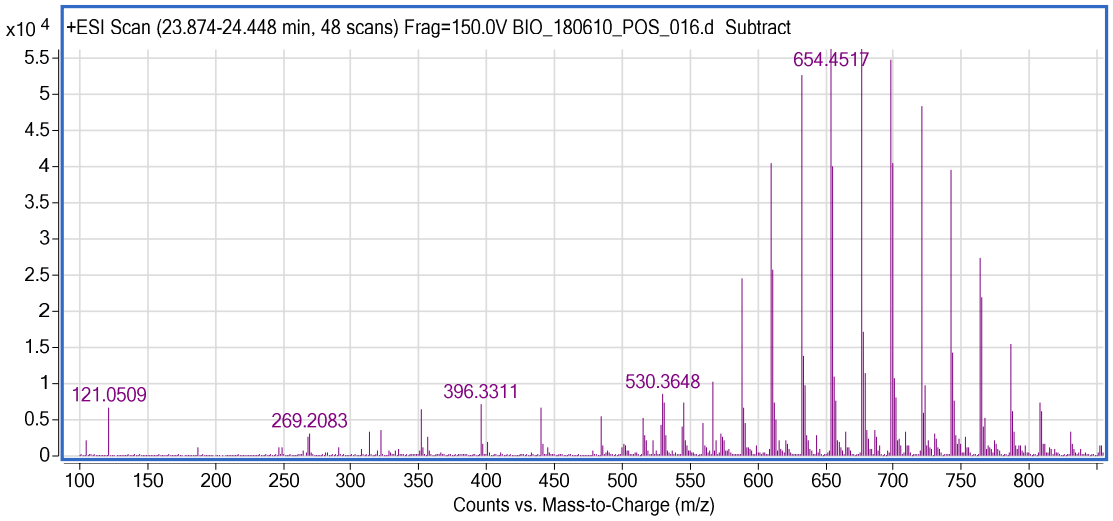
S09-012081 negative mode ESI, peak at 31.2 minutes



NOTE: 248.9753 co-eluting interference

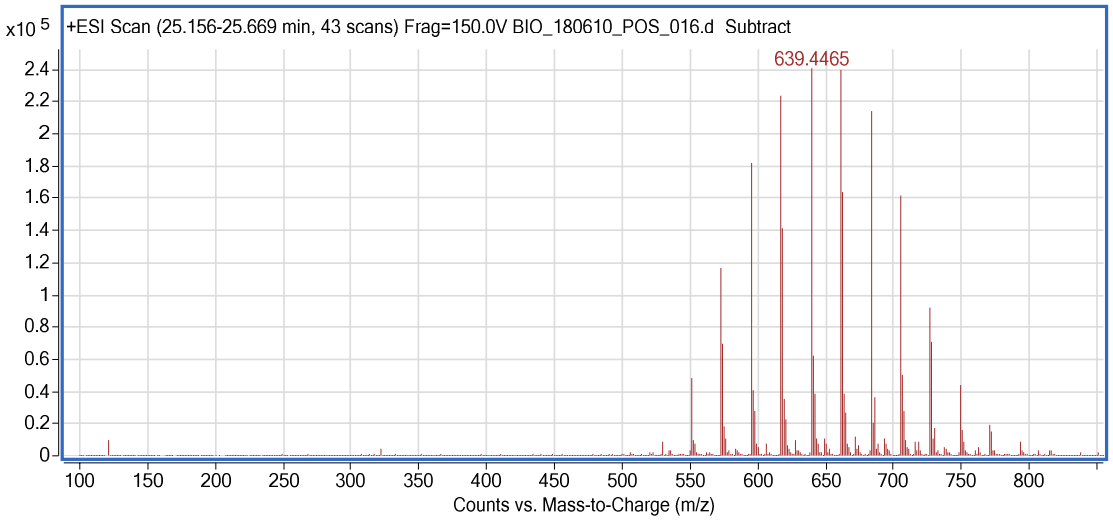
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O2	282.2588	-10.2	

S09-012082 positive mode ESI, peak at 24.1 minutes



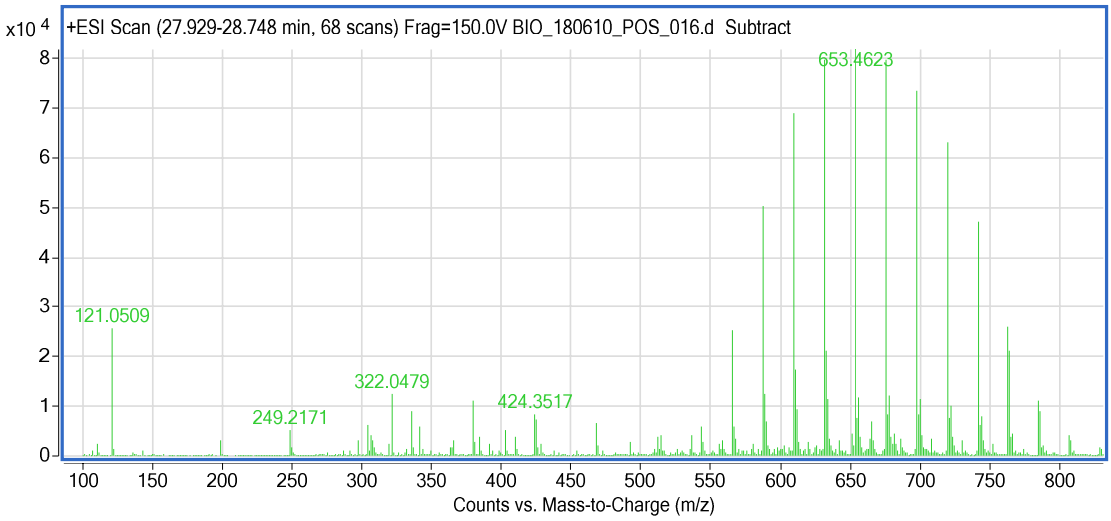
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	OLIGOMER SERIES			

S09-012082 positive mode ESI, peak at 25.4 minutes



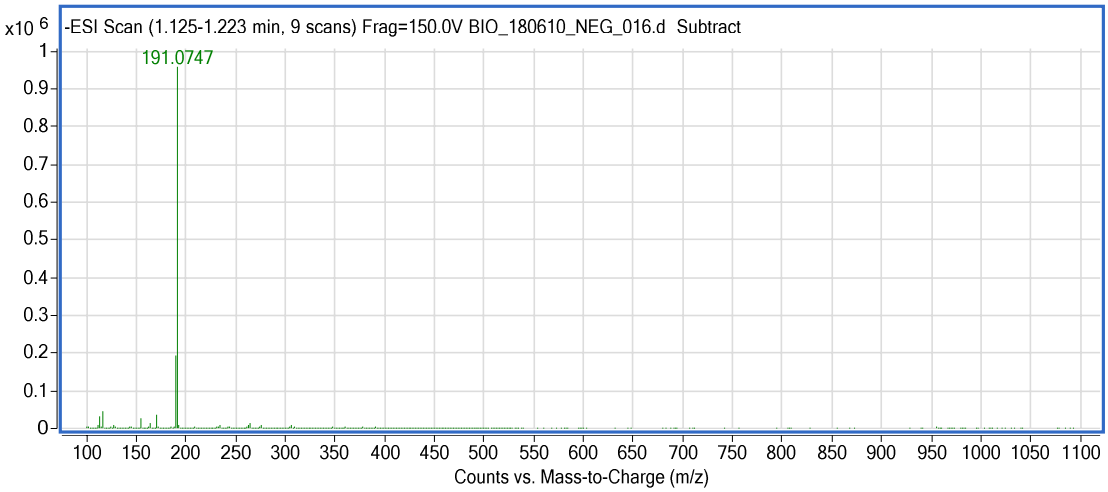
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	OLIGOMER SERIES			

S09-012082 positive mode ESI, peak at 28.3 minutes



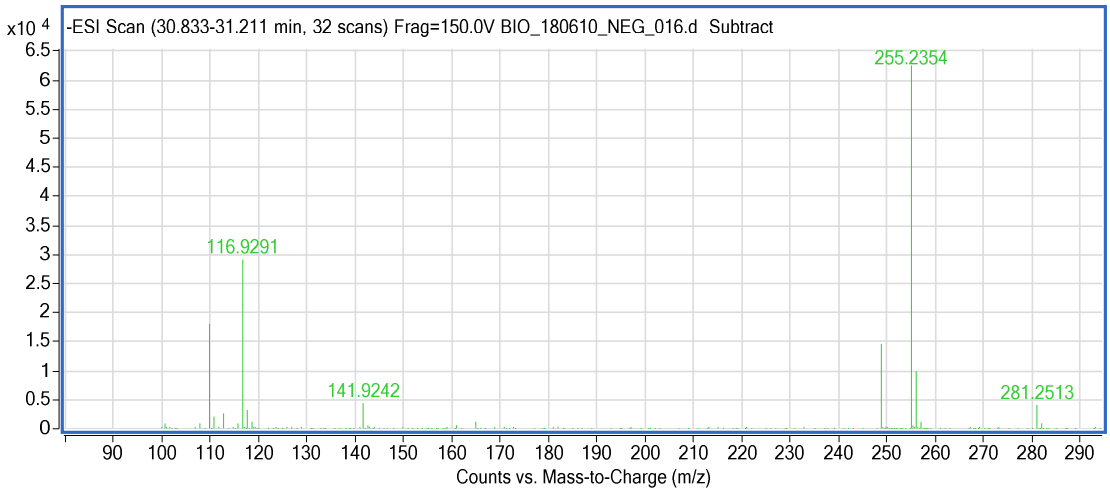
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	OLIGOMER SERIES			

S09-012082 negative mode ESI, peak at 1.1 minutes



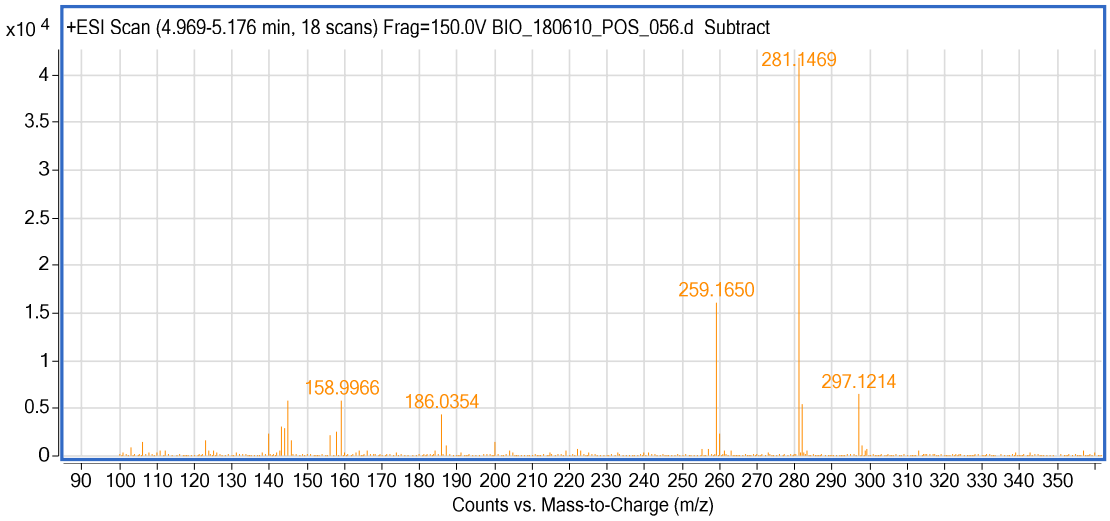
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C8H16O3S	192.0820	0.34	
2	C6H14N3O2S	192.0807	-6.7	
3	C14H10N	192.0813	-3.3	

S09-012082 negative mode ESI, peak at 30.6 minutes



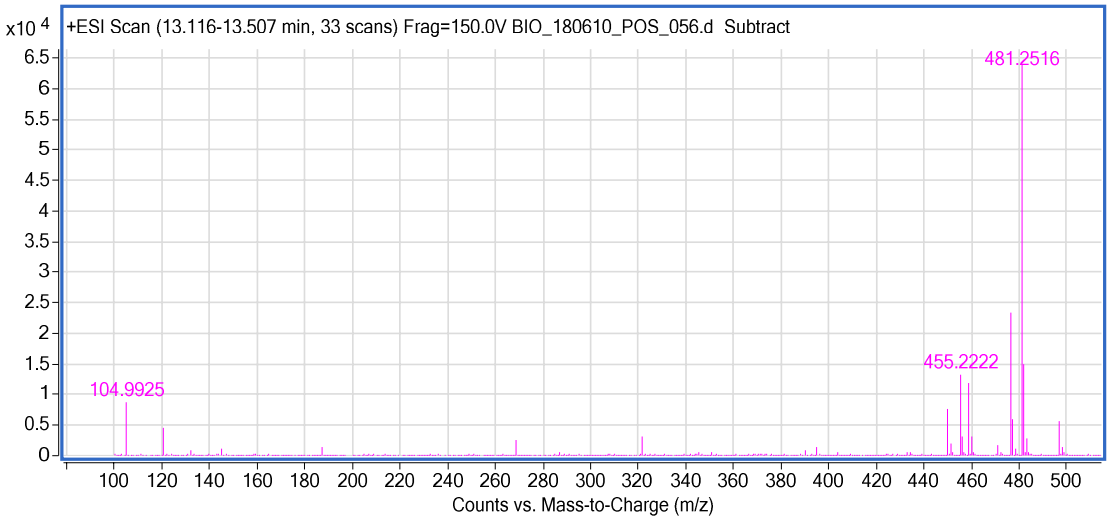
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	352.2325	-9.5	

S09-012083 positive mode ESI, peak at 5.1 minutes



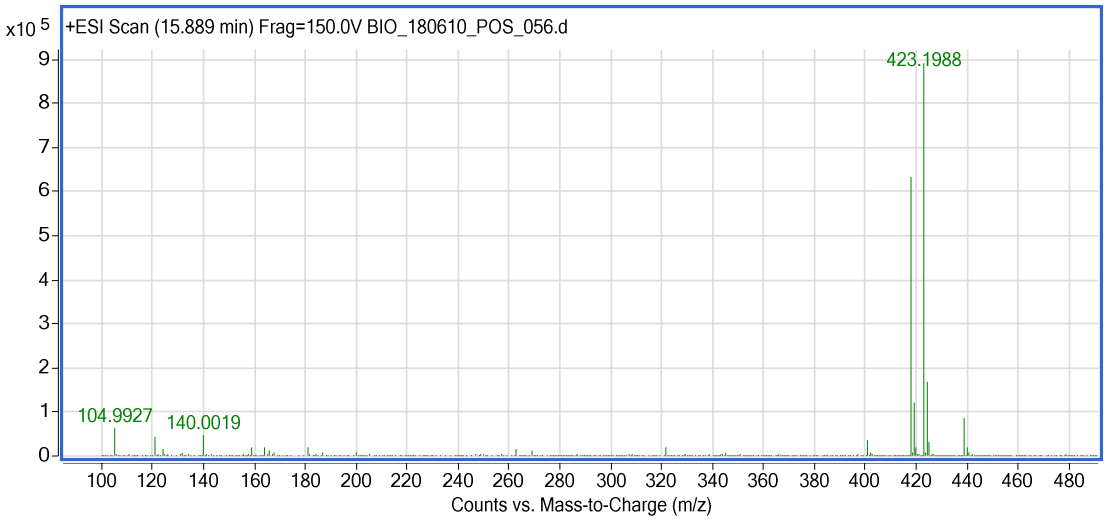
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C12H22N2O4	258.1580	0.94	

S09-012083 positive mode ESI, peak at 13.2 minutes



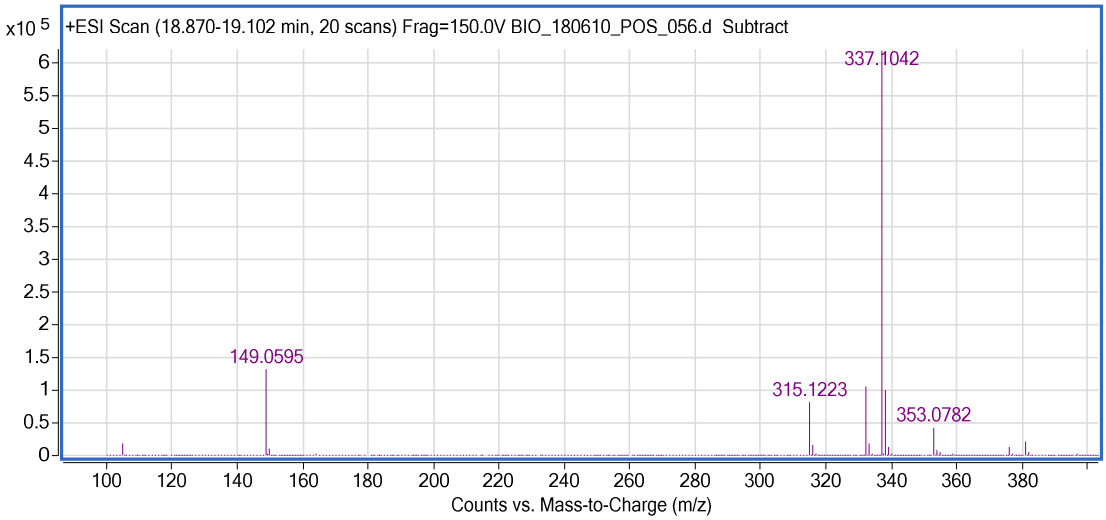
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H38N2O8	458.2628	1.0	
2	C20H36N5O7	458.2615	-1.9	

S09-012083 positive mode ESI, peak at 16.1 minutes



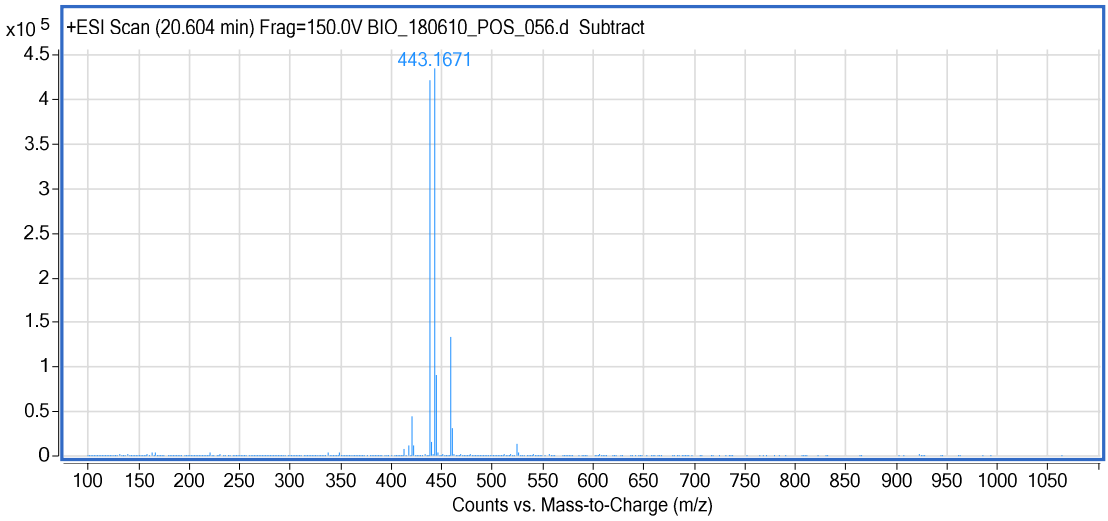
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H35NO8P	400.2100	1.1	
2	C20H32O8	400.2097	0.30	

S09-012083 positive mode ESI, peak at 19.0 minutes



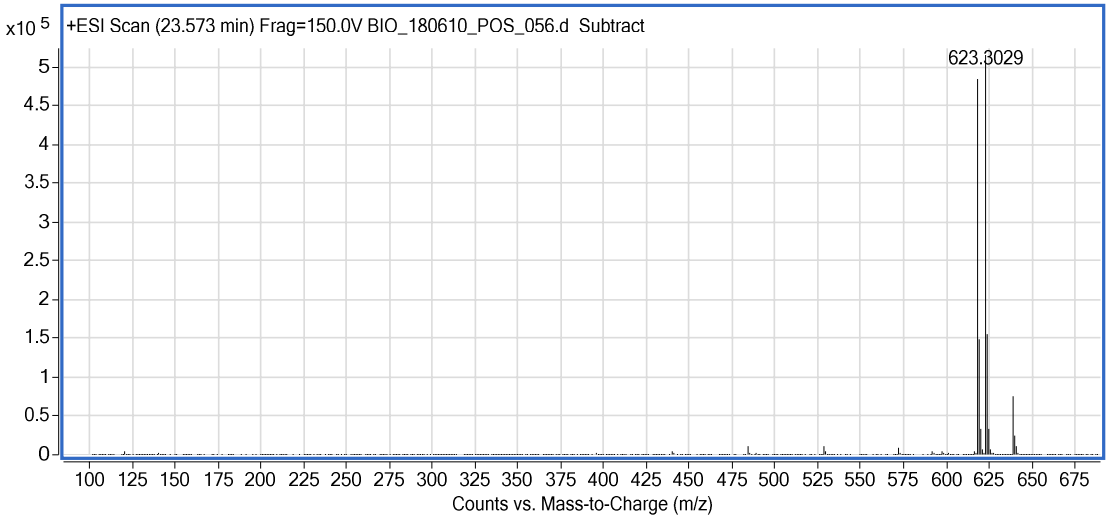
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C14H21NO5P	314.1157	2.4	
2	C12H19N4O4P	314.1144	-1.9	
3	C18H18O5	314.1154	1.4	

S09-012083 positive mode ESI, peak at 20.9 minutes



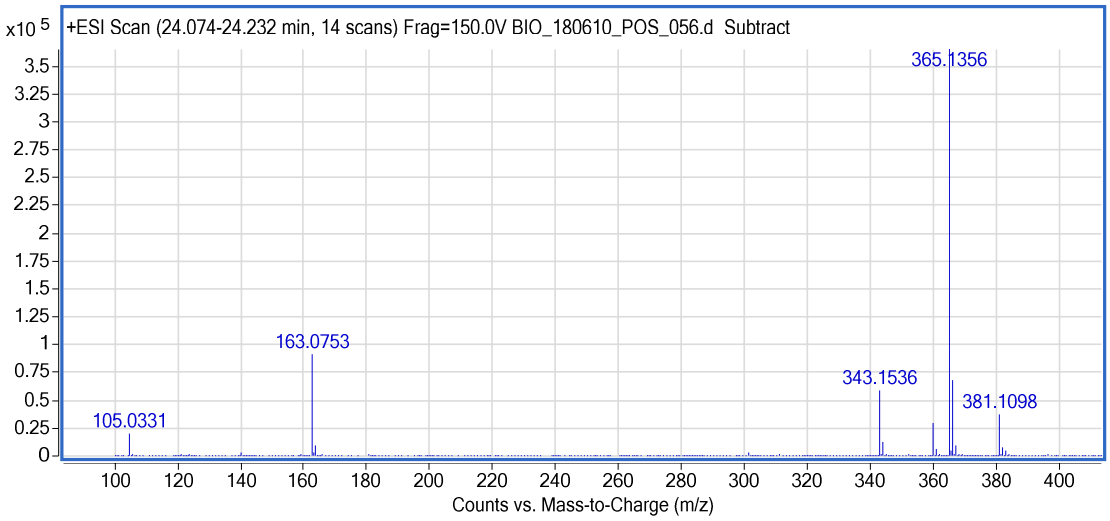
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H31NO8P	420.1787	2.0	
2	C20H26N3O7	420.1771	-1.9	
3	C22H28O8	420.1784	1.3	

S09-012083 positive mode ESI, peak at 23.7 minutes



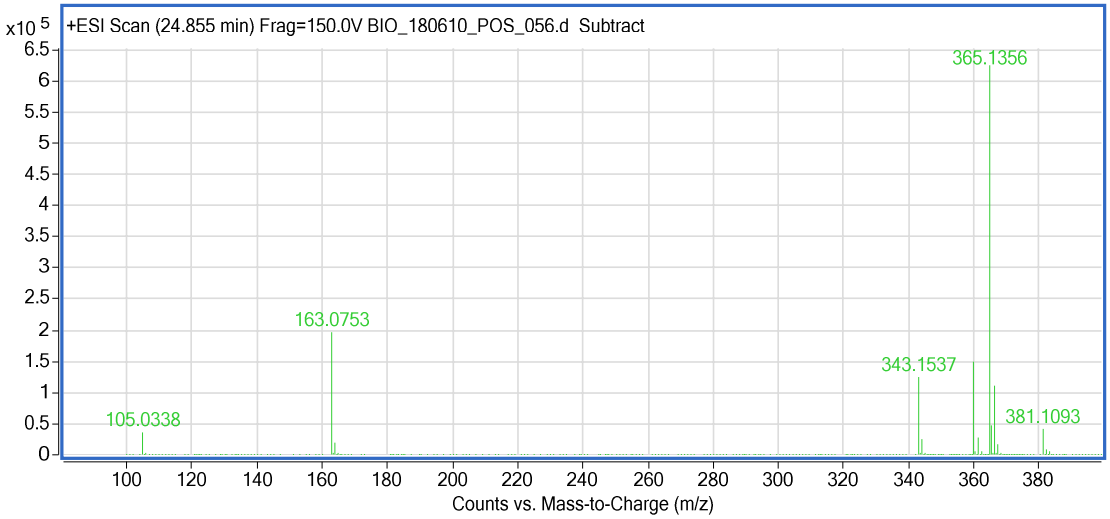
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C28H46N3O11	600.3132	-0.77	
2	C26H51NO12P	600.3149	2.0	
3	C30H48O12	600.3146	1.5	

S09-012083 positive mode ESI, peak at 24.1 minutes



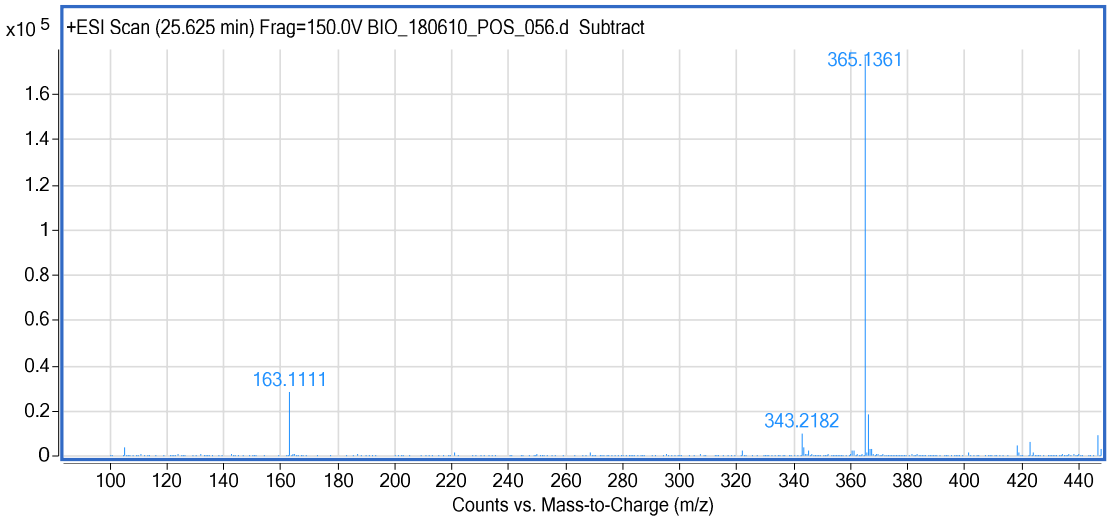
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H25NO5P	342.1470	2.1	
2	C14H23N4O4P	342.1457	-1.9	
3	C20H22O5	342.1467	1.1	

S09-012083 positive mode ESI, peak at 25.1 minutes



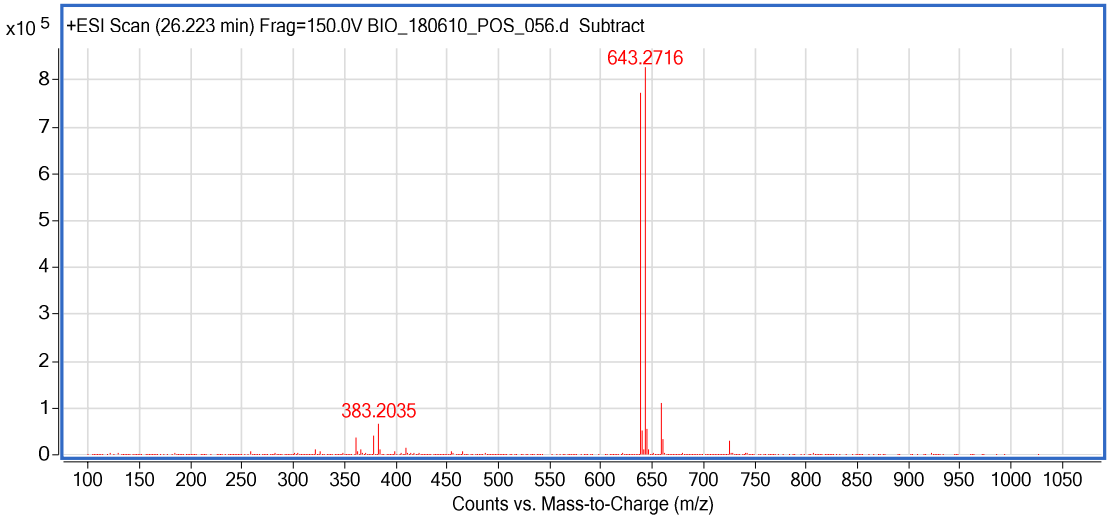
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H25NO5P	342.1470	2.0	
2	C14H23N4O4P	342.1457	-1.9	
3	C20H22O5	342.1467	1.1	

S09-012083 positive mode ESI, peak at 25.3 minutes



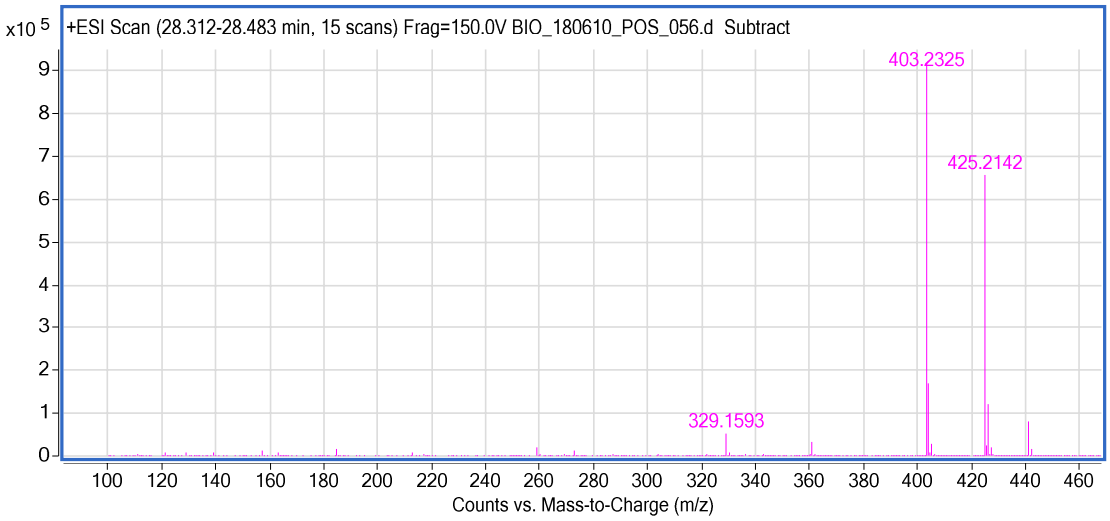
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H25NO5P	342.1470	0.55	
2	C14H23N4O4P	342.1457	-3.4	
3	C20H22O5	342.1467	-0.36	

S09-012083 positive mode ESI, peak at 26.1 minutes



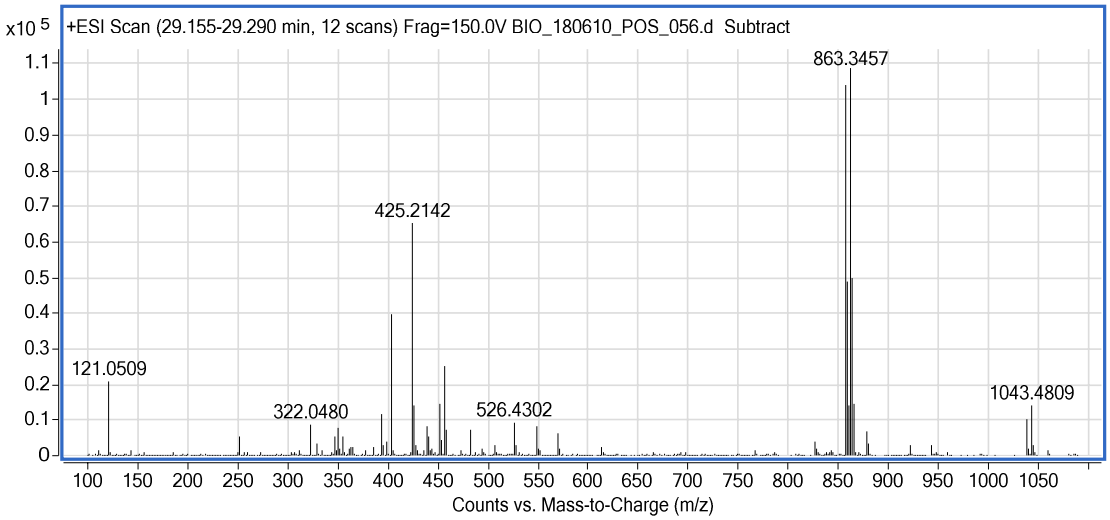
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C26H45N4O11P	620.2822	-0.18	
2	C20H42N3O11	620.2819	-0.68	
3	C32H44O12	620.2833	1.5	

S09-012083 positive mode ESI, peak at 28.1 minutes



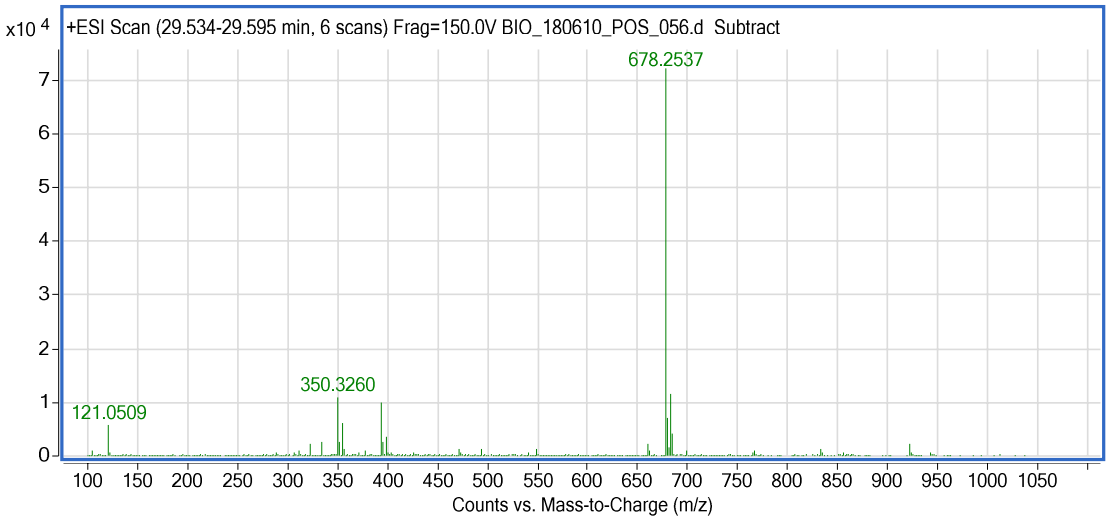
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H34O8	402.2254	1.0	

S09-012083 positive mode ESI, peak at 29.2 minutes



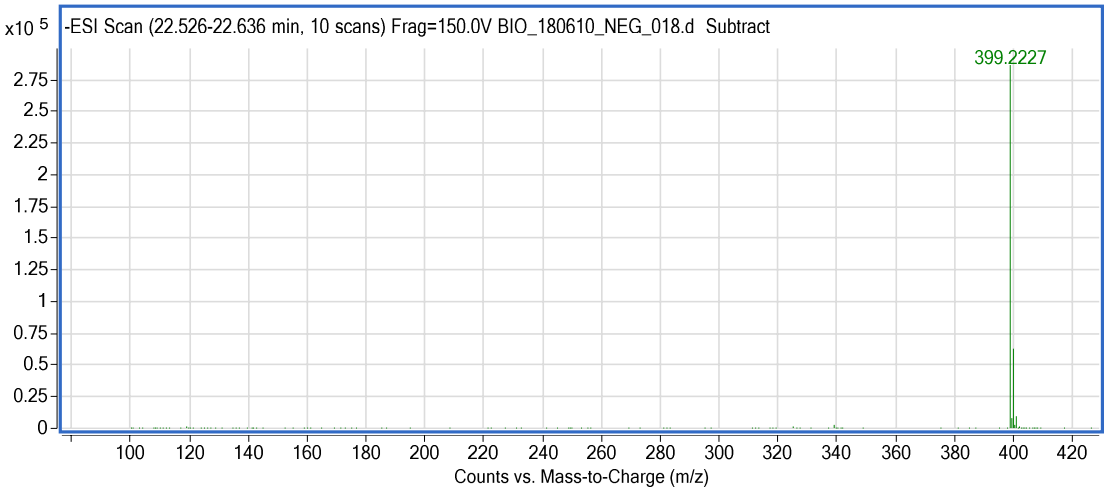
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C57H48N2O5	840.3563	-0.19	
2	C44H56O16	840.3568	0.42	

S09-012083 positive mode ESI, peak at 29.5 minutes



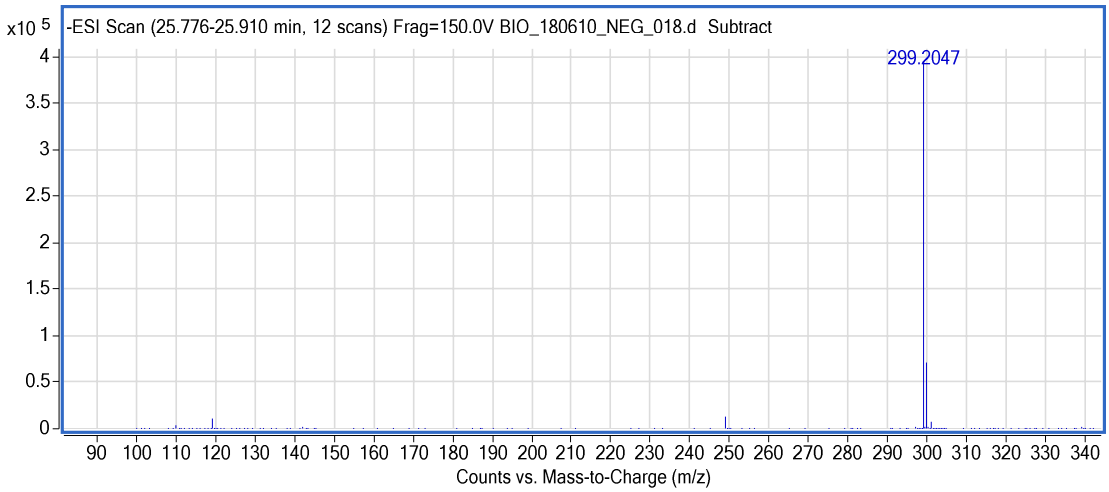
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C34H34N3O11	660.2193	-0.26	
2	C36H36O12	660.2207	-1.6	

S09-012083 negative mode ESI, peak at 22.6 minutes



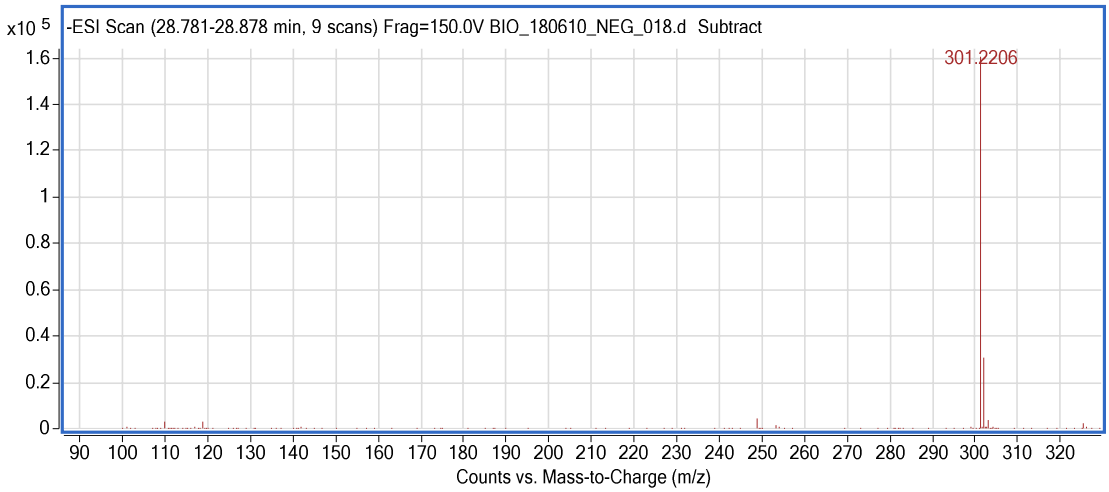
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H30N7O5	400.2308	2.2	
2	C17H36O10	400.2308	2.2	
3	C14H28N10O4	400.2295	-1.2	
4	C15H33N3O9	400.2295	-1.2	
5	C22H32N4OS	400.2297	-0.72	

S09-012083 negative mode ESI, peak at 25.8 minutes



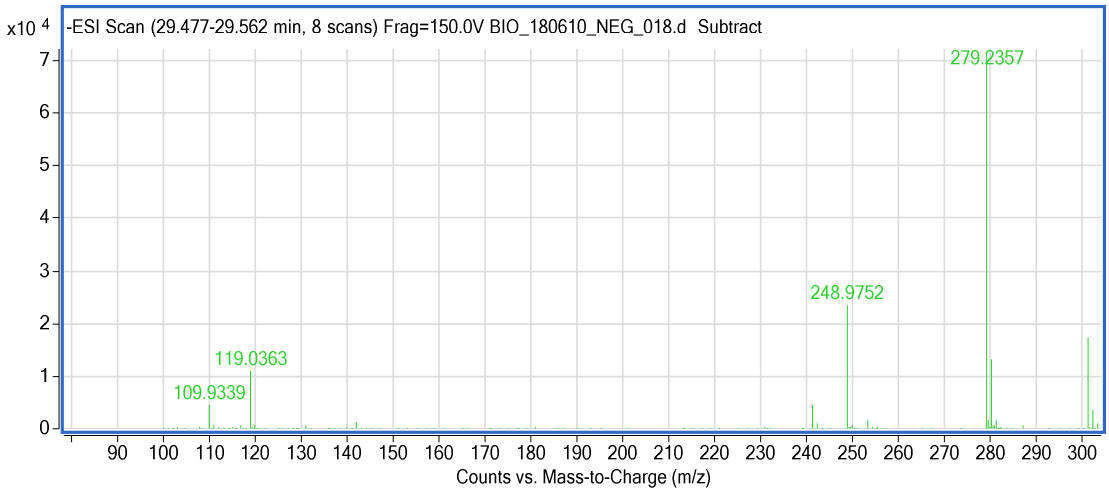
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C17H32O2S	300.2123	1.1	

S09-012083 negative mode ESI, peak at 28.8 minutes



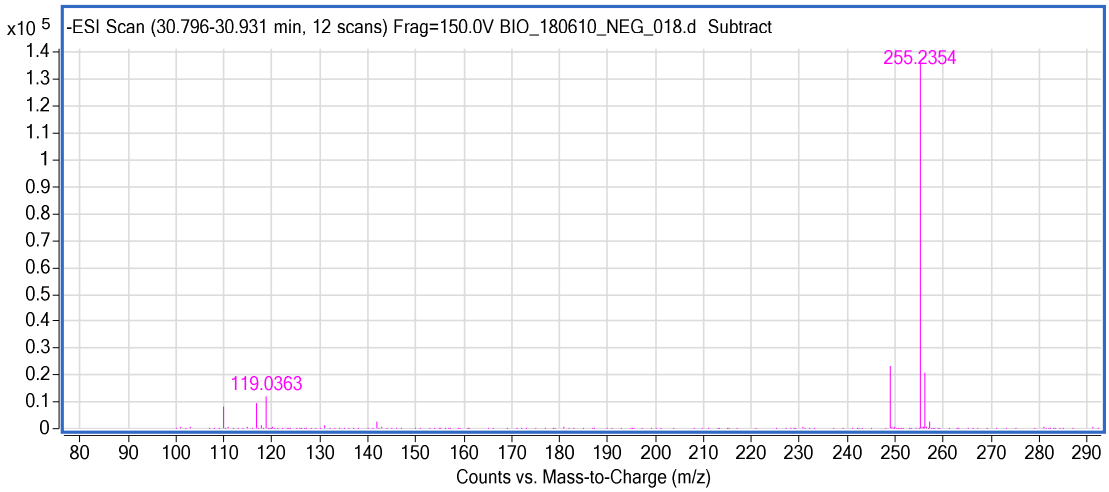
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C17H34O2S	302.2280	0.40	

S09-012083 negative mode ESI, peak at 29.5 minutes



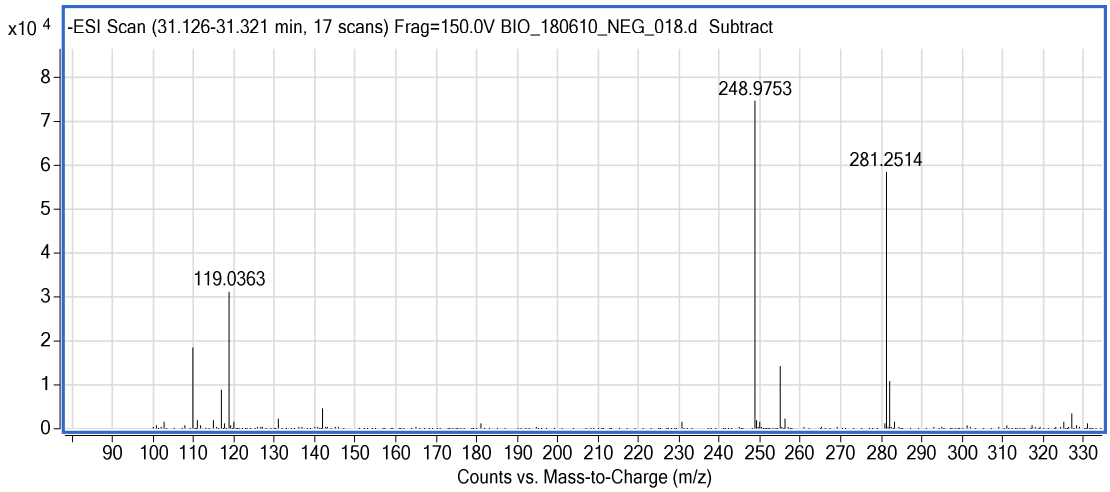
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H32O2	280.2402	-9.8	

S09-012083 negative mode ESI, peak at 30.8 minutes



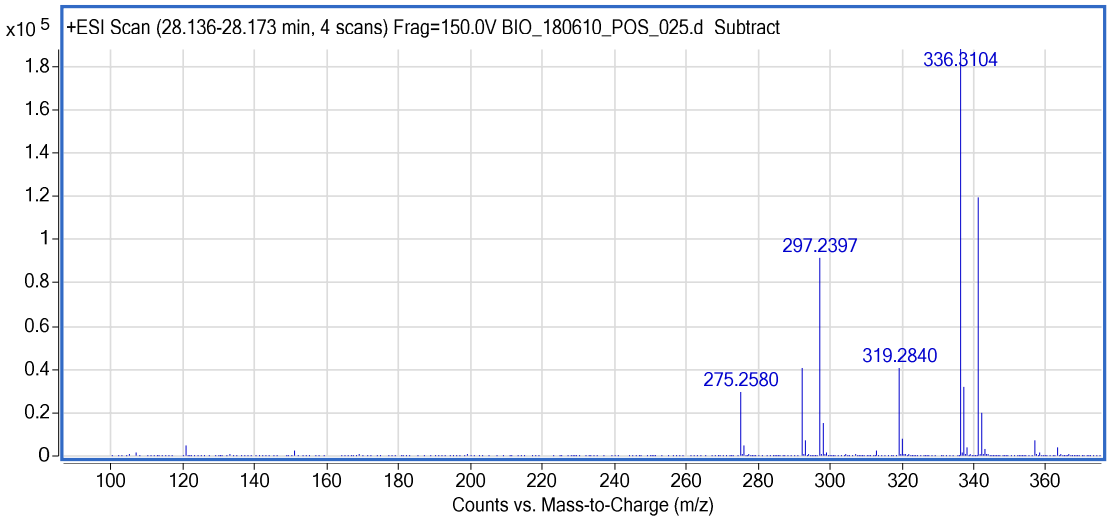
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2427	-9.5	

S09-012083 negative mode ESI, peak at 31.2 minutes



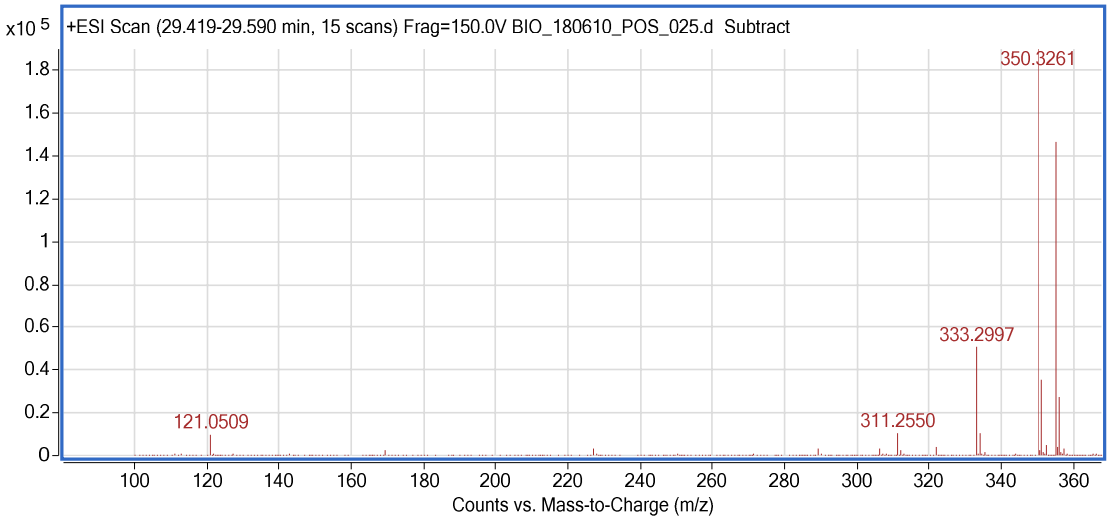
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O2	256.2427	-9.5	

S09-012092 positive mode ESI, peak at 28.1 minutes



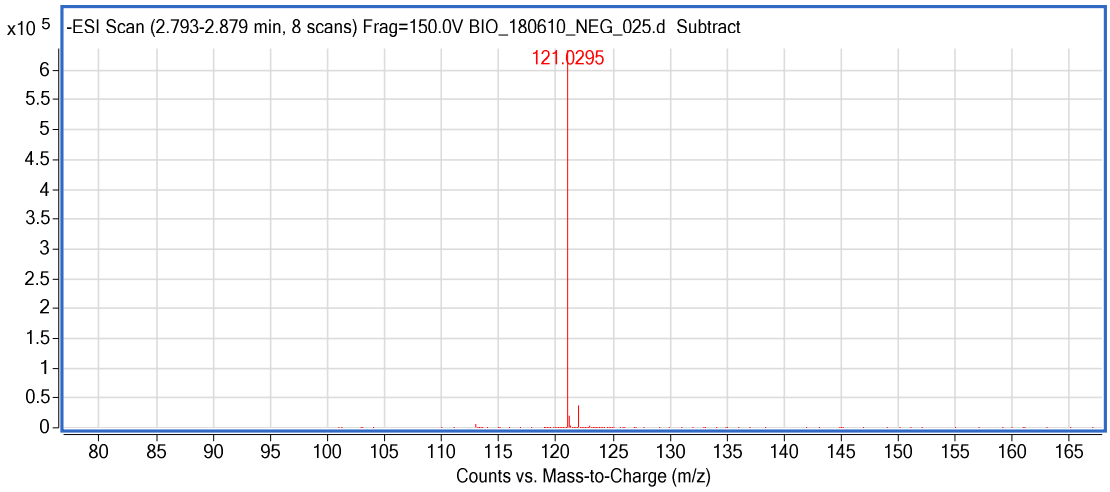
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H38O4	318.2770	1.3	
2	C16H36N3O3	318.2757	-3.0	

S09-012092 positive mode ESI, peak at 29.5 minutes



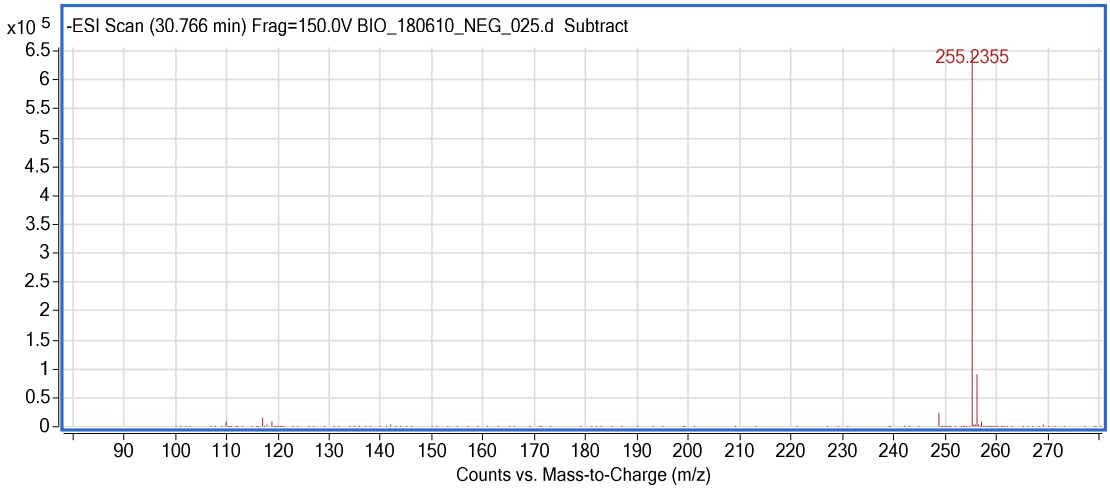
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C19H40O4	332.2927	1.4	
2	C17H38N3O3	332.2913	-2.6	

S09-012092 negative mode ESI, peak at 2.8 minutes



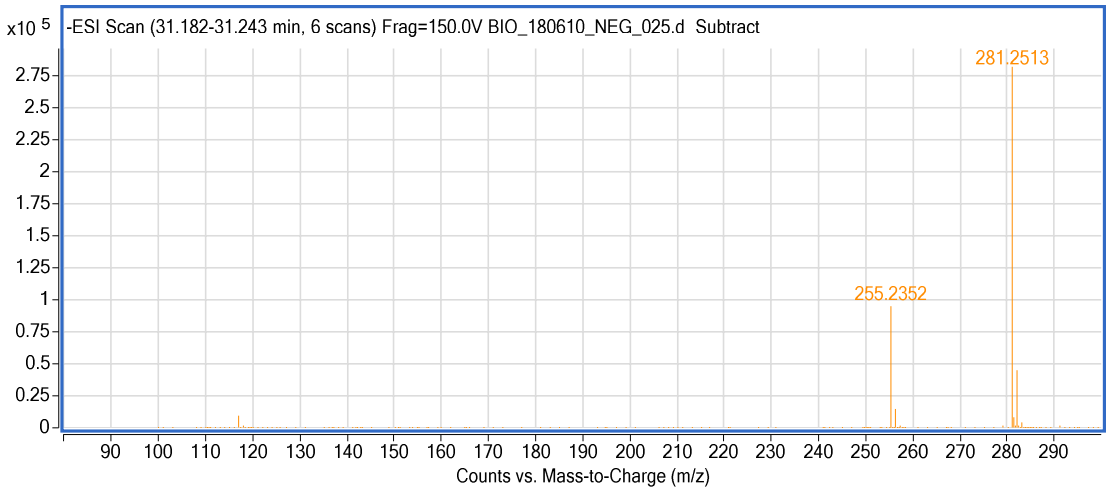
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C7H6O2	122.0368	-0.32	

S09-012092 negative mode ESI, peak at 30.9 minutes



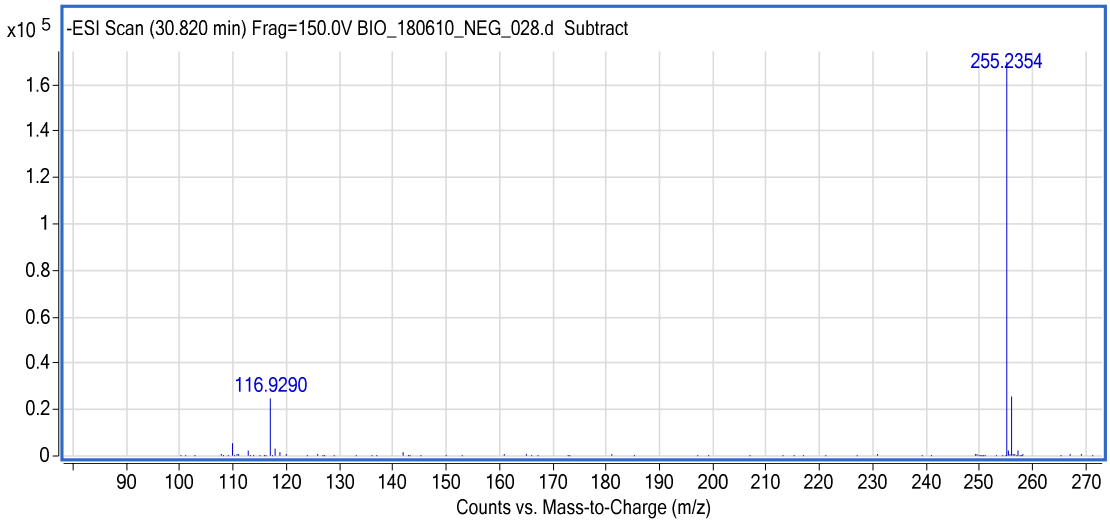
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2426	-9.43	

S09-012092 negative mode ESI, peak at 31.2 minutes



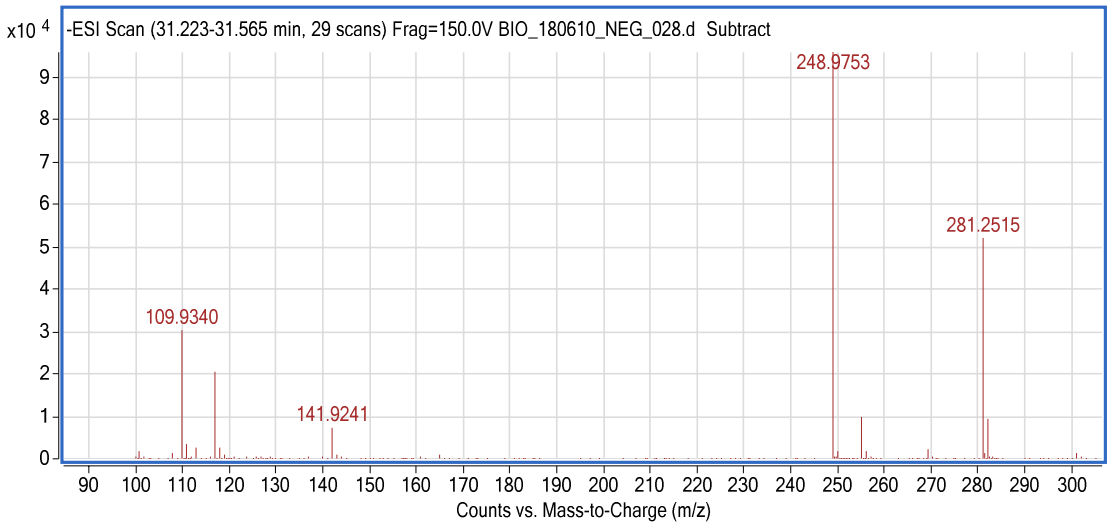
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O2	282.2587	-9.9	

S09-012093 negative mode ESI, peak at 30.9 minutes



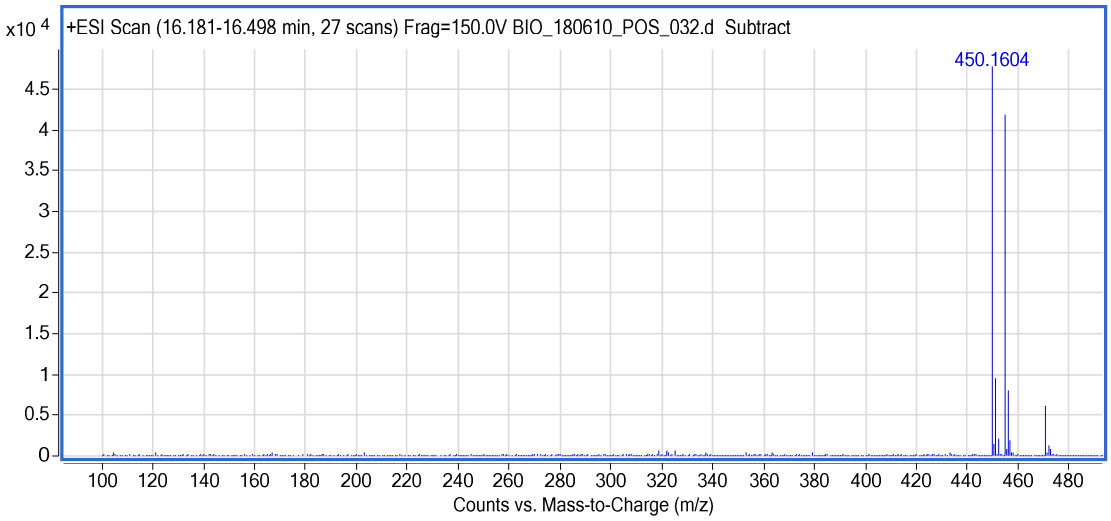
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2427	-9.6	

S09-012093 negative mode ESI, peak at 31.2 minutes



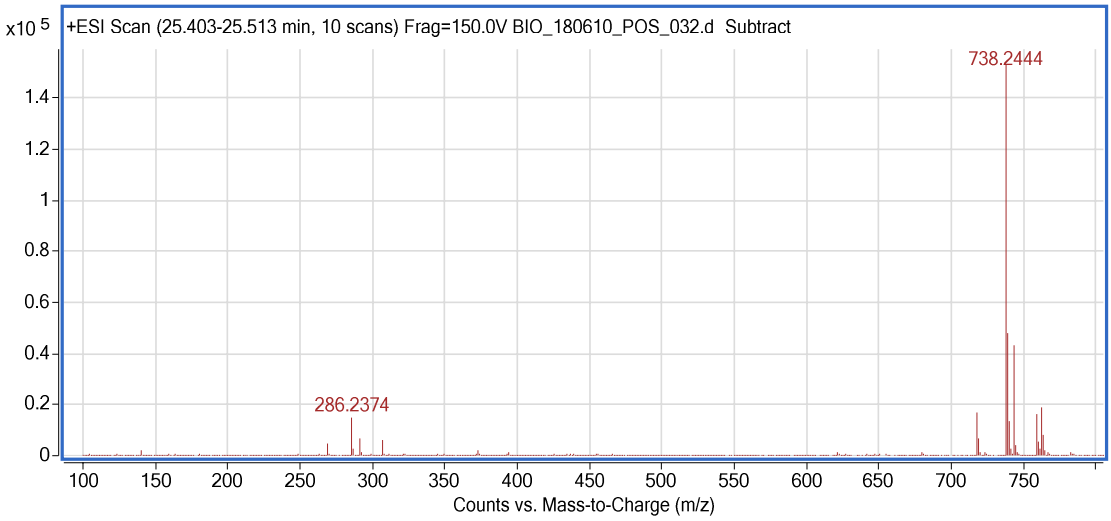
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O2	282.2588	-10.3	

S09-012095 positive mode ESI, peak at 16.3 minutes



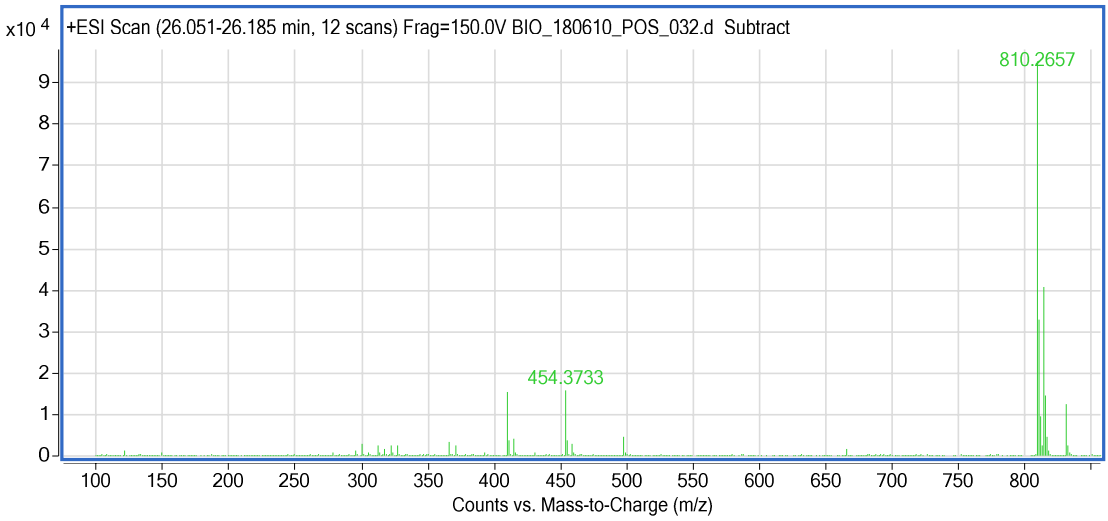
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H24O12	432.1268	0.33	

S09-012095 positive mode ESI, peak at 25.4 minutes



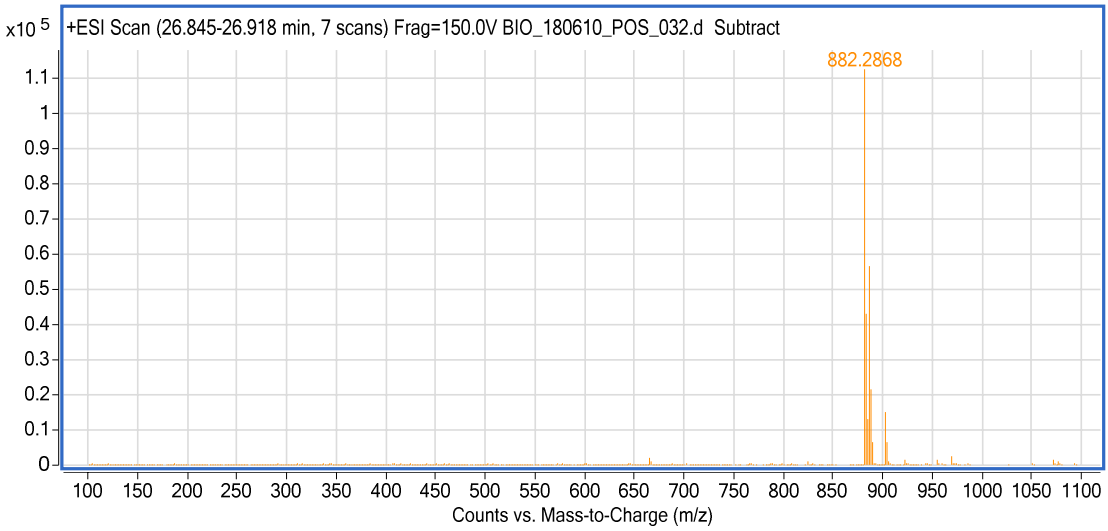
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C28H38N3O19	720.2100	-0.89	
2	C30H40O20	720.2113	0.98	

S09-012095 positive mode ESI, peak at 26.1 minutes



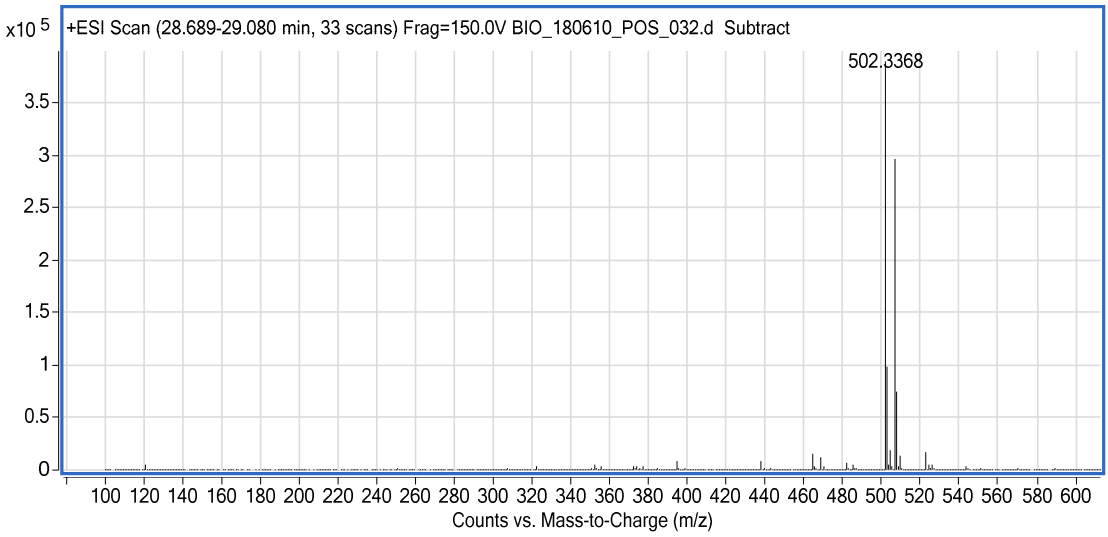
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C46H36N2O11	792.2319	-0.09	
2	C33H44O22	792.2324	0.56	

S09-012095 positive mode ESI, peak at 26.9 minutes



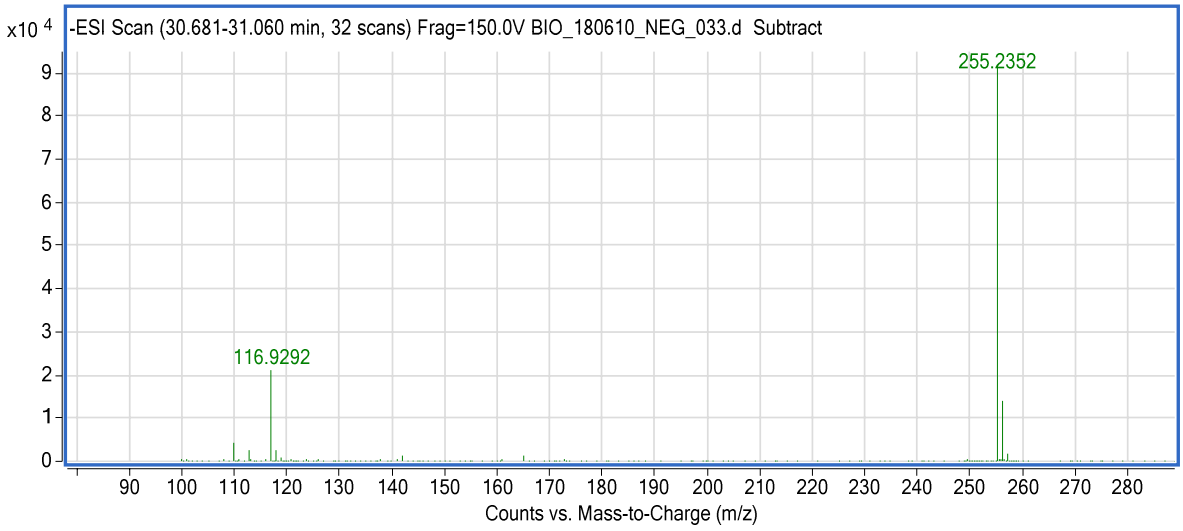
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C49H40N2O13	864.2530	-0.05	
2	C36H48O24	864.2536	0.55	

S09-012095 positive mode ESI, peak at 28.9 minutes



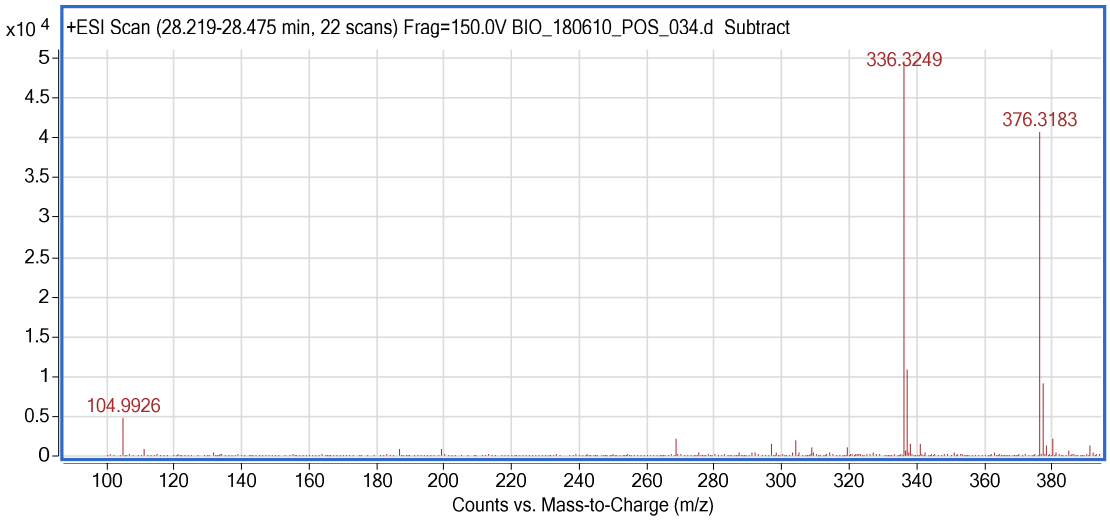
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C20H45N4O7P	484.3026	-0.92	
2	C26H44O8	484.3036	1.2	

S09-012095 negative mode ESI, peak at 30.9 minutes



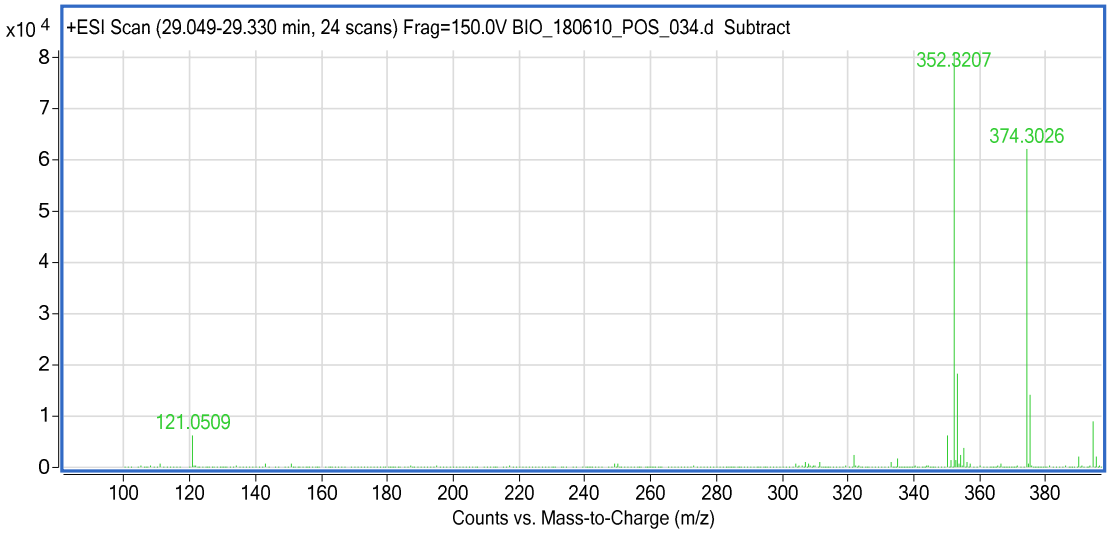
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2425	-9.6	

S09-012096 positive mode ESI, peak at 28.4 minutes



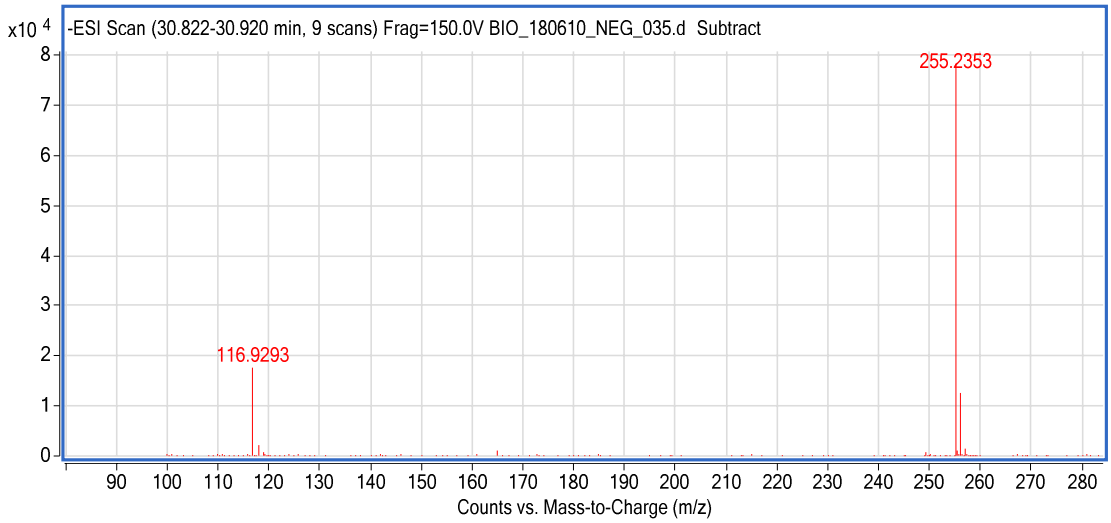
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H43NO2	353.3294	0.94	
2	C20H41N4O	353.3280	-2.9	

S09-012096 positive mode ESI, peak at 29.1 minutes



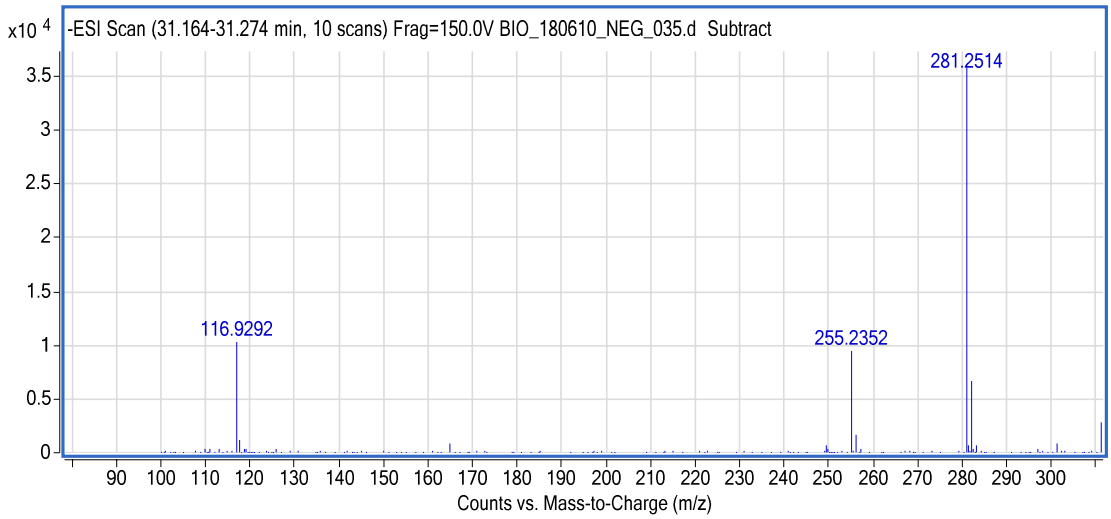
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H41NO2	351.3137	0.98	
2	C20H39N4O	351.3124	-2.9	

S09-012096 negative mode ESI, peak at 30.8 minutes



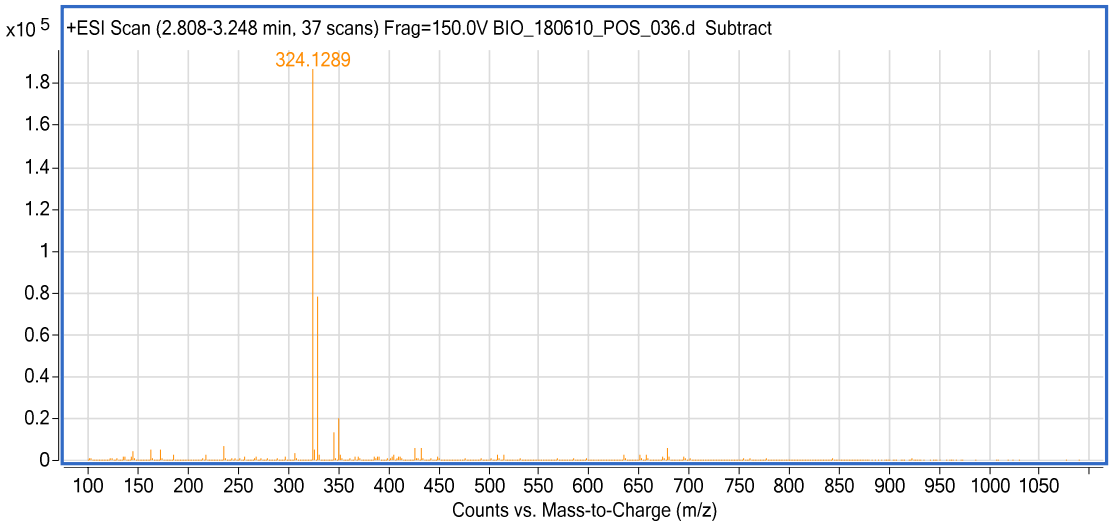
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2426	-9.2	

S09-012096 negative mode ESI, peak at 31.2 minutes



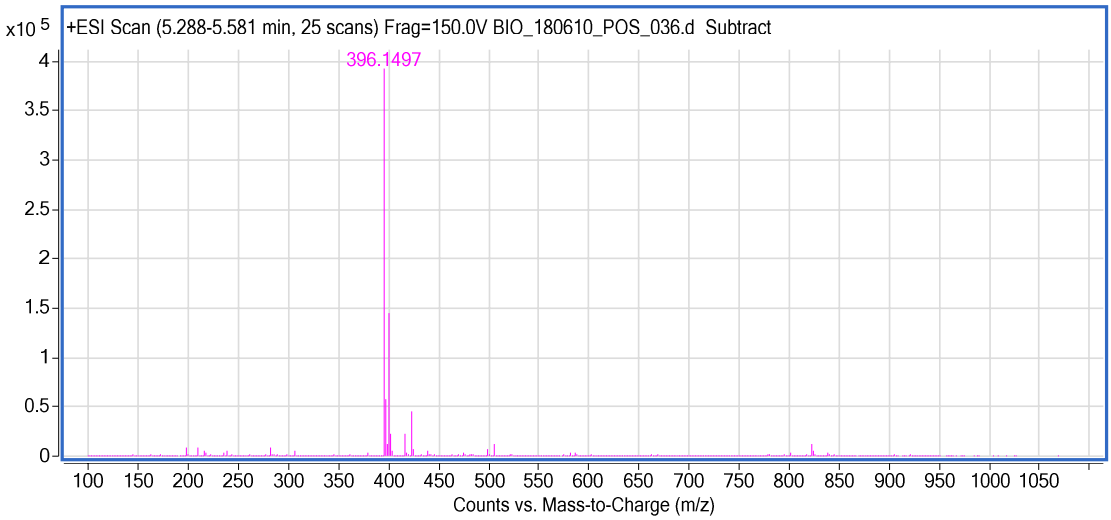
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H34O2	281.2490	-9.9	

S09-012097 positive mode ESI, peak at 3.1 minutes



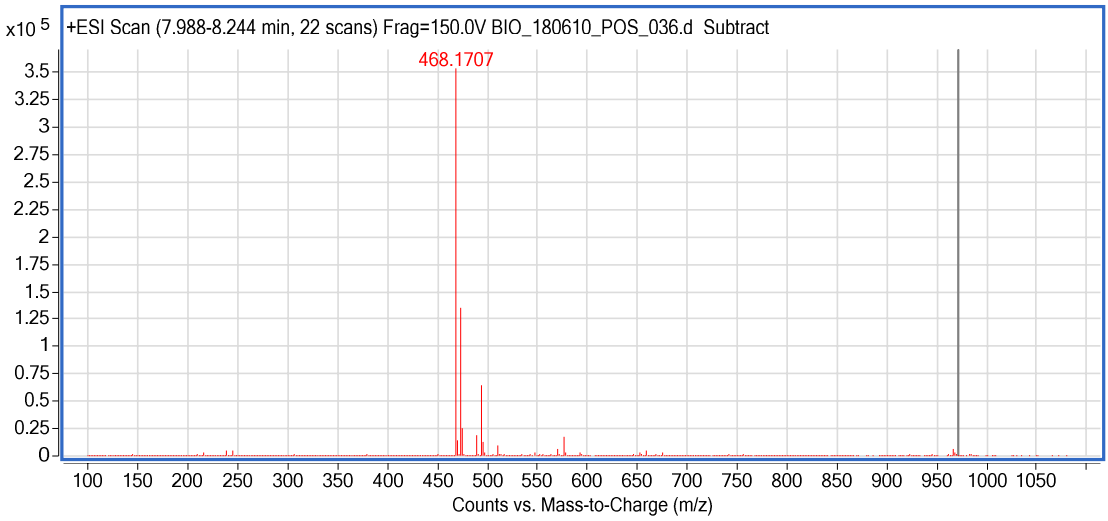
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C12H18O9	306.0951	0.58	

S09-012097 positive mode ESI, peak at 5.4 minutes



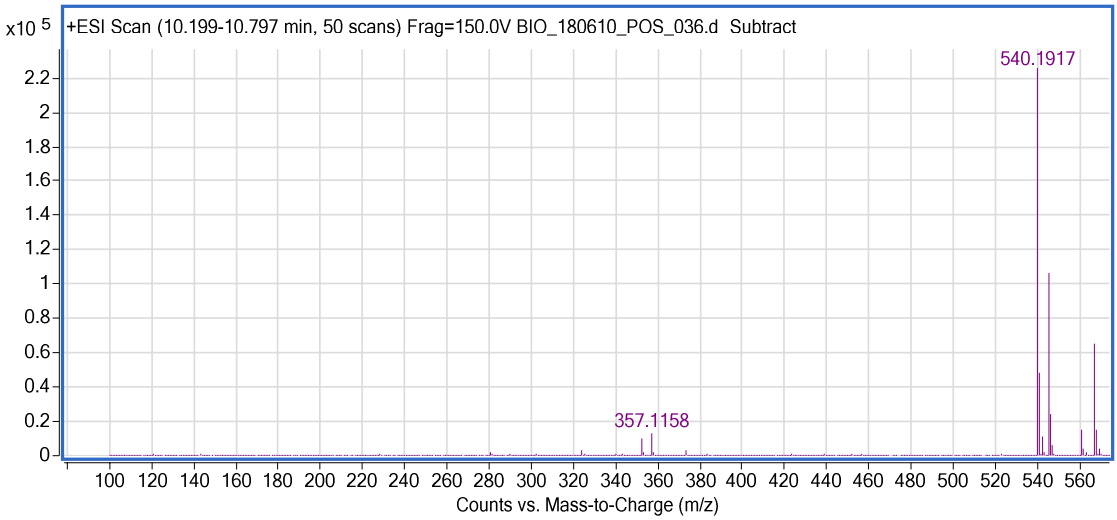
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C15H22O11	378.1162	0.98	

S09-012097 positive mode ESI, peak at 8.2 minutes



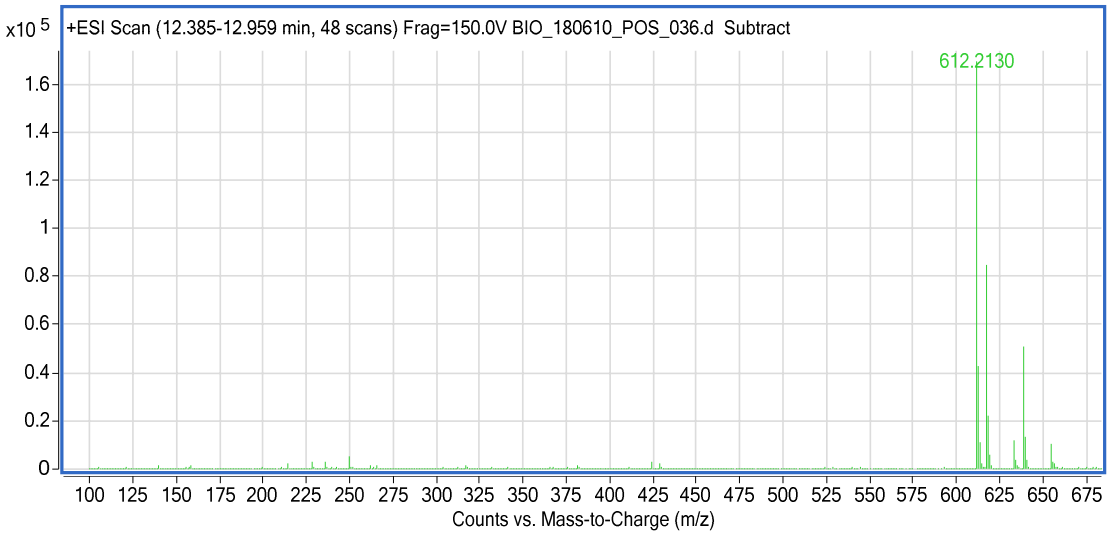
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H26O13	450.1373	0.07	

S09-012097 positive mode ESI, peak at 10.6 minutes



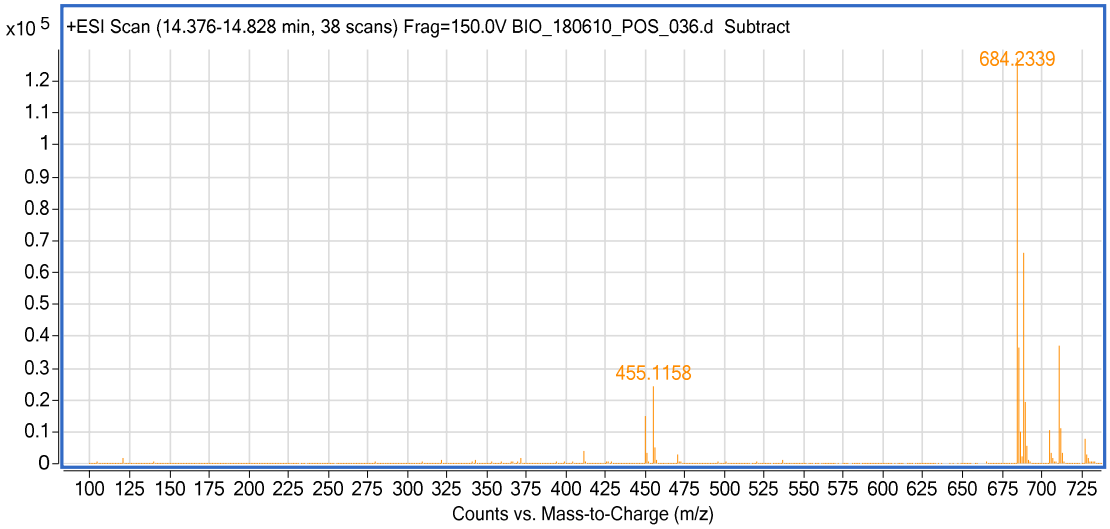
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C21H30O15	522.1585	1.2	

S09-012097 positive mode ESI, peak at 12.8 minutes



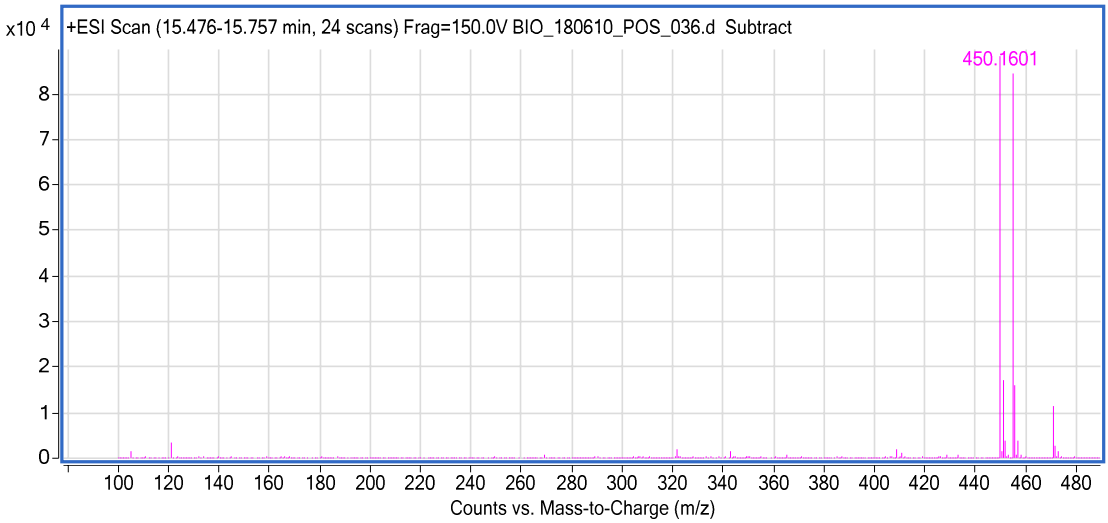
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C24H34O17	594.1796	0.49	

S09-012097 positive mode ESI, peak at 14.4 minutes



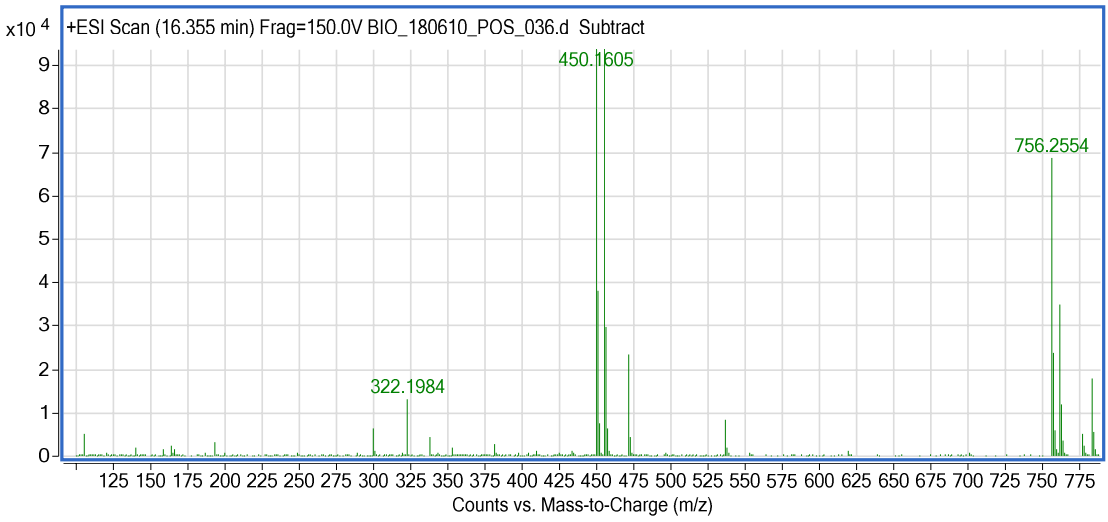
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C27H38O19	666.2007		

S09-012097 positive mode ESI, peak at 15.5 minutes



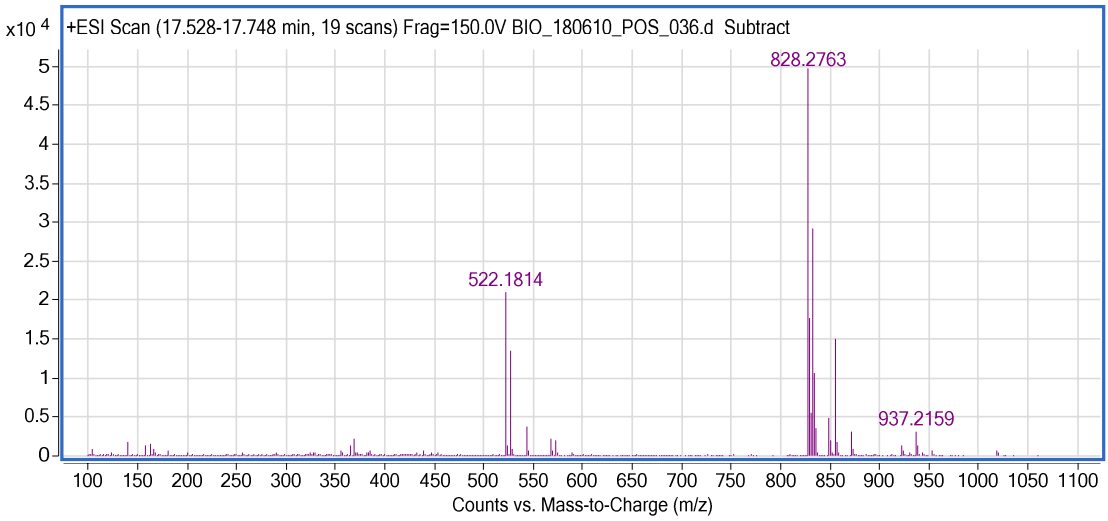
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H24O12	432.1298	0.66	

S09-012097 positive mode ESI, peak at 16.3 minutes



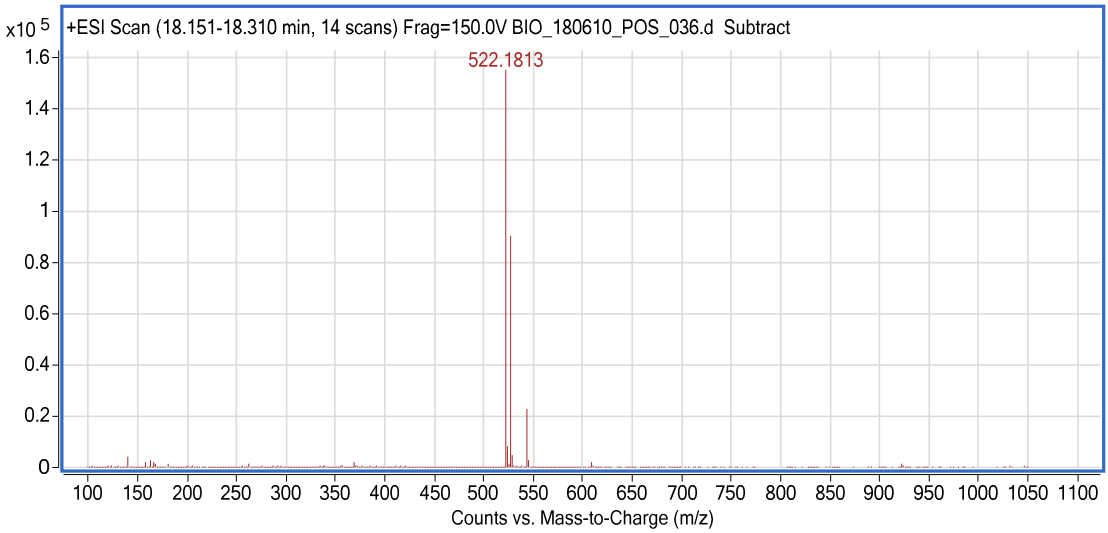
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C30H42O21	738.2219	0.78	

S09-012097 positive mode ESI, peak at 17.6 minutes



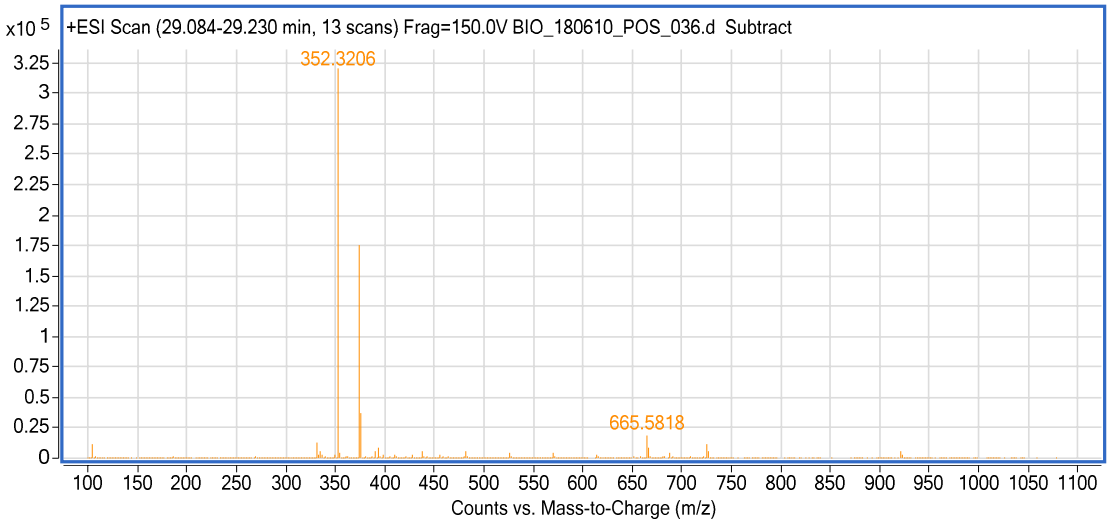
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C33H46O23	810.2430	0.50	

S09-012097 positive mode ESI, peak at 18.2 minutes



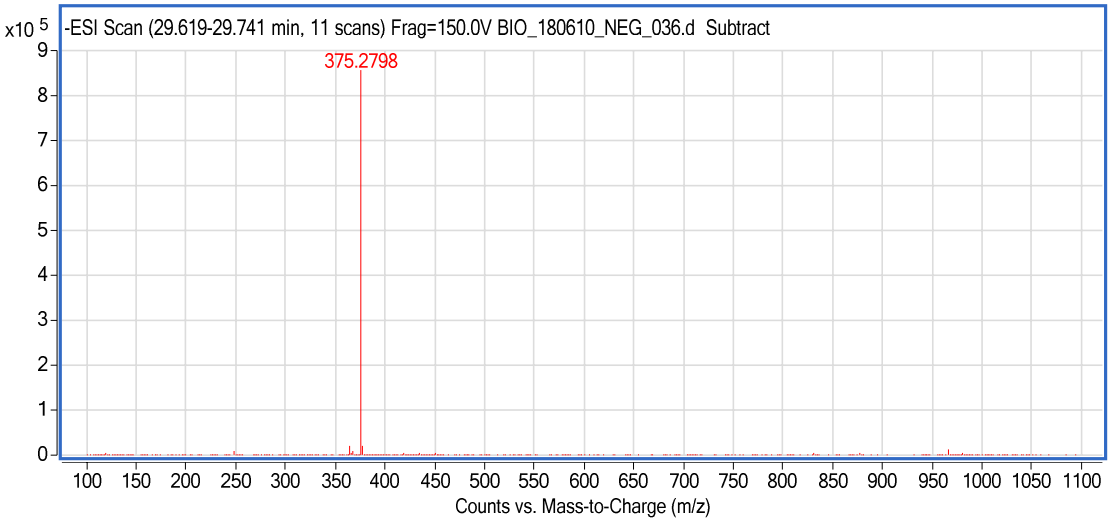
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C21H28O14	504.1479	0.86	

S09-012097 positive mode ESI, peak at 29.2 minutes



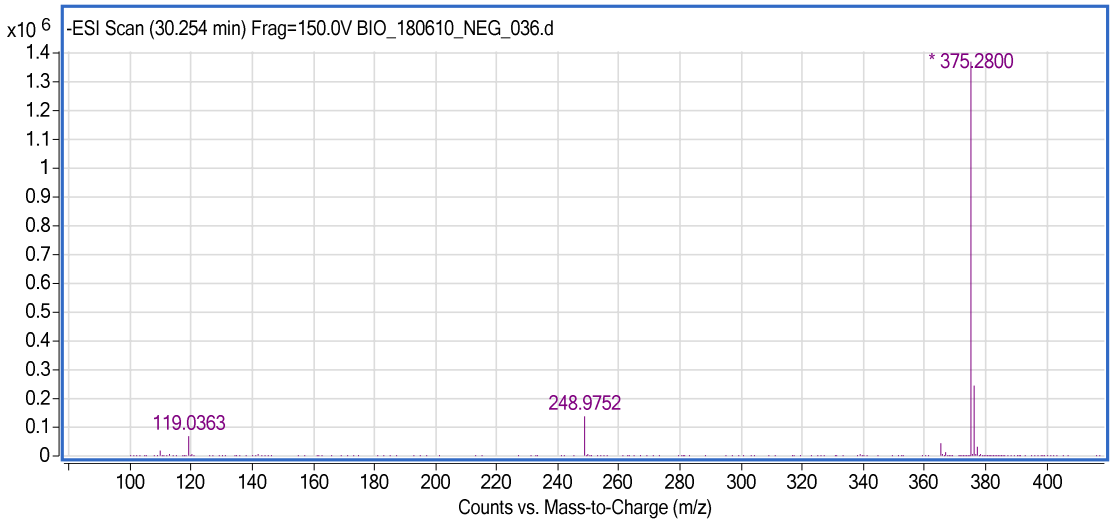
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C22H41NO2	351.3137	1.1	
2	C20H39N4O	351.3124	-2.7	

S09-012097 negative mode ESI, peak at 29.7 minutes



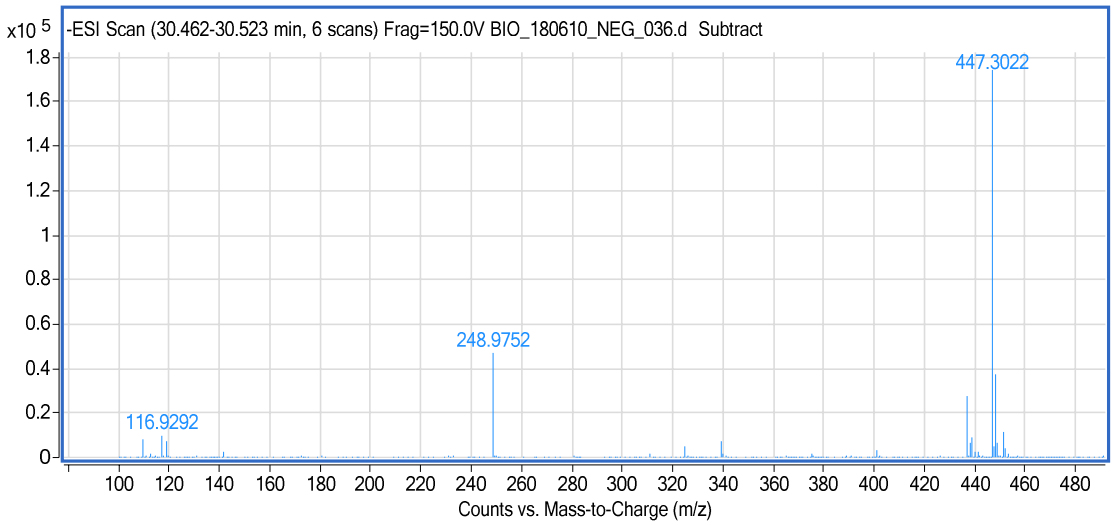
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H39N4O2S	376.2872	0.33	

S09-012097 negative mode ESI, peak at 30.1 minutes



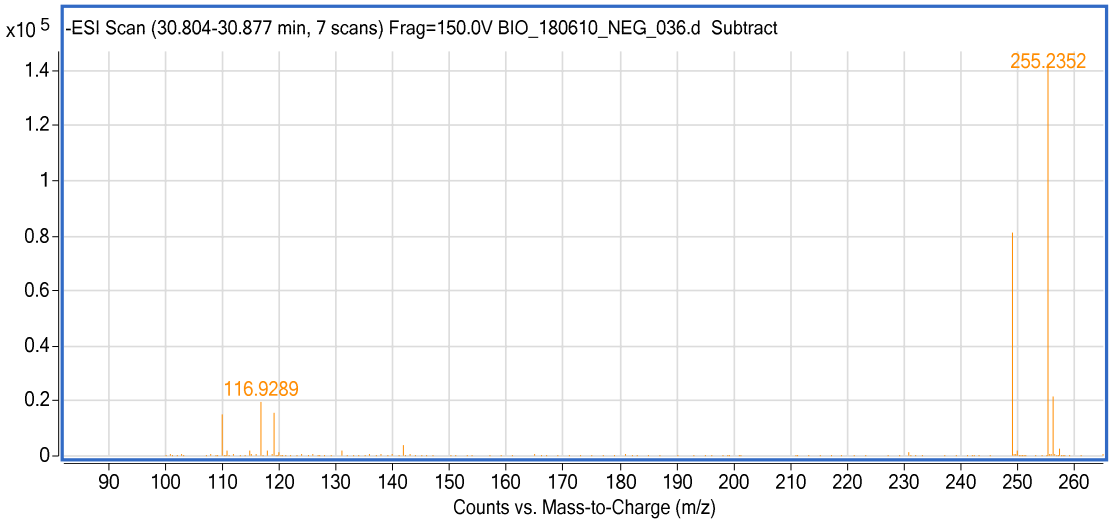
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H39N4O2S	376.2872	-0.24	

S09-012097 negative mode ESI, peak at 30.5 minutes



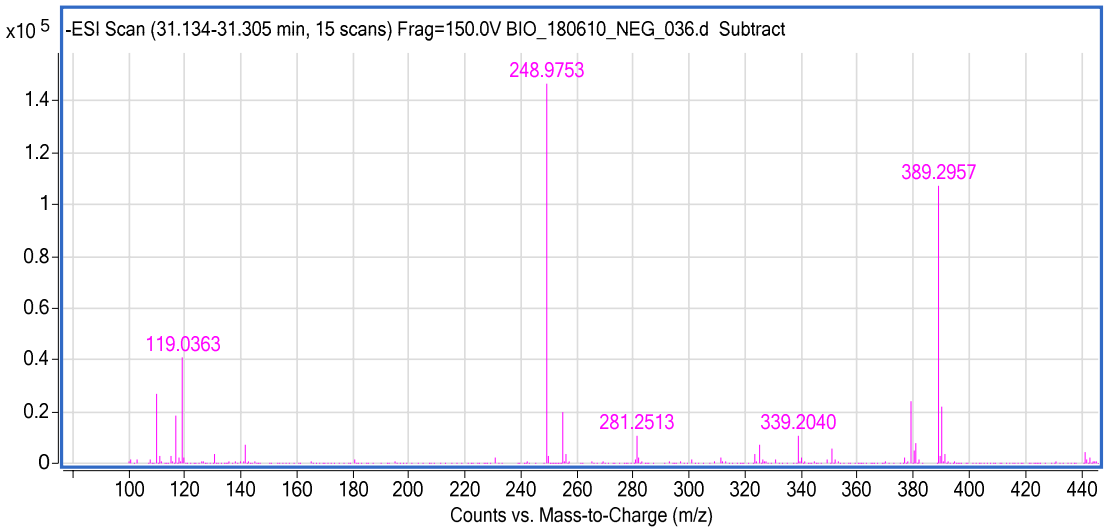
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C23H46NO5S	448.3095	0.41	

S09-012097 negative mode ESI, peak at 30.9 minutes



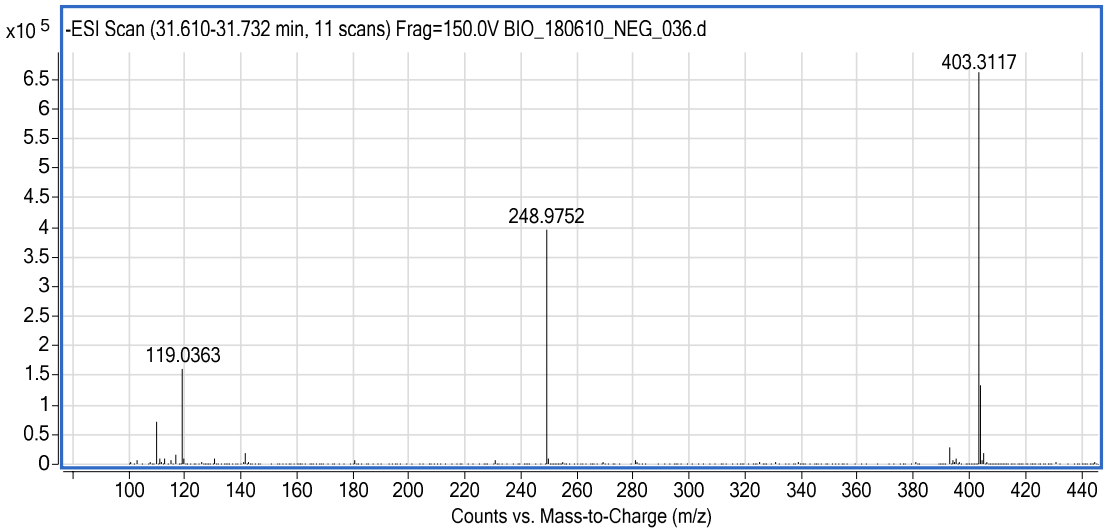
	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C16H32O2	256.2425	-8.9	

S09-012097 negative mode ESI, peak at 31.2 minutes



	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C19H42N4O2S	390.3028	-0.22	

S09-012097 negative mode ESI, peak at 31.7 minutes



	Formula	Calculated m/z	Error (ppm)	Proposed identity
1	C18H42N7OS	404.3190	-4.7	