INFORME DE ANÁLISE DE GRANULADOS DE PLÁSTICO

Informe realizado por:

- ***Isaac Rodríguez Pereiro***
- ***Mª Rosario Rodil Rodríguez***
- ***José Benito Quintana Álvarez***
- ***Javier López Vázquez***
- ***Miguel Cobo Golpe***

Contacto: isaac.rodriguez@usc.es, rosario.rodil@usc.es, jb.quintana@usc.es

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CONTEXTUALIZACIÓN

Analizáronse 3 mostras de granulado:

A petición de INTECMAR:

- ***Mostra recibida o 11 de Xaneiro de granulado recollido en saco sen contacto coa area (código de mostra INTECMAR 1)***
- ***Mostra recibida o 12 de Xaneiro de granulado recollido con contacto coa area (código de mostra INTECMAR 2)***
- ***Mostra recollida por una ONG no Concello de Muros e recibida no laboratorio o 11 de Xaneiro (código ONG)***

ANÁLISE:

Análise cualitativo (a espera de recibir patróns puros) directo mediante FTIR (modo ATR), e tras extracción Soxhlet mediante cromatografía de gases con espectrometría de masas de alta e baixa resolución (GC-MS, QTOF) e cromatografía de líquidos con espectrometría de masas de alta resolución (LC-MS, QTOF)

RESUMO DAS CONCLUSIÓNS ATA O MOMENTO

Trátase dun granulado de polietileno contendo maioritariamente:

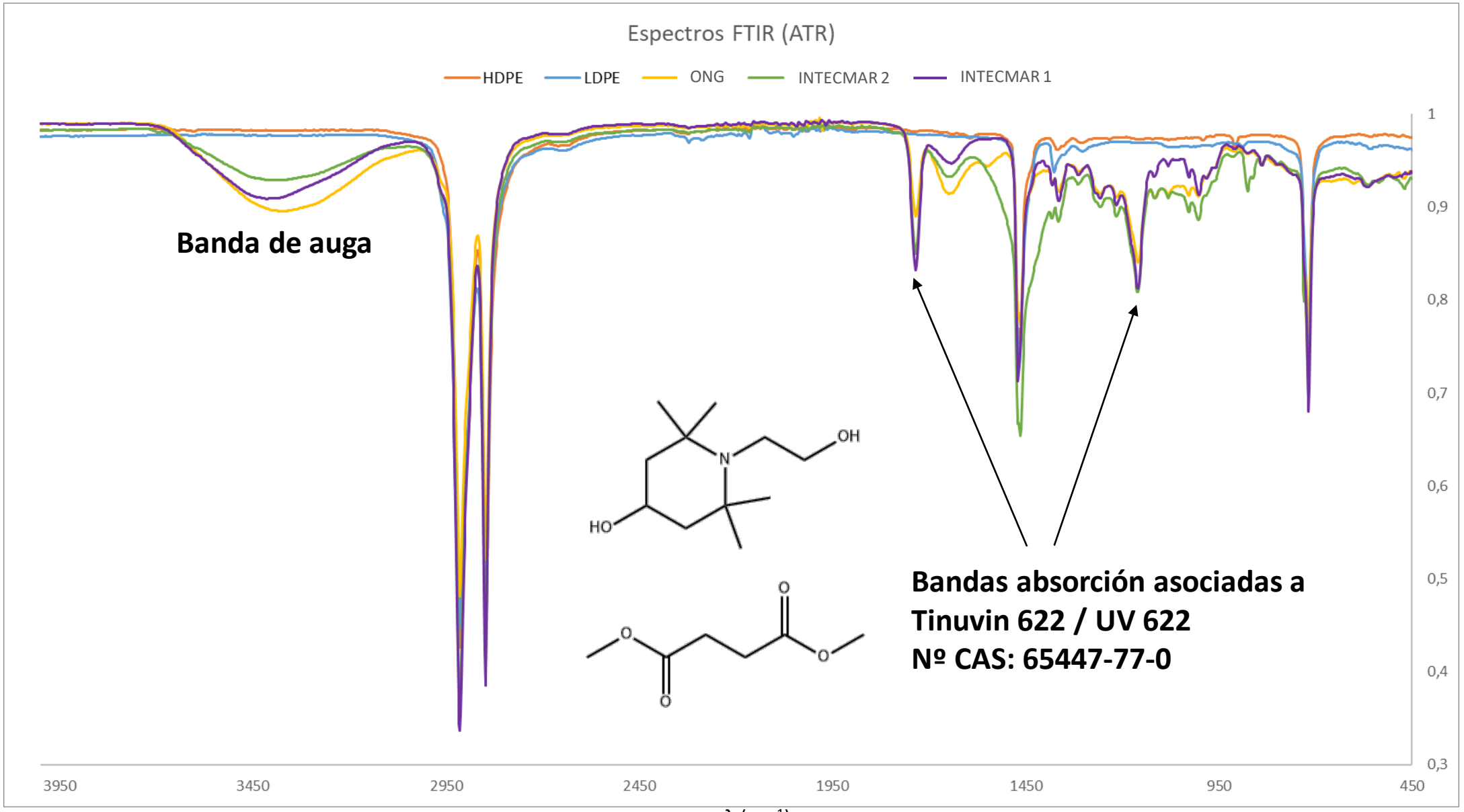
A) Compostos declarados na folla de especificación do produto:
- Tinuvin 622 (UV 622, Nº CAS: 65447-77-0): ver FTIR
- Irgafos 168 (Nº CAS: 31570-04-4): ver GC-MS e LC-MS – Confirmado con patrón analítico
- Irganox 1010 (Nº CAS: 6683-19-8): ver LC-MS – Confirmado con patrón analítico

B) Compostos non declarados na folla de especificación do produto
- Irgafos 168 fosfato (Nº CAS: 95906-11-9): ver GC-MS e LC-MS (produto formado por oxidación do Irgafos 168) – Confirmado con patrón analítico
- Irganox 1076 (Nº CAS: 2082-79-3): ver GC-MS e LC-MS
- 8-Pentadecanona (Nº CAS: 818-23-5): ver GC-MS
- 10-Nonadecanona (Nº CAS: 504-57-4): ver GC-MS

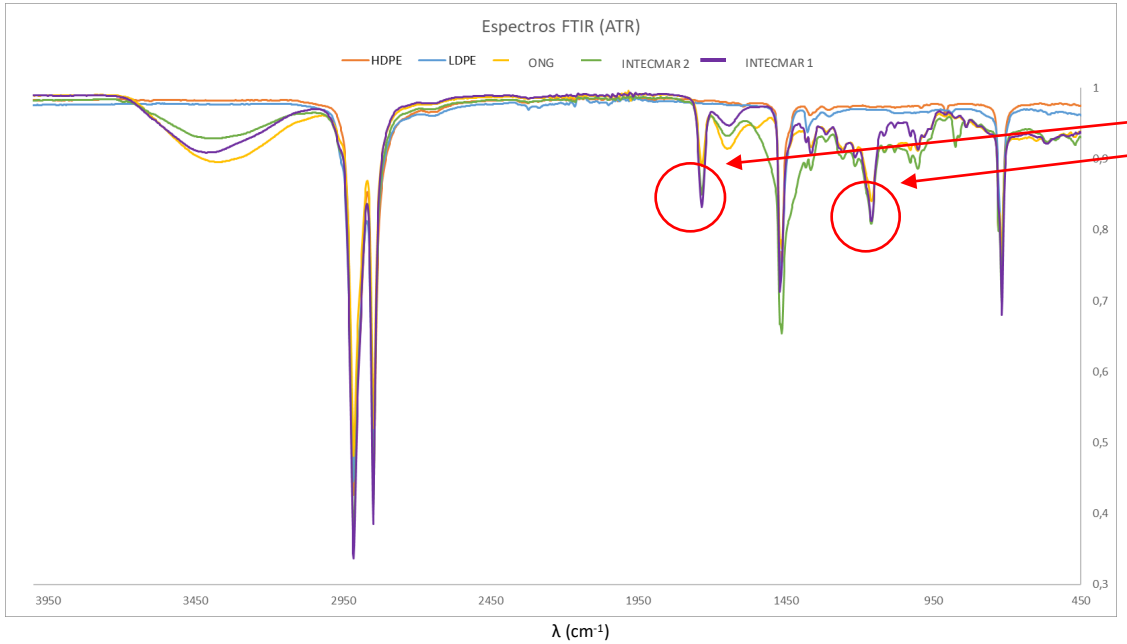
As tres mostras presentan o mesmo perfil cromatográfico (GC-TOF-MS), polo que serían o mesmo material.

Estase traballando na identificación de compostos adicionais

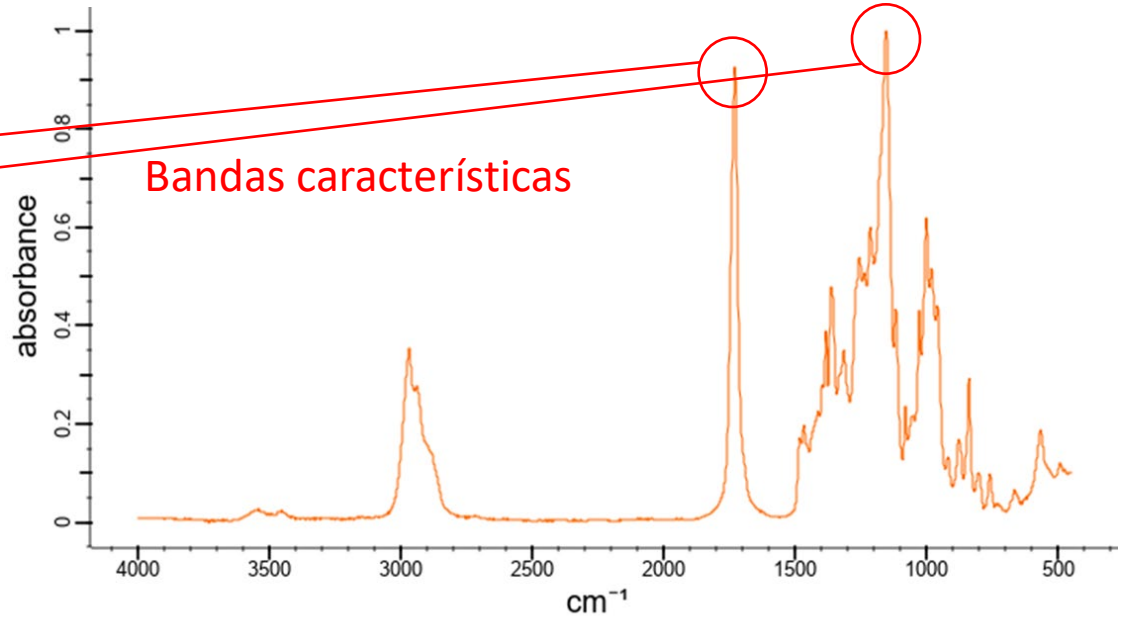
Espectros experimentais das mostras comparados con LDPE e HDPE



Espectros experimentais Vs referencia Tinuvin 622

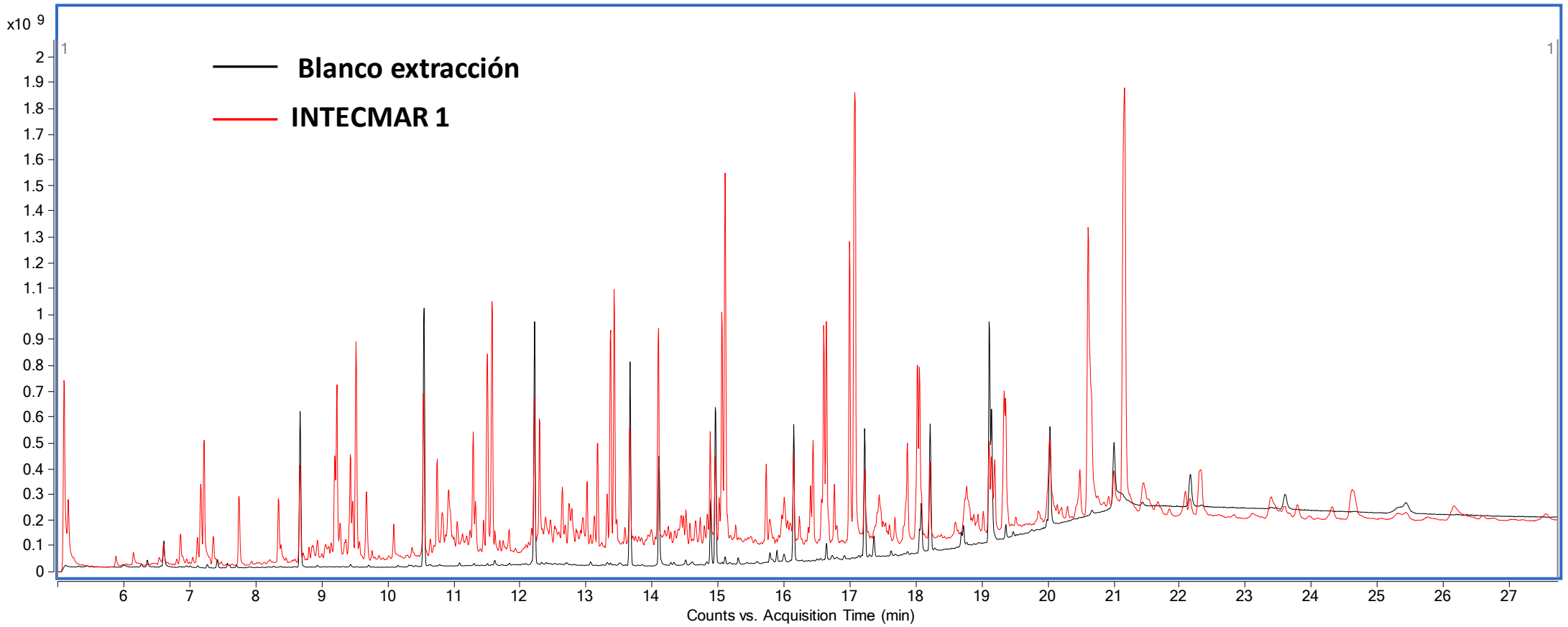


Espectros experimentais



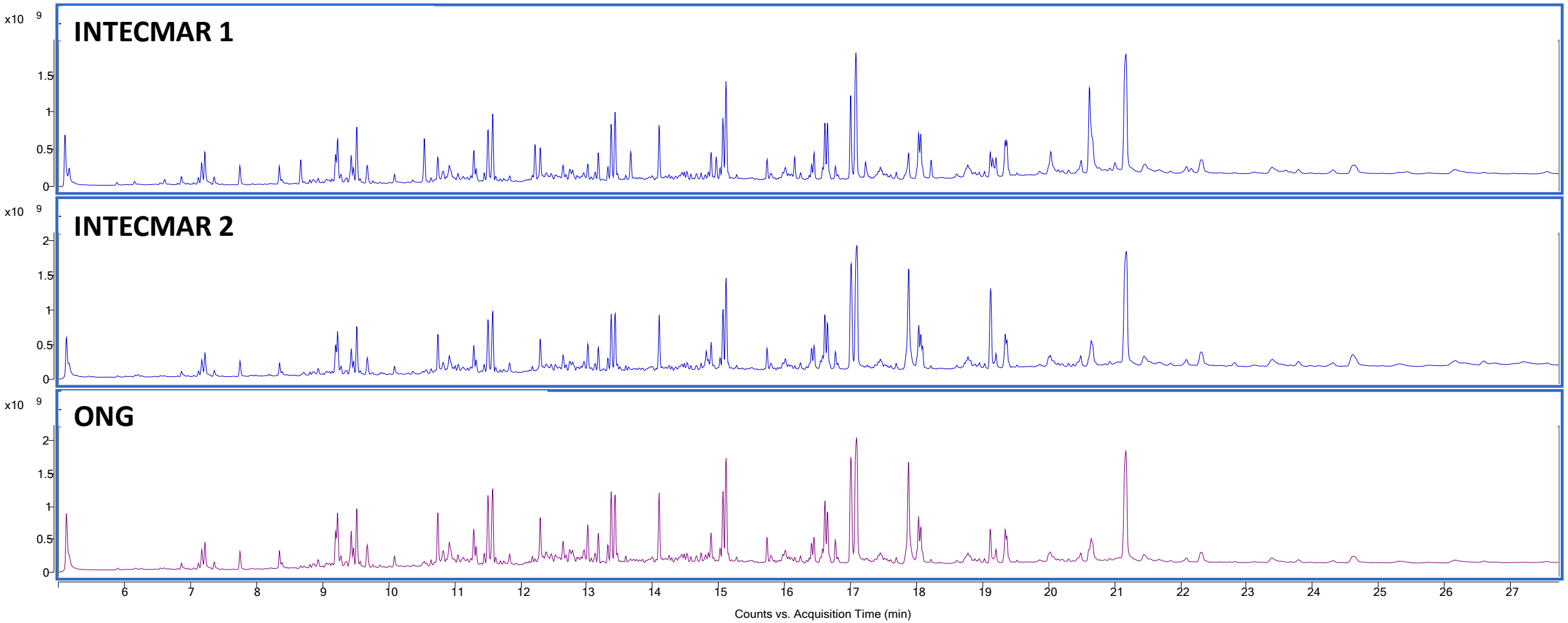
Espectro de referencia (bibliografía) de Tinuvin 622
https://spectrabase.com/spectrum/2HhLdKaHhWf

Perfís das mostras: GC-MS (QTOF) – método A



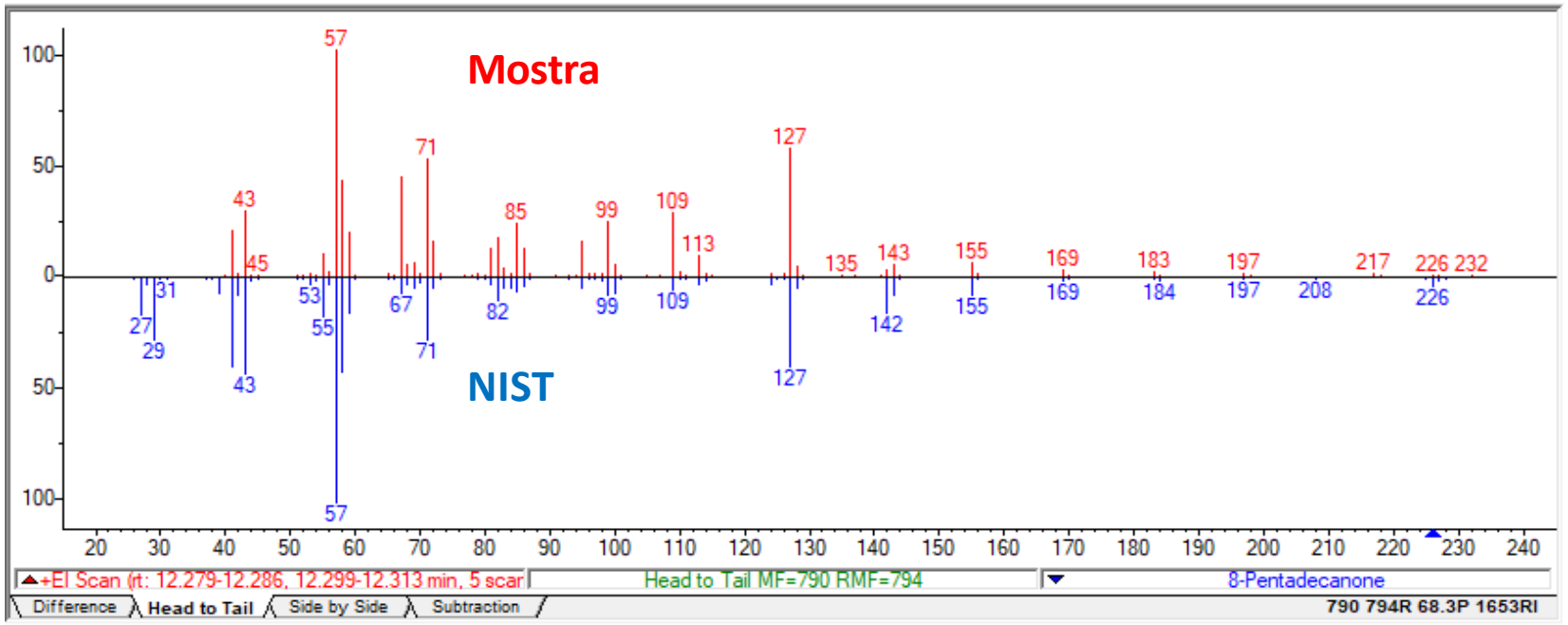
Comparación perfil da mostra sen contacto coa area do blanco de proceso

Perfís das mostras: GC-MS (QTOF) – método A



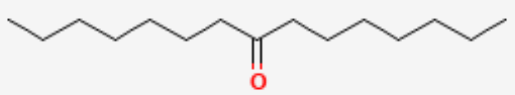
Perfís similares para as tres mostras analizadas. As pequeñas diferencias foron identificadas como picos de siloxanos

Identificación: GC-MS (QTOF) fronte biblioteca NIST



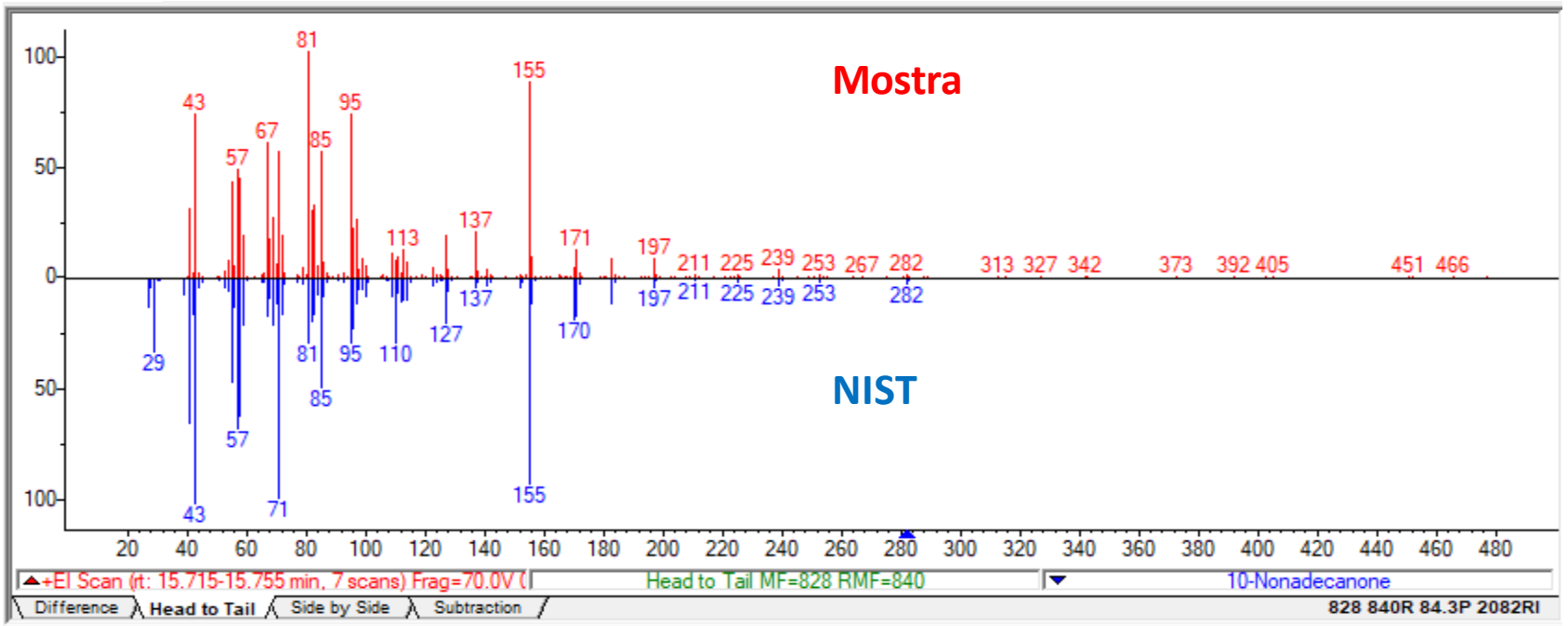
Name: 8-Pentadecanone
Formula: $C_{15}H_{30}O$
MW: 226 Exact Mass: 226.229666 CAS#: 818-23-5 NIST#: 154921 ID#: 6986 DB: replib
Other DBs: Fine, RTECS, HODOC, NIH, EINECS, IRDB
Contributor: Chemical Concepts
InChIKey: PQYGSSYFJIJDFK-UHFFFAOYSA-N Non-stereo
10 largest peaks:
57 999 | 43 422 | 58 415 | 41 393 | 127 386 |
29 274 | 71 269 | 55 172 | 27 159 | 59 154 |
Synonyms:
1.Caprylone
2.Diheptyl ketone
3.Heptyl ketone
4.Pentadecan-8-one
5.di-n-Heptyl ketone
6.8-Oxopentadecane

Experimental RI median±deviation (#data)
Standard non-polar: 1653±6 (4)
Estimated non-polar retention index (n-alkane scale):
Value: 1648 iu



8-Pentadecanona
Nº CAS: 818-23-5

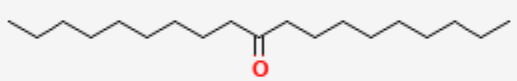
Identificación: GC-MS (QTOF) fronte biblioteca NIST



Name: 10-Nonadecanone
Formula: $C_{19}H_{38}O$
MW: 282 Exact Mass: 282.292265 CAS#: 504-57-4 NIST#: 118298 ID#: 9753 DB: mainlib
Other DBs: Fine, TSCA, HODOC, NIH, EINECS, IRDB
Contributor: NIST Mass Spectrometry Data Center, 1990.
InChIKey: YUPOCHDBBHTUBJ-UHFFFAOYSA-N Non-stereo
10 largest peaks:
43 999 | 71 961 | 155 898 | 57 654 | 41 636 |
58 601 | 85 476 | 55 449 | 29 320 | 81 283 |
Synonyms:
1.Di-n-nonyl ketone
2.Dinonyl ketone

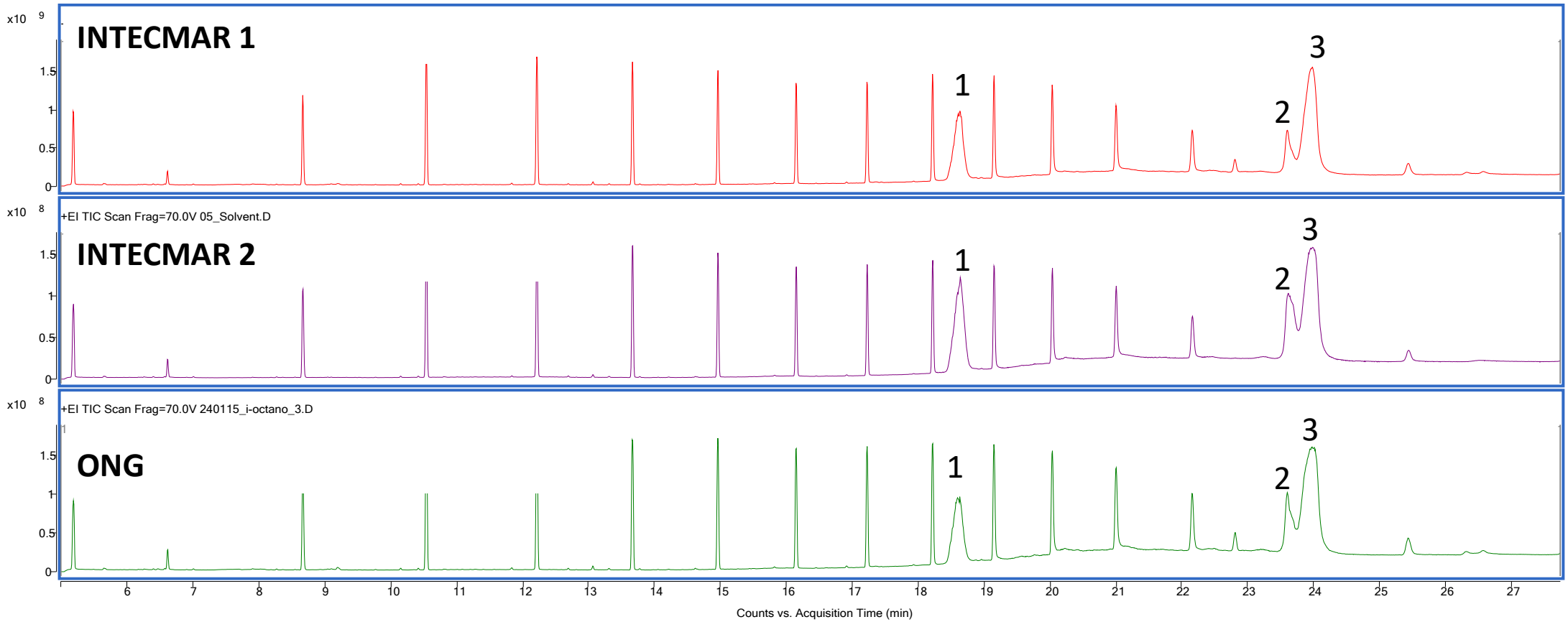
Experimental RI median±deviation (#data)
Semi-standard non-polar: 2082±N/A (1)
Standard non-polar: 2045±0 (3)
Estimated non-polar retention index (n-alkane scale):
Value: 2046 iu
Confidence interval (Ketones): 57(50%) 246(95%) iu

Retention index



10-Nonadecanona
Nº CAS: 504-57-4

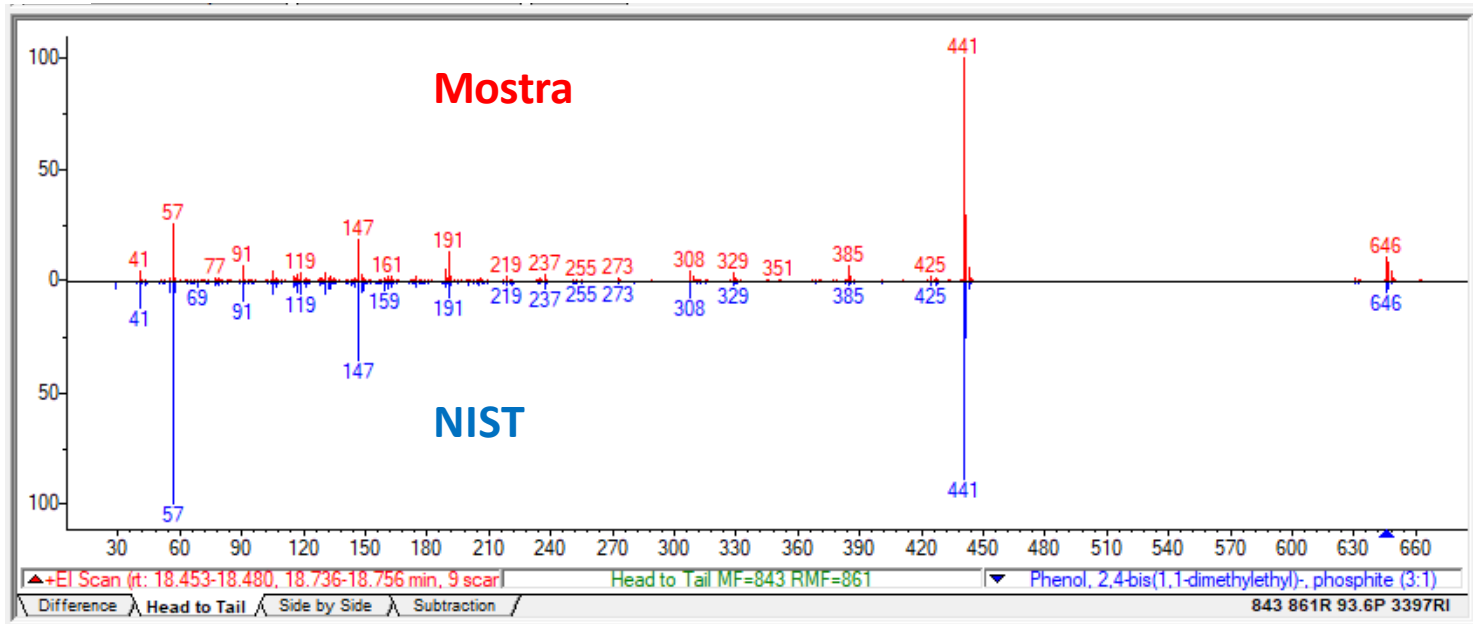
Perfís das mostras: GC-MS (QTOF) – método B



1. Irgafos 168. Confirmado contra patrón analítico.
2. Irganox 1076
3. Irgafos 168 fosfato (producto oxidación). Confirmado contra patrón analítico.

NOTA: Resto de picos: siloxanos do sistema (non proveñen da mostra)

Identificación: GC-MS (QTOF) fronte biblioteca NIST



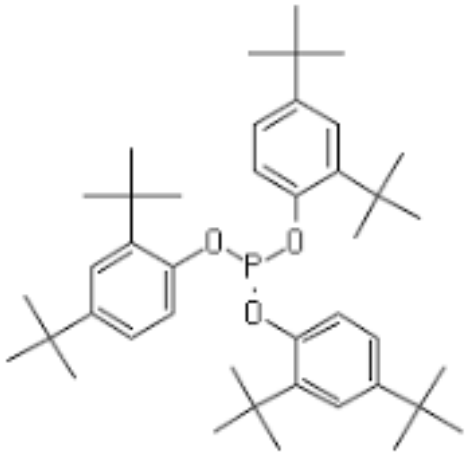
Name: Phenol, 2,4-bis(1,1-dimethylethyl)-, phosphite (3:1)
Formula: $C_{42}H_{63}O_3P$
MW: 646 Exact Mass: 646.45148 CAS#: 31570-04-4 NIST#: 384145 ID#: 29937 DB: mainlib
Other DBs: TSCA, EINECS
Contributor: NIST Mass Spectrometry Data Center, 2010
InChIKey: JKIJEFPNVSHHEI-UHFFFAOYSA-N Non-stereo
10 largest peaks:

57	999	441	875	147	349	442	247	41	116
91	84	308	67	191	65	131	52	105	49

Synonyms:
1.Alkanox 240
2.Hostanox PAR 24
3.Lowinox 242
4.Naugard 524
5.Tris-(2,4-di-t-butylphenyl) phosphite
6.Tris(2,4-di-tert-butylphenyl) phosphite
7.Trisdibutylphenyl phosphite

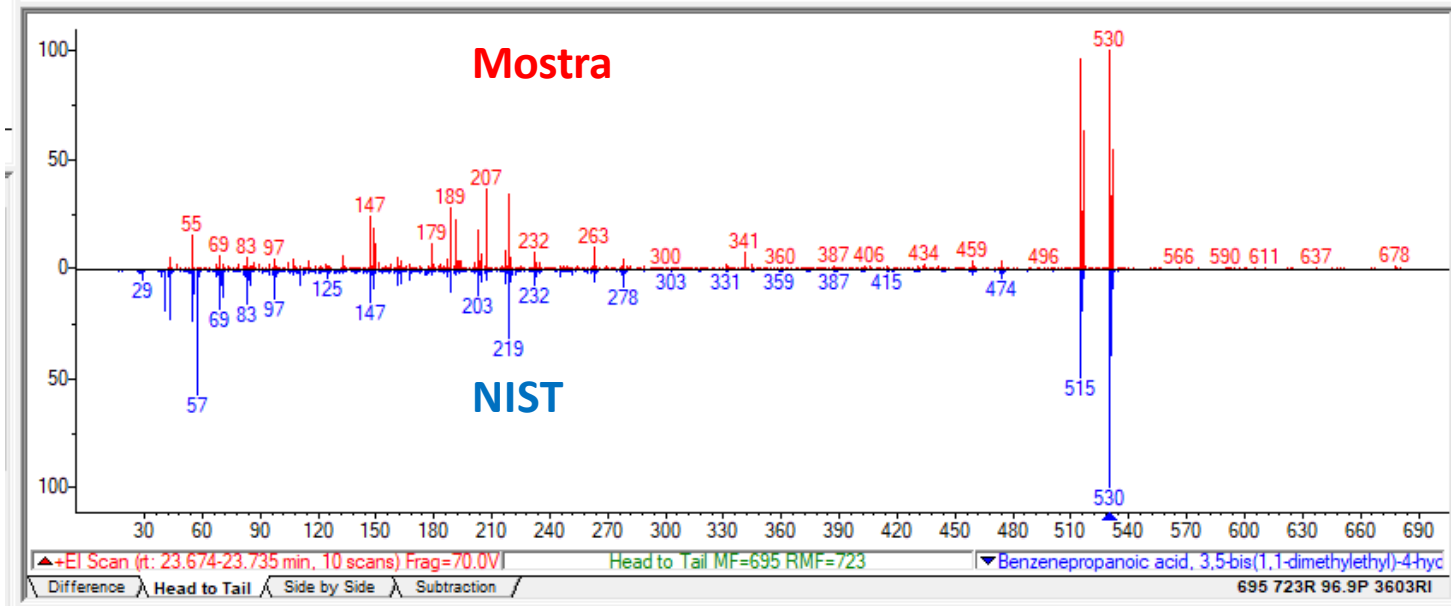
Experimental RI median±deviation (#data)
Semi-standard non-polar: 3397±N/A (1)
Estimated non-polar retention index (n-alkane scale):
Value: 4356 iu
Confidence interval (Diverse functional groups): 89(50%) 382(95%) iu

Plot/Text of Hit | Plot of Hit

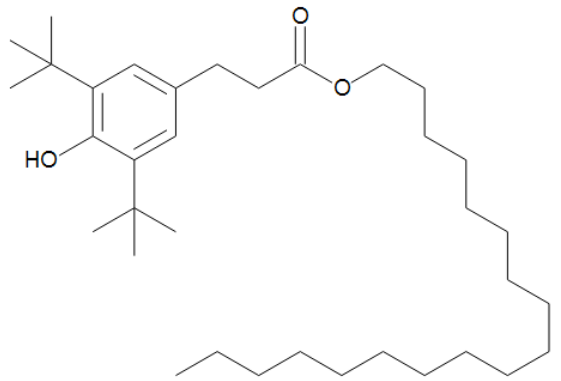


Irgafos 168
Nº CAS: 31570-04-4

Identificación: GC-MS (QTOF) fronte biblioteca NIST

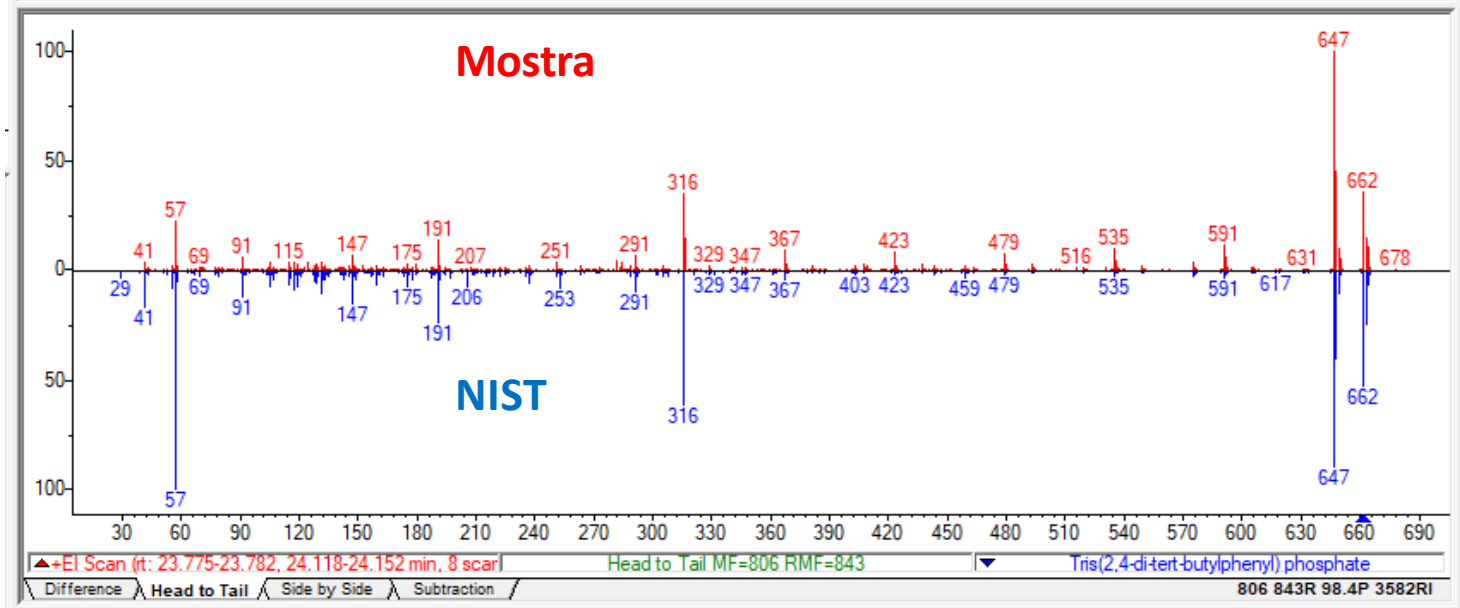


Name: Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester
Formula: $C_{35}H_{62}O_3$
MW: 530 Exact Mass: 530.469894 CAS#: 2082-79-3 NIST#: 236281 ID#: 266599 DB: mainlib
Other DBs: TSCA, EINECS
Contributor: Japan AIST/NIMC Database- Spectrum MS-IW-6347
Related CAS#: 69093-37-4; 156511-59-0; 109265-64-7; 80693-11-4; 119764-08-8
InChIKey: SSDSCDGVMJFTEQ-UHFFFAOYSA-N Non-stereo
10 largest peaks:
530 999 | 57 566 | 515 488 | 531 383 | 219 308 |
55 231 | 43 221 | 516 183 | 41 181 | 69 174 |
Synonyms:
1.2,6-Di-tert-butyl-4-[(2-octadecyloxycarbonyl)ethyl]phenol
2.3,5-Bis(1,1-dimethylethyl)-4-hydroxybenzenepropanoic acid octadecyl ester
3.Anox PP 18
4.Irganox 1076
5.Lowinox PO35
6.Naugard 76
7.Octadecyl 3-(3,5-di-t-butyl-4-hydroxyphenyl) propionate
8.Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate
9.Octadecyl-3(3',5'-di-t-butyl-4'-hydroxyphenyl)propionate
10.Octadecyl-3,5-di-tert-butyl-4-hydroxyhydrocinnamate
11.Octadecyl 3,5-di-t-butyl-4-hydroxyhydrocinnamate
12.Ralox 530
Plot/Text of Hit | Plot of Hit



Irganox 1076
Nº CAS: 2082-79-3

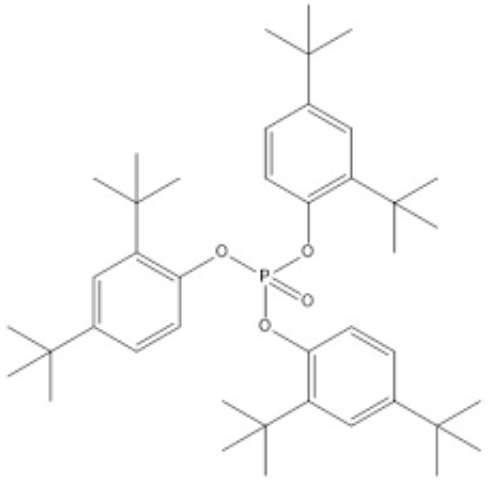
Identificación: GC-MS (QTOF) fronte biblioteca NIST



Name: Tris(2,4-di-tert-butylphenyl) phosphate
Formula: $C_{42}H_{63}O_4P$
MW: 662 Exact Mass: 662.446396 CAS#: 95906-11-9 NIST#: 414679 ID#: 29955 DB: mainlib
Other DBs: None
Contributor: NIST Mass Spectrometry Data Center
InChIKey: AZSKHRTUXHLAHS-UHFFFAOYSA-N Non-stereo
10 largest peaks:
57 999 | 647 888 | 316 604 | 662 516 | 648 393 |
663 238 | 191 227 | 41 162 | 147 143 | 91 110 |
Synonyms:
1.Phenol, 2,4-bis(1,1-dimethylethyl)-, 1,1',1"-phosphate
2.Phenol, 2,4-bis(1,1-dimethylethyl)-, phosphate (3:1)
3.D 16-834

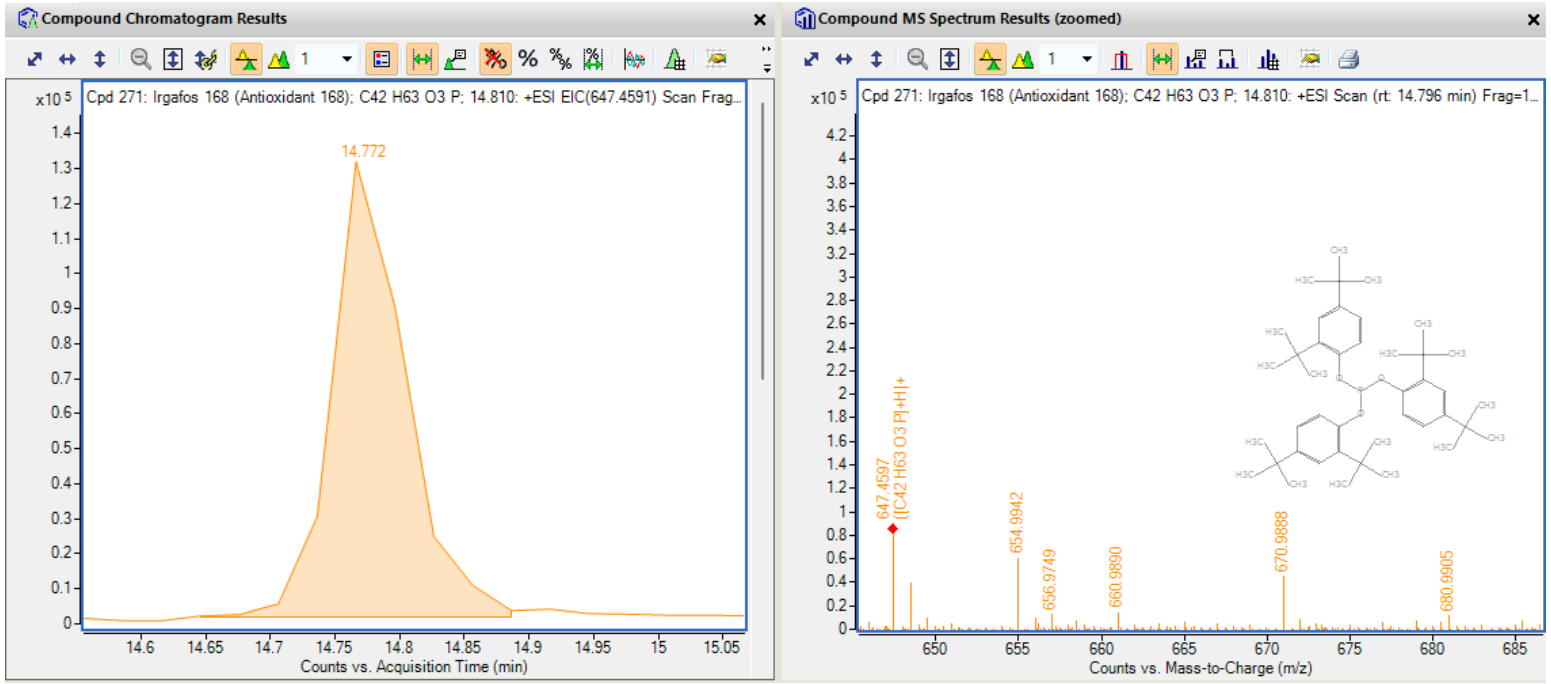
Experimental RI median±deviation (#data)
Semi-standard non-polar: 3582±N/A (1)
Retention index.
1. Value: 3582.3 iu
Column Type: Capillary
Column Class: Semi-standard non-polar
Active Phase: HP-5MS
Column Length: 30 m
Carrier Gas: He

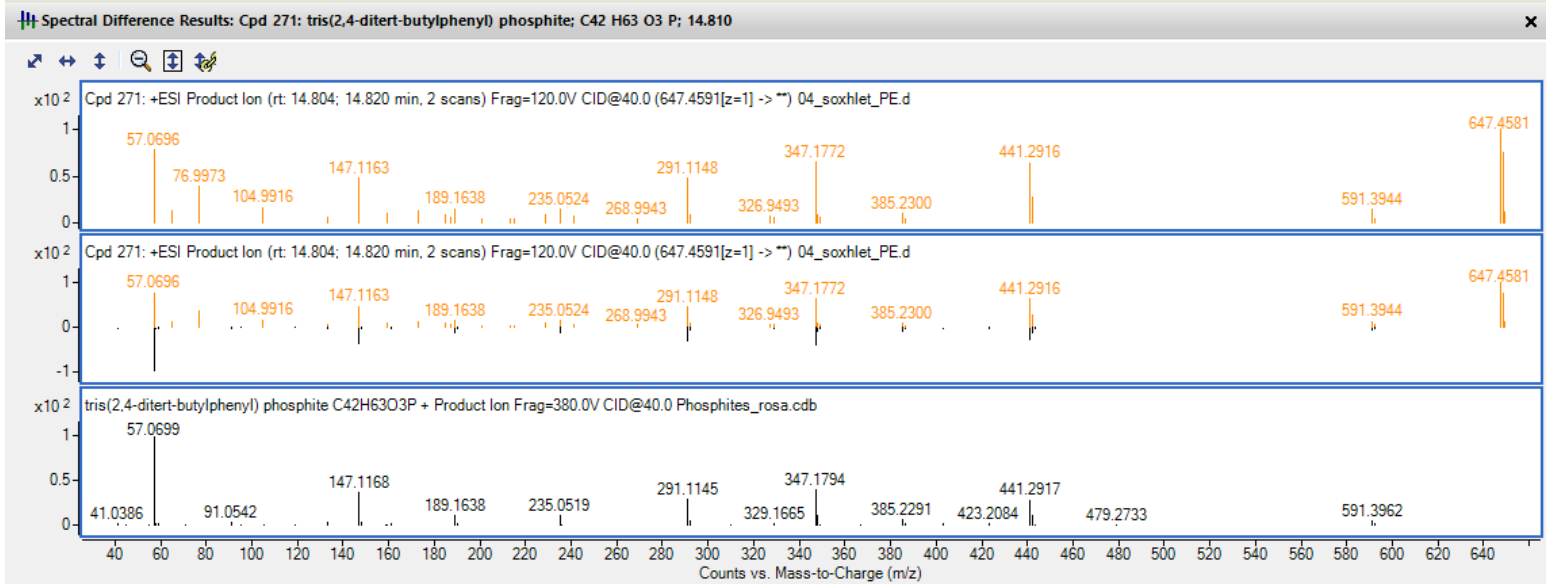
Plot/Text of Hit | Plot of Hit



Irgafos 168 fosfato
Nº CAS: 995906-11-9

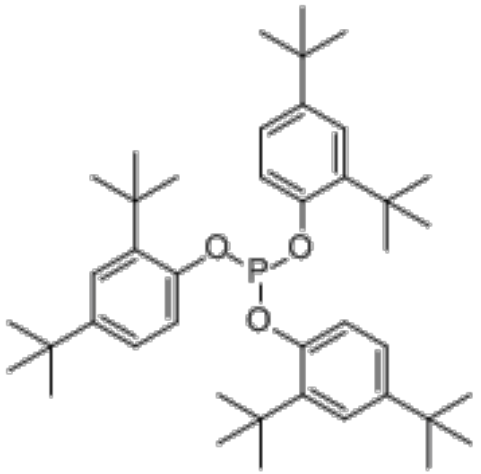
Identificación: LC-MS (QTOF) fronte biblioteca Agilent





Mostra

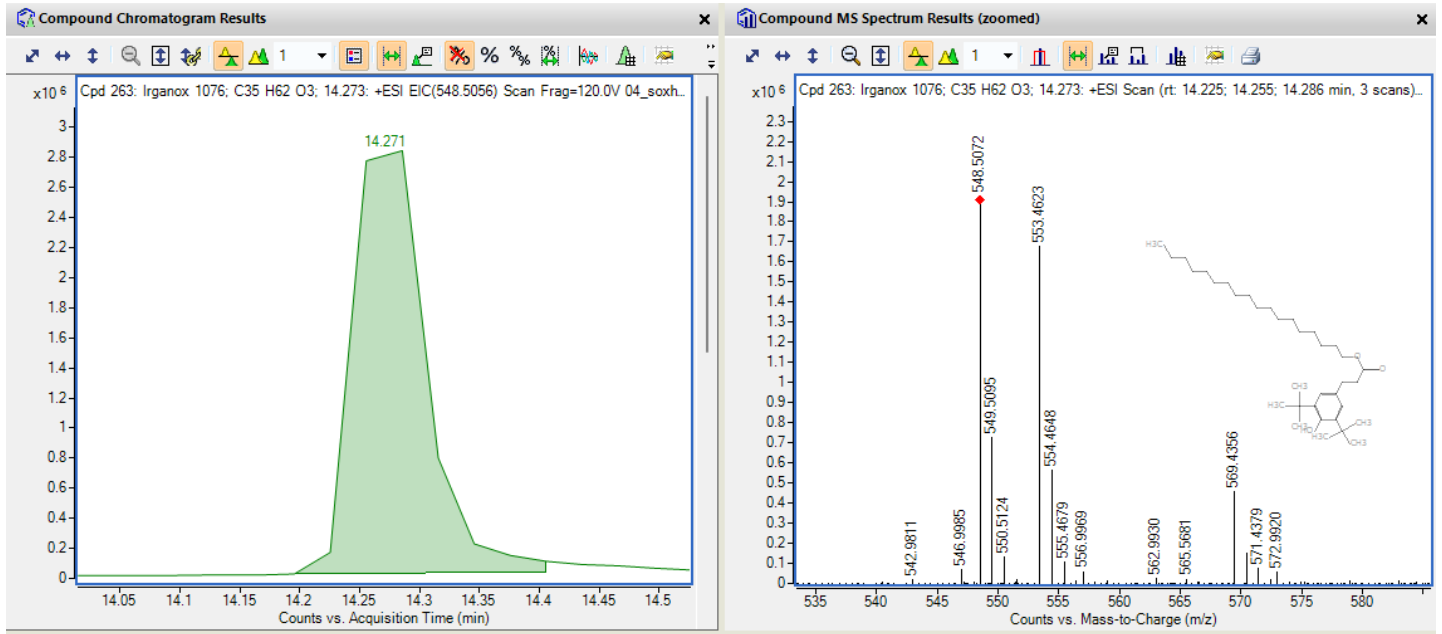
Librería

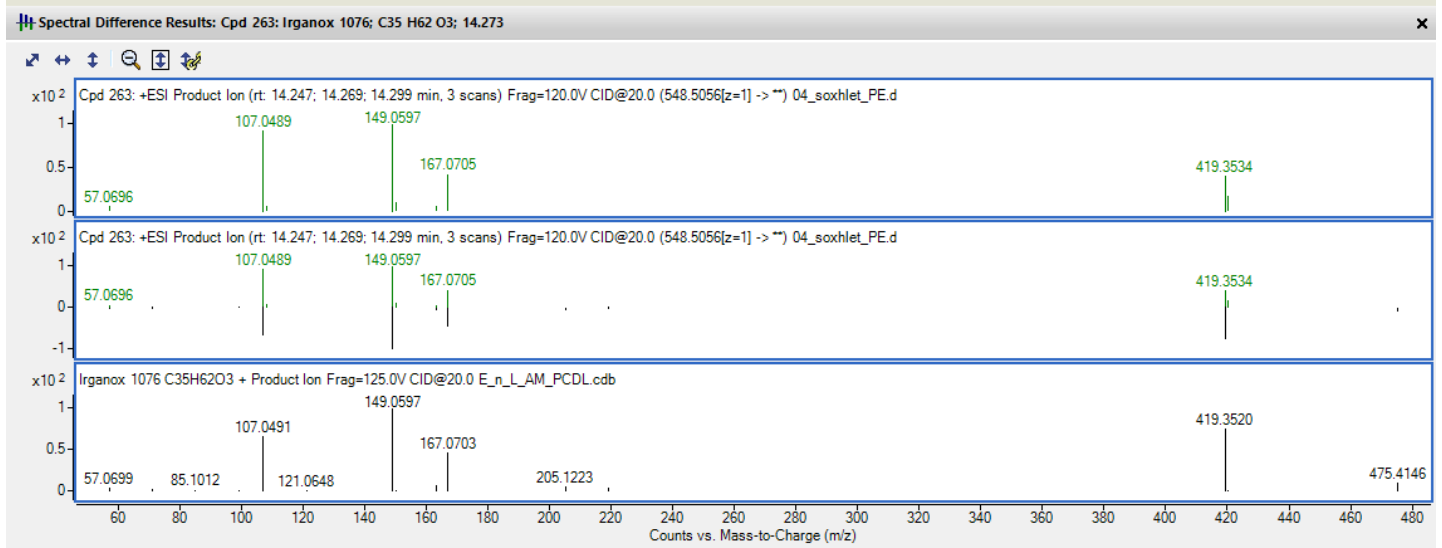


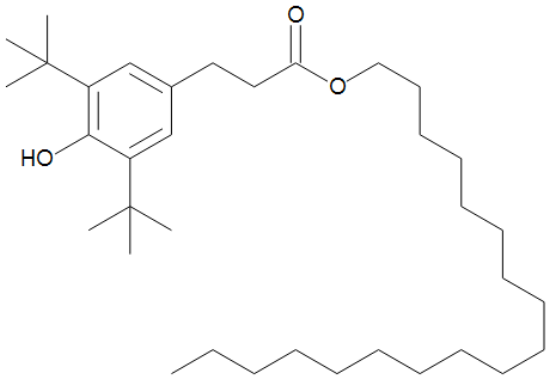
Irgafos 168

Nº CAS: 31570-04-4

Identificación: LC-MS (QTOF) fronte biblioteca Agilent





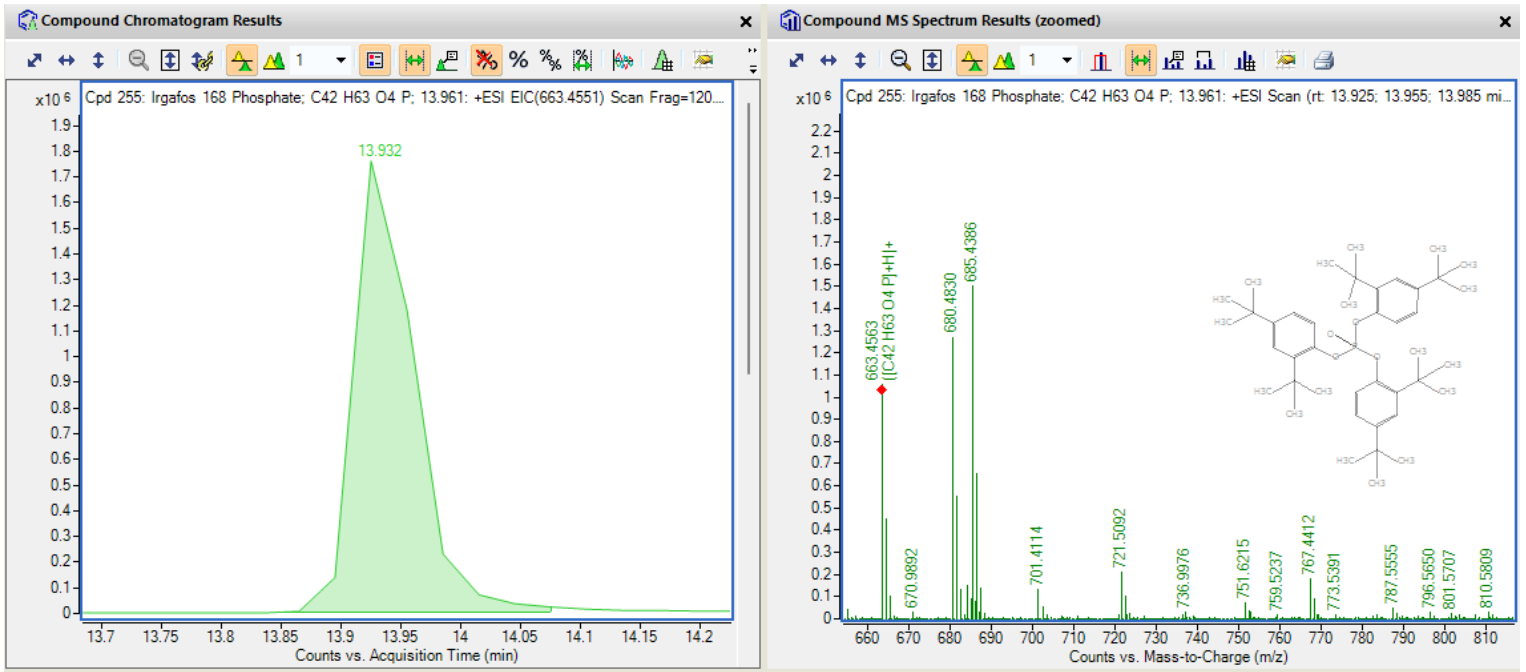


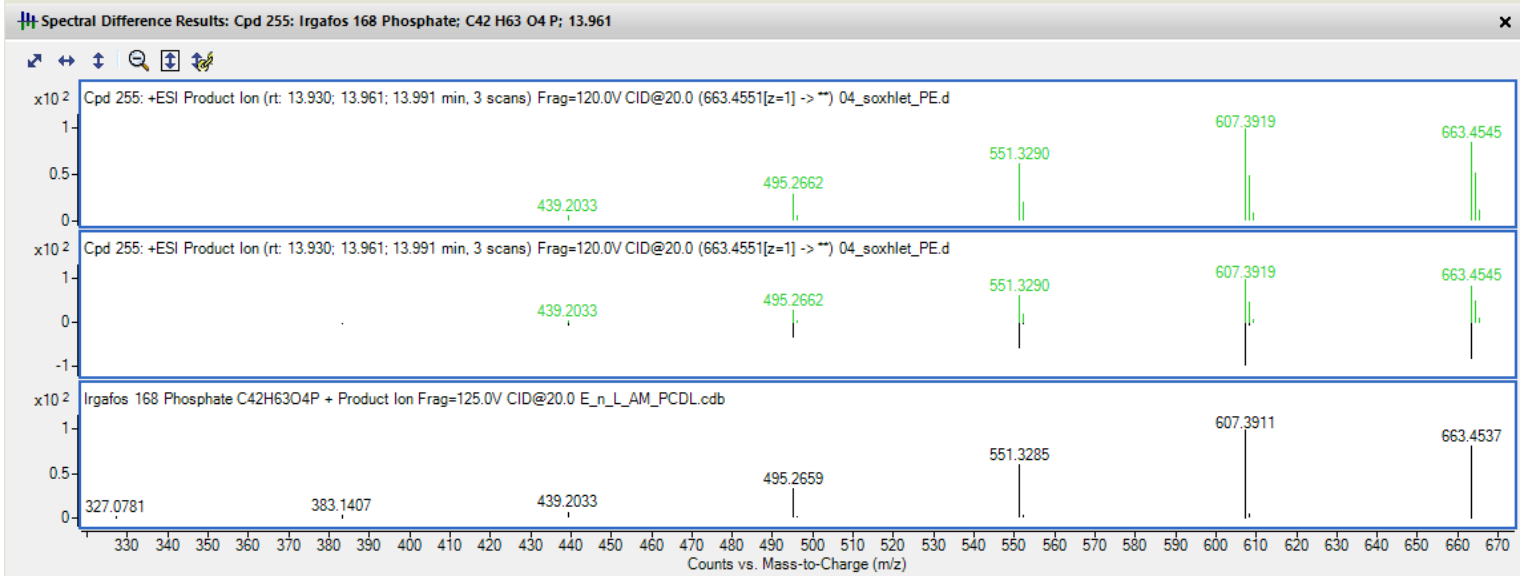
Mostra

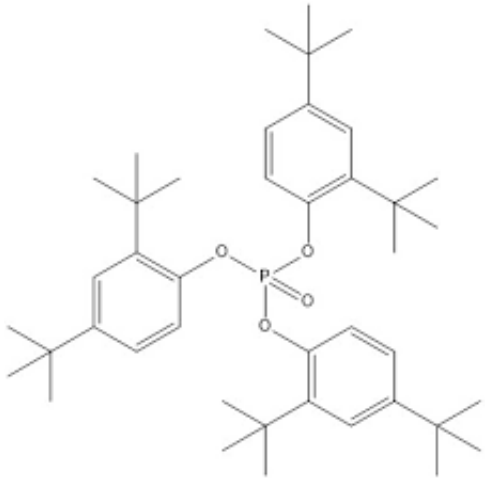
Librería

Irganox 1076
Nº CAS: 2082-79-3

Identificación: LC-MS (QTOF) fronte biblioteca Agilent





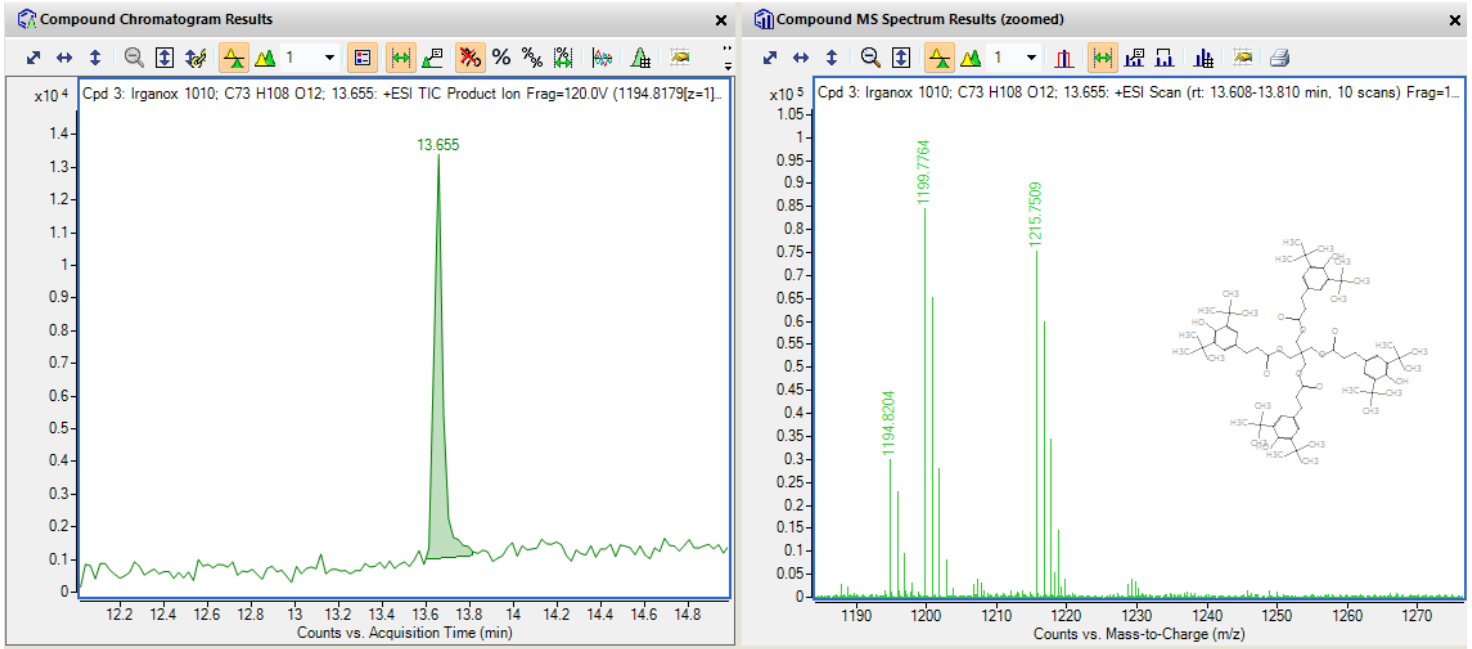


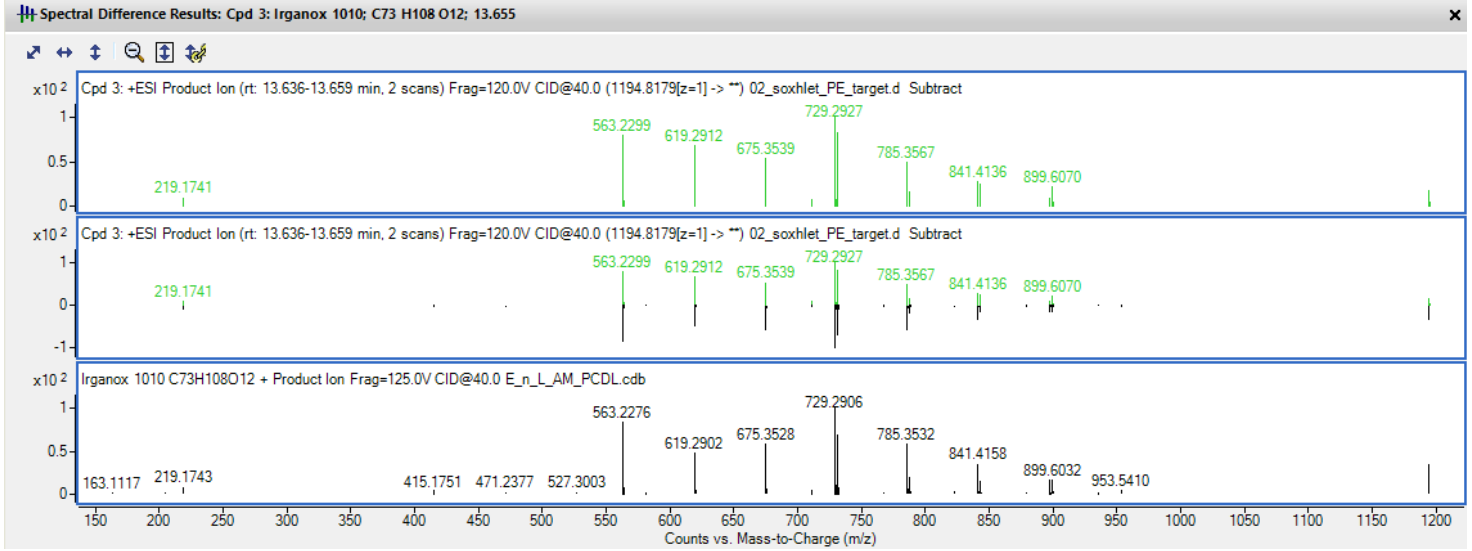
Mostra

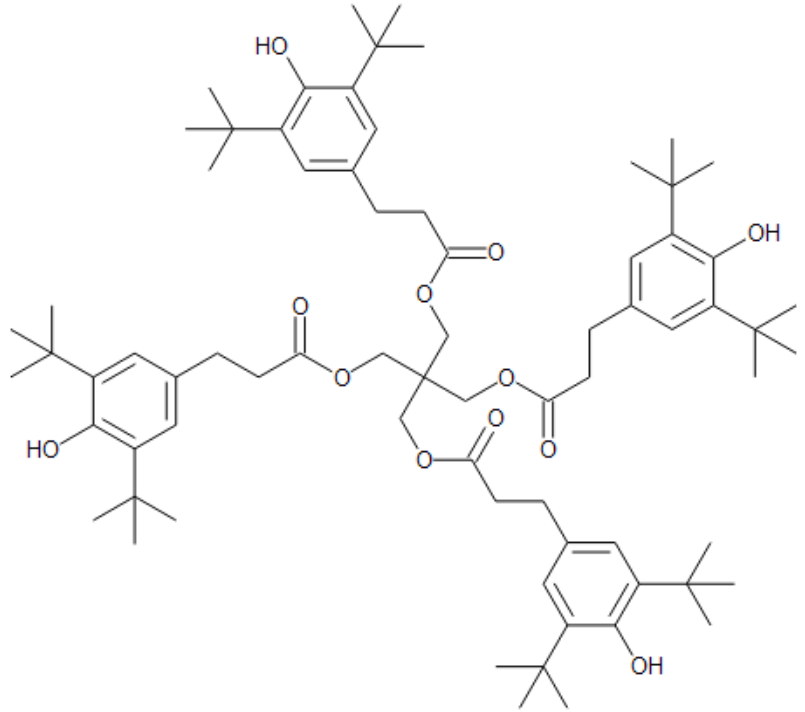
Irgafos 168 fosfato
Nº CAS: 995906-11-9

Librería

Identificación: LC-MS (QTOF) fronte biblioteca Agilent







Mostra

Irganox 1010
Nº CAS: 2082-79-3

Librería