ITAKA

Collaborative Project

FP7 – 308807

D3.1ASTM Proposal to modify D7566

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With contributions from: [names of contributors (partner)]

Project title:	Initiative Towards sustAinable Kerosene for Aviation
Deliverable nature:	Report (R)
Dissemination level: (Confidentiality)	Restricted to group (RE) or programme (PP) ¦
Start date of the project	1st November 2012
Duration	36 months
Contractual delivery date:	<dd/mm/yyyy>
Actual delivery date:	17/03/2013
Status:	*Submitted*
Contractual:	*Yes/No*
Version:	*0.0*
Total number of pages:	9
WP number:	3
Leader of WP:	SkyNRG (11)
Lead Beneficiary of deliverable:	SkyNRG (11)
Comments:	
Keywords:	ASTM D7566/proposal for change/

This project has received funding from the European Union's Seventh Programme for research, technological development and demonstration under grant agreement No 308807

Executive summary (can be used for dissemination purposes)

During the sustainable jet fuel production and blending processes SkyNRG has encountered a number of issues regarding the D7566 specification for HEFA renewable jet fuel. Those issues make the operational process and supply chain for sustainable jet fuel less efficient. With this report to ASTM we want to address the issues with the aim to improve this specification, creating a more efficient supply chain for this jet fuel.

The issues are all related to table A2.2 in D7566: '*Other Detailed Requirements; SPK from Hydroprocessed Esters and Fatty Acids'*. The proposed changes to this table are:

1. Aromatics content: Cancel maximum aromatics content in table 2.2 of D7566.
2. D2425 Analysis method for Hydrocarbon Composition: Find and allow alternative method
3. D5291 Analysis method for Carbon and Hydrogen: Find and allow alternative method
4. Metals content (trace metals): Instead of individual limits per metal, use overall maximum of 25 ppm
5. UOP 389 analysis method for Metals: Allow alternative method D7111

Each of these issues and proposals is explained in detail in the report and also in the presentation (see Annex)

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SkyNRG
Sky Energy: The Fuel Future

REQUEST TO MODIFY JET FUEL SPECIFICATION D7566

To	ASTM sub-committee D02.J.06 (Emerging Turbine Fuels)
From	Bart Rosendaal (SkyNRG)
CC	SkyNRG ops team
Date	November 25th, 2013
Reference	SKYNRG-TP251113

SkyNRG has been founded in 2010 to grow into the new market of renewable jet fuels. After the publication of ASTM D7566-11 specification including the new HEFA Jet Fuel, SkyNRG has grown into the world market leader in supply of HEFA renewable jet fuel to the global aviation industry. To date, SkyNRG has supplied to more than 25 airlines and aviation companies on 5 continents and has gained a large operational experience in working with the new ASTM D7566 specification for HEFA jet fuel.

Context

The Joint Venture SkyNRG has been founded in 2010 by KLM Royal Dutch Airlines, oil company Argos and consultancy firm Spring with primary goal to create the market for Renewable jet fuel and become a leading player in this novel sector. After the issuing of ASTM D7566-11 specification for HEFA renewable jet fuel in July 2011, we immediately started to source, blend and distribute the fuel to various airlines. These include a.o. KLM, Boeing, Airbus, Air France, Finnair, Alaska Airlines, LAN Chile, Thai airways and Qantas.

Over the past years we have gathered operational experience regarding purchasing, blending, transport and airport distribution of this new jet fuel. In doing so, we have encountered a number of issues regarding the D7566 specification for HEFA renewable jet fuel that we hereby want to address within ASTM with the aim to improve this specification, creating a more efficient supply chain for this jet fuel.

The issues are all related to table A2.2 in D7566: '*Other Detailed Requirements; SPK from Hydroprocessed Esters and Fatty Acids*'. The proposed changes to this table are:

1. Aromatics content: Cancel maximum aromatics content in table 2.2 of D7566..
2. D2425 Analysis method for Hydrocarbon Composition: Find and allow alternative method
3. D5291 Analysis method for Carbon and Hydrogen: Cancel and replace by oxygen content
4. Metals content (trace metals): Instead of individual limits per metal, use overall maximum of 25 ppm
5. UOP 389 analysis method for Metals: Allow alternative method D7111

Each of these issues and proposals will now be explained in detail in the following chapters.

1. AROMATICS CONTENT

Proposal 1. Cancel the maximum limit of aromatics in table 2.2 of D7566.

Aromatics are an important component of jet fuel as many gaskets and seals in an airplane fuel system require aromatics to swell sufficiently to become tight and leak-free. For D1655 jet fuels from conventional sources (mineral oil based) this has never been an issue as they will always contains aromatics due to the nature and composition of the originating crude oils. As a result, the D1655 specification only specifies a maximum aromatics content (25 vol-%) and not a minimum content.

This has changed with the touch-down of Synthetic Paraffinic Kerosene (SPK) jet fuels which are created through Fisher-Tropsch (FT) or Hydrotreated Esters and Faty Acids (HEFA) pathways and theoretically consist of only paraffins and iso-paraffins and no aromatics. As aromatics are required for a well-functioning jet fuel, the D7566 specification therefore requires blending with more than 50% standard D1655 jet fuel to raise the aromatics content a minimum of 8 vol.-%.

The newly added annex 2 to D7566 opened the possibility to produce SPK from biomass resulting in renewable jet fuel instead of fossil jet fuel. These low-carbon fuels have been of specific interest to airlines to both reduce their GHG-emissions and to reduce future dependency on fossil fuels. Reasons why, contrary to FT jet fuels which have not seen significant deployment, airlines around the world decided to start using these new HEFA renewable jet fuels. The HEFA process is entirely different than the FT-process, yet resulting jet fuel is quite similar and the specifications for FT jet fuel and HEFA jet fuel are almost identical. As SkyNRG has never dealt with FT jet fuels (which were the first SPK to be authorized under D7566) we have no experience with the aromatics content (if any) of these fuels.

However, for HEFA derived jet fuels we have noticed a slight hint of aromatics to be always present due inevitable hydrocarbon ring-closing reactions in the catalytic section. Especially if the catalyst is ageing and approaching end-of-life, aromatics content can increase to levels exceeding those specified in D7566 for pure SPK. We have seen aromatics contents of neat SPK as high as 1.4 vol.-% while all other parameters were within specification. Removal of aromatics in relatively small batches without affecting other parameters (such as trace metal content) is not only very difficult, but also very costly and time-consuming.

The current maximum limit of 0.5 vol.-% in the D7566 specification for pure SPK seems to conflict with aromatics being indispensable for jet fuel, reflected in the minimum limit of 8 vol.-% of aromatics in table 1 of D7566 for the blended final jet fuel. From a technical point of view, any aromatics present in renewable jet fuel should add to the overall final specification of minimum 8 vol.-% for the blended fuel and therefore we hereby propose to cancel the maximum limit of aromatics content in table 2.2. of D7566.

2. D2425 ANALYSIS METHOD FOR HYDROCARBON COMPOSITION

Proposal 2. Find and allow alternative method for D2425.

Specification D7566 requires to analyse the hydrocarbon composition for both the pure FT as the pure HEFA jet fuel to determine the amount of paraffins, cycloparaffins and aromatics. This method is very uncommon and results in operational difficulties as almost no independent surveyor has equipment installed that is required for this method, nor experience with the method. Currently all (global) D2425 analyses of SkyNRG have been done by Southwest Research institute (SWRI) in Texas and they have 2 machines running. Global surveyors such as SGS, Intertek and Inspectorate have all stated to us that they do not have the capability for this test in-house and all refer to SWRI. We have seen analysis times up to 14 days (!) before results were communicated, at considerable cost. These response times are far too long, especially when producing renewable jet fuel and the produced quality has to be verified within a certain period to be able to intervene.

Furthermore, as renewable jet fuel production and sale continues to grow, this (extremely) limited availability of analysis capability greatly threatens the supply chain. We have had the situation where 1 of the 2 machines had to be repaired, further delaying analysis results.

From our discussions with SWRI and Intertek regarding the use of D2425 and how to interpret the results, we think that D2425 is not a precise method and thus not suitable for this application. See also SWRI comments in annex 1.

As this parameter (hydrocarbon composition) is not part of the standard D1655 jet fuel specification, the impact of the hydrocarbon composition (apart from the aromatics content) on the technical performance of jet fuel and fit-for-use is probably not significant and analysis is done primarily to verify that the product indeed is SPK.

Given the non-performance related background to require the hydrocarbon composition in table 2.2., the very limited availability of the prescribed method D2425 and the question if this method is suitable to give, SkyNRG proposes to review alternative acceptable methods and allow at least one other method to determine the hydrocarbon composition, such as UV or HPLC.

3. D5291 ANALYSIS METHOD FOR CARBON AND HYDROGEN

Proposal 3. Cancel the Carbon+Hydrogen method D5291 and replace by Oxygen content.

Specification D7566 requires for pure HEFA in table 2.2 to determine the carbon + hydrogen (C+H) content using method D5291. The requirement for C+H content is more than 99.5 mass-%, while the accuracy of the method is ±3.5 mass-% according our surveyor Intertek. Clearly, it is impossible to determine without doubt a level of more than 96.5 mass-% C-H content, yet the specification requires a level of >99.5 mass-%.

In annex 2 an Intertek analysis is shown of our HEFA (which was off-spec on aromatics) and the Carbon+Hydrogen has been determined twice. First analysis yielded 98.05 and the second yielded 99.3%. Yet the total of aromatics, paraffins and cyclo-paraffins in both these tests was exactly 100%.

As can be seen, the variation in resulting C+H values from identical batches is (much) too large compared to the required minimum, implying time-consuming and costly repetitive analysis to obtain values that comply with table 2.2. See also the comment of Intertek surveyors (annex 3) regarding the use of this method. Based on our experience and Interteks statement, we state that the method D5291 cannot be used for the intended purpose and has to be removed from the D7566 specification.

Furthermore, if the aim of the C+H parameter is to verify that no oxygen is present in the HEFA jet fuel, it would be more logical to determine the oxygen content directly. For example by using methods common for gasoline, such as (taken from the European gasoline specification EN-228) EN-238, EN-12177 or EN-ISO-22854.

Therefore SkyNRG proposes to cancel the Carbon + Hydrogen analysis using D5291 and replace this with a suitable method to determine the oxygen content in renewable HEFA jet fuel.

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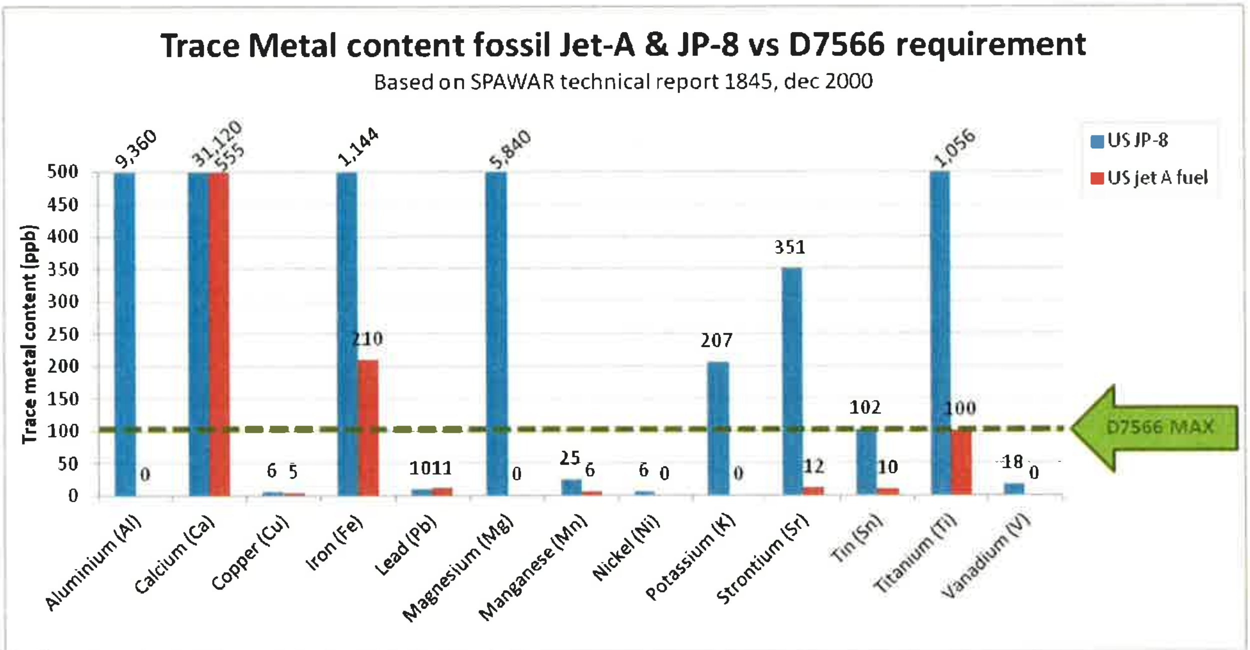
4. METALS CONTENT (TRACE METALS)

Proposal 4. Instead of individual limits per metal, use overall maximum of 25 ppm.

Contrary to standard D1655 jet fuel, specification D7566 table 2.2 requires to determine trace metal contents of 21 metals plus phosphor using method UOP-389, whereby each may not exceed 0.1 ppm-wt. This is extremely tight and in order to obtain good results it is crucial to use ASTM sampling guidelines in combination with a laboratory familiar with the UOP-389 analysis method. And even then, our experience shows that off-spec readings can occur easily without tangible cause. This might be related to the fact that the required maximum levels are close to or beyond the accuracy level of this method. See annex x for a statement from Intertek on this. Obviously, parameters cannot be limited on a level beyond analysis accuracy.

Furthermore, the reason to determine these metals has to be questioned, especially given the fact that standard D1655 jet fuel is not required to do these analysis. In technical report 1845 of SPAWAR several jet fuels were tested for a.o. metal content and revealed that Jet-A, JP-5 and JP-8 can have metal contents far exceeding the required levels for HEFA. (SPAWAR report: http://www.dtic.mil/cgi-bin/GetTRDoc?Location=U2&doc=GetTRDoc.pdf&AD=ADA396143)

Below graph shows the results of this SPAWAR report:



Graph 1: Trace metal contents fossil Jet-A and JP-8 vs. D7566 standard for HEFA renewable jet fuel. Y-axis has been cut to 500 ppb maximum.

As can be seen from above graph, standard fossil jet fuel contains much higher level of trace metals when compared to the D7566 specification for HEFA renewable jet fuel. As the HEFA jet fuel is required to be blended with at least 50 vol.-% standard D1655 jet fuel prior to use as aviation fuel, the very stringent levels required for pure HEFA in table 2.2. can be questioned. To comply with customer requirements demanding Jet A-1 quality, SkyNRG has used JP-8 jet fuel in the US (instead of Jet A) as fossil jet fuel to blend with HEFA.

The huge difference in metal content between HEFA jet fuel and standard jet fuel in combination with the level of accuracy of method UOP-389 justify our proposal to change from reporting individual metals to an overall value of 25 ppm-wt.

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5. UOP-389 METHOD FOR METALS CONTENT (TRACE METALS)

Proposal 5. Allow ASTM D7111 as alternative analysis method for UOP-389.

Contrary to standard D1655 jet fuel, specification D7566 table 2.2 requires to determine trace metal contents of 21 metals plus phosphor using method UOP-389. This is also a very uncommon method and many laboratories are not equipped for using this method. As a result, samples have to travel to specialized laboratories, adding analysis time and costs. Response times cannot be too long, especially when producing renewable jet fuel which has to be verified within a certain period to be able to intervene.

The above in combination with the fact that metal content in HEFA jet fuel is far lower than standard jet (see graph 1) supports our proposal to allow alternative method ASTM D7111 for the determination of metals in HEFA jet fuel.

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ANNEX 1

EMAIL INTERTEK BELGIUM FEBRUARY 20th, 2013 INCLUDING SWRI COMMENTS AND REPORT

Bart Rosendaal

Van: Kurt Tyssen Intertek [Kurt.Tyssen@intertek.com]
Verzonden: Wednesday, February 20, 2013 3:12 PM
Aan: Bart Rosendaal
CC: Eline Schapers (eline@skynrg.com)
Onderwerp: FW: Analysis request from Intertek Belgium
Bijlagen: Investigation of Synthetic Jet fuel 021813.pdf

Bart,

Zie in bijlage nog extra informatie met betrekking tot de ASTM 2425 (aromaat analyse) op het eerste staal.
Gisteren is het nieuwe genomen staal richting SWRI vertrokken voor recheck analyse.
We houden jullie op de hoogte.

Mvg,Kurt

-----Original Message-----
From: Childress, Kenneth H. [mailto:kenneth.childress@swri.org]
Sent: Tuesday, February 19, 2013 2:34 AM
To: Kurt Tyssen Intertek
Cc: Patrick Van Den Berge Intertek
Subject: RE: Analysis request from Intertek Belgium

Hello, attached is some more information about the D2425 analysis of the sample.

The pdf file consist of an overview page, a page about the analysis of each fraction followed by graphics; and a page of some extra work performed on the polar fraction followed by graphics.

I just hope this doesn't confuse the issue more than it is.

From our point of view, this sample does contain more aromatics than we have seen with other synthetic jet fuels over the last several years.

Anyways, please review the attachment.

Please, any questions or comments are encourage.

Ken
-----Original Message-----
From: Kurt Tyssen Intertek [mailto:Kurt.Tyssen@intertek.com]
Sent: Monday, February 18, 2013 9:26 AM
To: Childress, Kenneth H.
Cc: Patrick Van Den Berge Intertek
Subject: FW: Analysis request from Intertek Belgium
Importance: High

Ken,

We reviewed the data together with our client and indeed our client is expecting a max of 0.5% aromatics in the synthetic jet fuel. So your result is our of spec!
Comparing to our data from a similar production plant with same type of feedstock it should not be containing any aromatics.
Also noticed that the product contains mainly alkylbenzene and no other type of aromatics? Are you sure this is alkylbenzene, could it be there has been a wrong identification of the peak?

Can you review the data once more please and let us know today your feedback.
Many thanks.

Best regards,

Kurt

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Business line Manager, Analytical Assessment
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direct : +32 3 544 10 90
mobile : +32 477 37 91 84
email : kurt.tyssen@intertek.com

www.intertek.com

-----Original Message-----
From: Childress, Kenneth H. [mailto:kenneth.childress@swri.org]
Sent: Thursday, February 14, 2013 1:09 AM
To: Peter Gysen Intertek; Kurt Tyssen Intertek; Kristof Dom Intertek
Cc: *OCA EMEA BEL Antwerp Shift Supervisor; Jacobson, Patsy; Ann Peeters Intertek
Subject: RE: Analysis request from Intertek Belgium

Hello: Attached are the D2425 results for the latest sample.

Some observations:

By the label, I suspect this is the type of sample that is generally referred to as "Synthetic Jet."

The specification for synthetic jet is for the aromatics to be <0.5%.

We are coming up with a result that is >0.5%, which is a cause of concern for us.

This sample was analyzed more than once, with similar results.

Additionally, I analyzed the sample without the pre-separation (neat), which provided us with an even higher aromatic value.

D2425 is a very "vague" analysis which is always a concern when unexpected results happen.

However, I looked into the results in more detail and found evidence that the aromatic content appears to be there. There may be a debate on exactly how much (D2425 is not a precise method (my opinion)), but the aromatic evidence I noticed is usually not present with previous synthetic jet fuels we have analyzed in the past.

If needed, I can pass that information upon request.

Of course, I am just speculating about this concern. A >0.5% value may not be unexpected.

Feel free to comment, questions are always welcomed.

Ken Childress

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Aromatics in Synthetic Jet Fuel Investigation

The appendix in ASTM D7566 references D2425 as the method to determine that the aromatic and cycloparafin content for synthetic jet fuel is within specification limits.

The aromatic requirement is <0.5%.

D2425 breaks down the sample into its hydrocarbon types (groups) but does not provide individual identifications of the compounds in the sample.

D2425 results for the submitted sample indicates the aromatic content to be >0.5%

This is an obvious concern.

D2425 analysis is unusual. It is referred to as a GCMS procedure (gas chromatography-mass spec) but it is actually just a mass spec analysis. In other words, there is no chromatography (the separation of compounds) involved.

The vaporized sample fills up a glass chamber, then the sample is introduced into the mass spec detector.

The entire sample enters the detector and the result is a "lump-a-gram" as oppose to a series of resolved peaks associated with chromatography.

We obtain a mass spec profile of the lump. Since the entire lump represents the entire sample, the profile can be obtain anywhere within the lump; however, the center of the lump is most often used.

The mass spec profile consists of a pattern of mass fragments (commonly called ions). The abundance of each mass fragment is used by the software to calculate the percentage of each hydrocarbon type.

Before analysis, the sample is to be split into its non-polar/polar fractions due to possible overlapping ions. Quite often, the non-polar are also referred to as the saturates (or paraffins) fraction and the polar is referred to as the aromatic fraction.

With synthetic fuels, the polar (aromatic) fraction is very small. Also, it usually consist of the saturates that were not collected in the non-polar fraction. With this separation, there will always be some carry-over. However, the mass spec and D2425 software will look for carry-over material in both fractions and make the adjustments for the final results.

Each fraction is individually analyzed, and the calculating software uses the abundances of each ion in both fractions to calculate the final result.

Analysis of Each Fraction

Please keep in mind all these lumps will look the same and is the result of the entire sample (fraction) entering the detector.

The mass spec is able to determine the mass fragment (ion) pattern of each lump. This ion pattern indicates the composition, in terms of hydrocarbon type, of each lump.

Page A contains two pictures. The top is the lump-a-gram of the non-polar fraction of the sample.

The lower picture is the mass fragment pattern of that same non-polar (saturates) fraction of the sample. Notice the fragments of 57, 71, and 85. These are the most prominent ions usually found with non-polar (saturates) hydrocarbon types.

Page B contains two pictures. The top is the lump-a-gram of the polar fraction of the sample.

The lower picture is the mass fragment pattern of the polar (aromatic) fraction of the sample.

This is the first indicator of the possible presence of aromatics!

Usually, with synthetic jet fuel samples, the polar fraction looks like the non-polar fraction. With the expected low amount of aromatics, all we should be seeing is mostly carry over from the saturate fraction (mainly 57,71,85).

However, with this pattern notice the other ions, particularly the ions of 91 and 105. These two ions are the prominent ions found in the "alkyl benzenes" type of polar hydrocarbons. Particularly toluene, xylene, methyl and ethyl benzenes.

Today, I found the polar/non-polar fraction from a previous synthetic jet fuel sample we ran months ago. For comparison, I ran the polar fraction of this sample and the polar fraction of our current sample back-to-back.

Page C is the polar fraction of the previous sample. Notice the same primary ions found in our non-polar sample (57,71, 85) even though this is the polar fraction. There are not any aromatics present.

Page D is a rerun of the polar fraction of our current sample. They do not look similar (due to the ions such as 91 and 105,plus many others).

The fractions in page C and D were run back to back today. I wanted to do this to show that the instrument was able to see the difference between two types of polar fractions.

```
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'perator    :
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ample Name  : OD15438   NP
isc Info    :
ial Number  : 2
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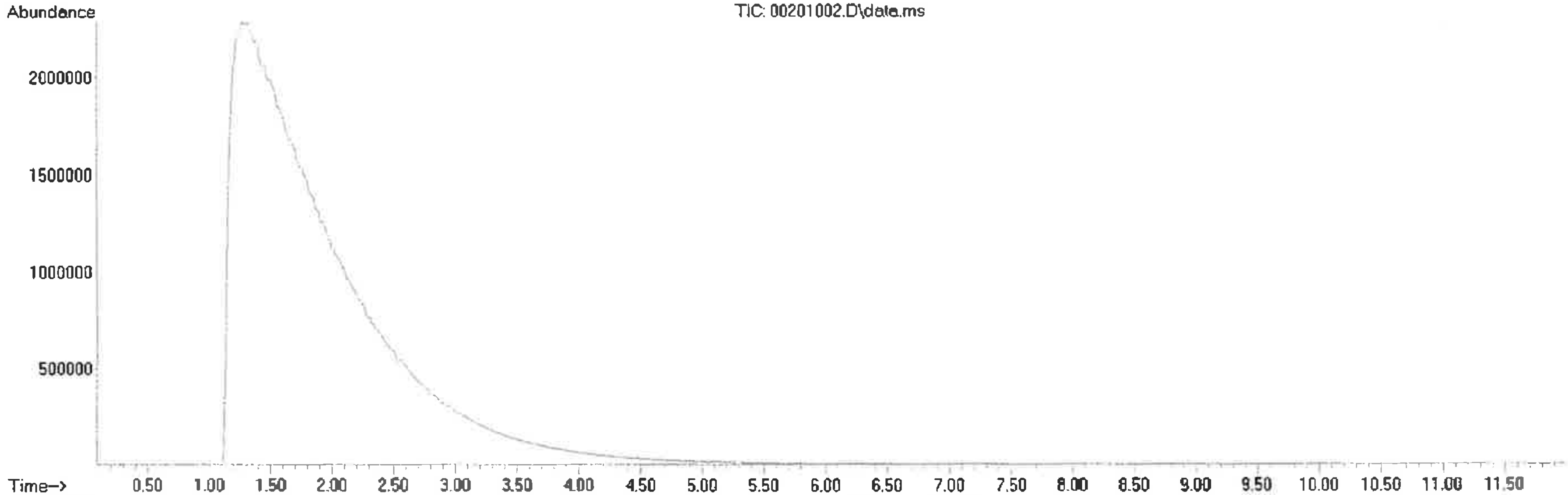
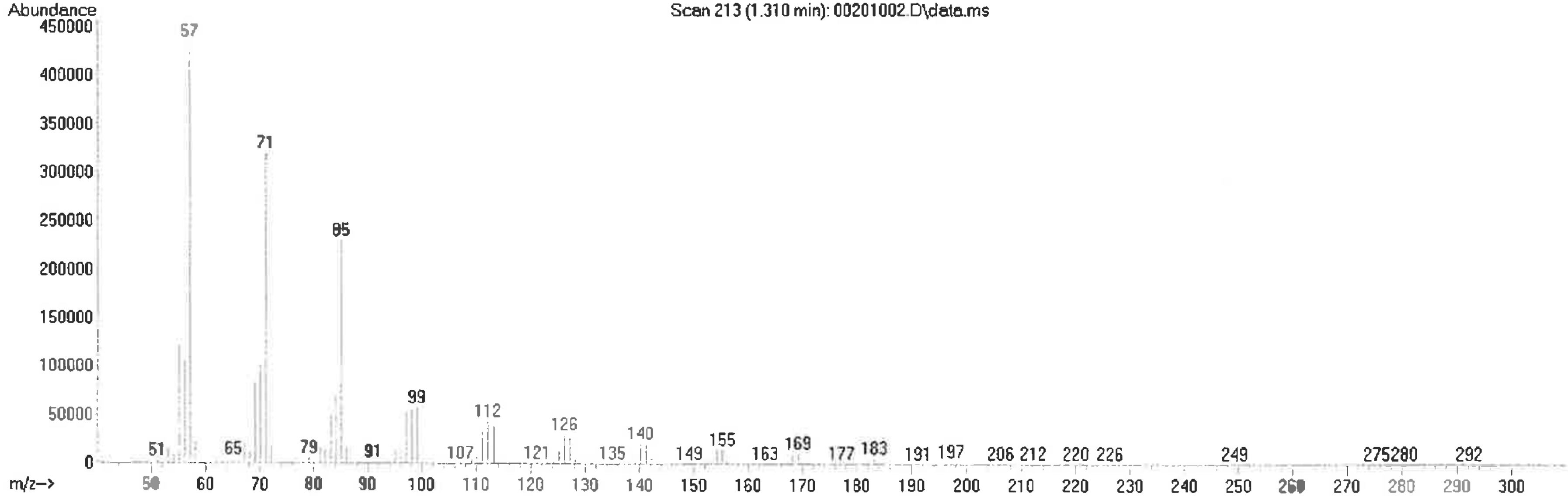
(A)





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nstrument  :      GCMS02
ample Name: OD15438B P
isc Info    :
ial Number: 3
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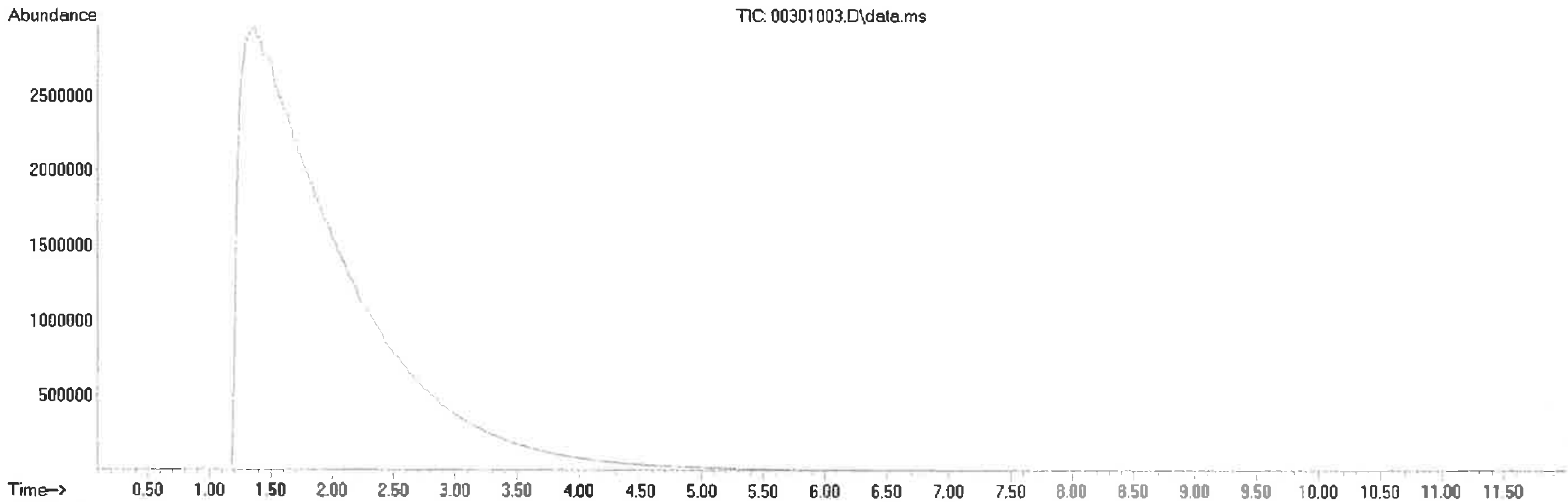
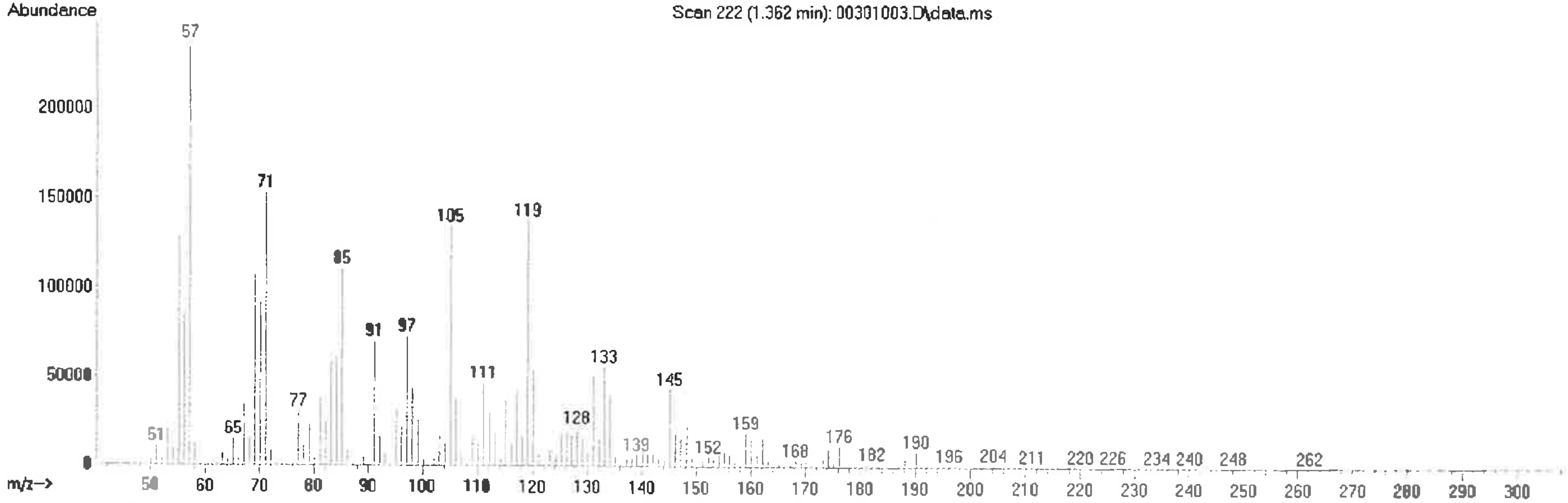
B





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nstrument   :    GCMS02
ample Name  : ODDB 6077  P  RR
isc Info    :
ial Number  : 1
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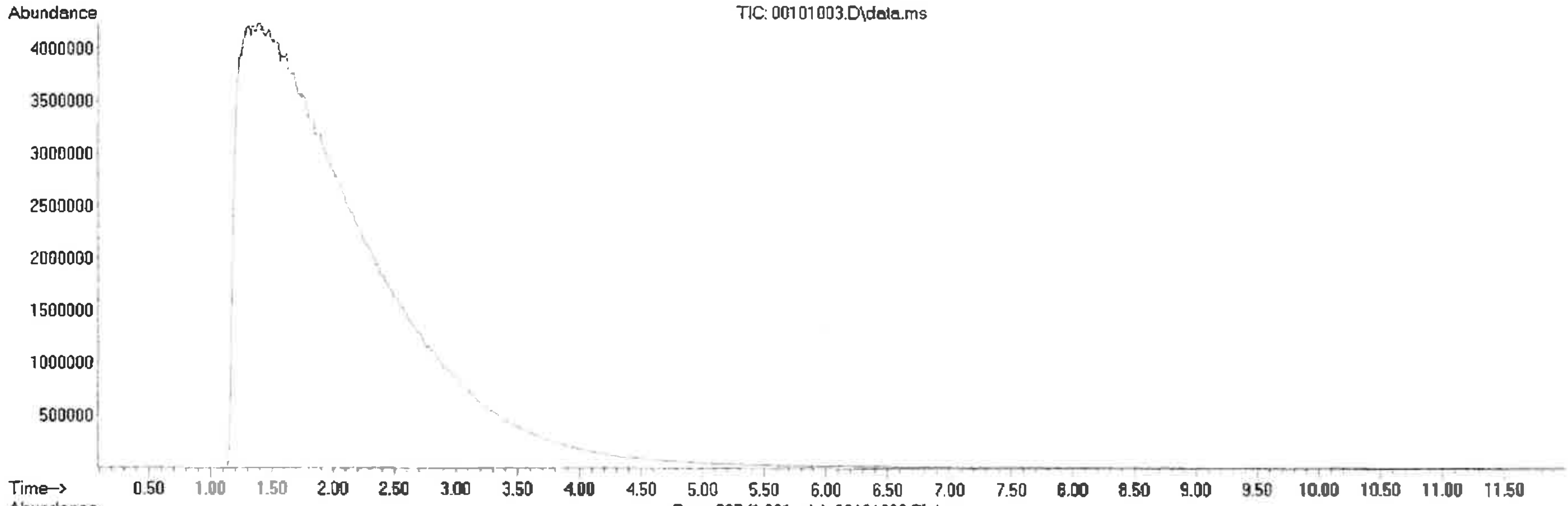
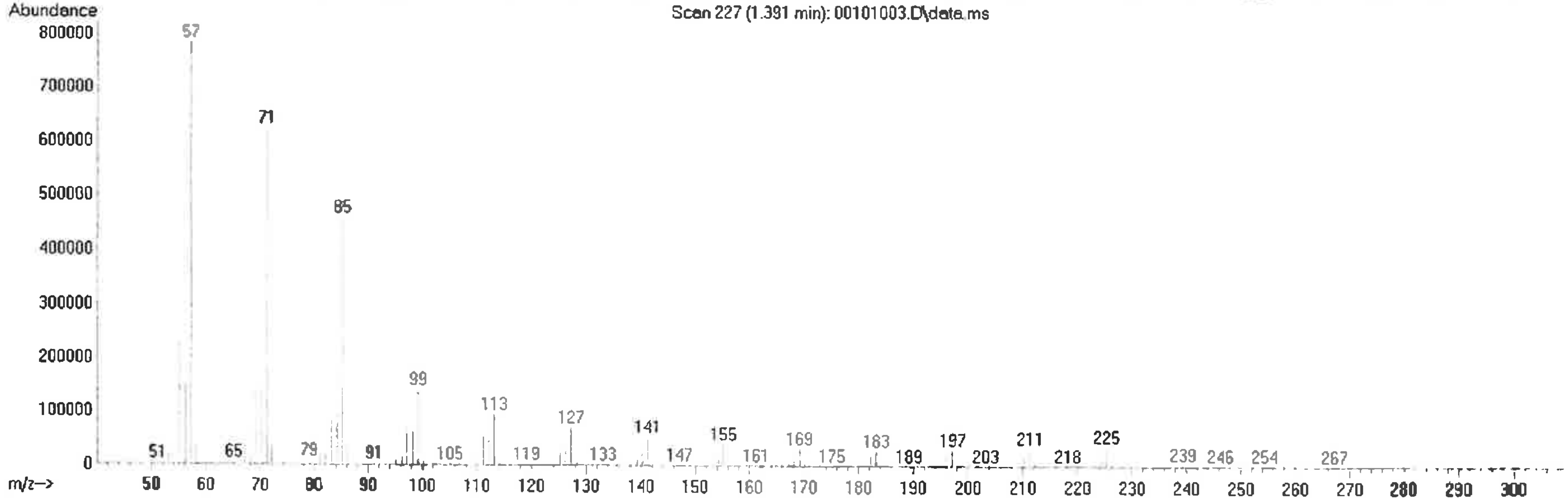
(C)





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Acquired    : 18 Feb 2013   14:02      using AcqMethod D2425M.M
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Sample Name: ODDB 15438   P   RR
Misc Info   :
Vial Number: 2
```
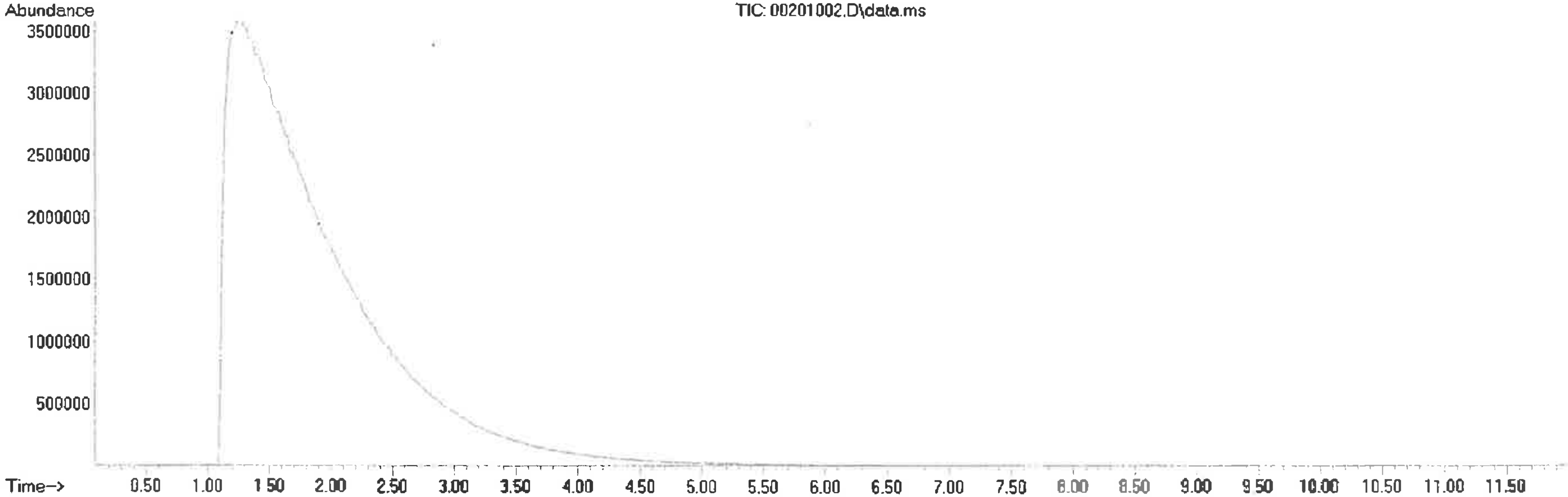
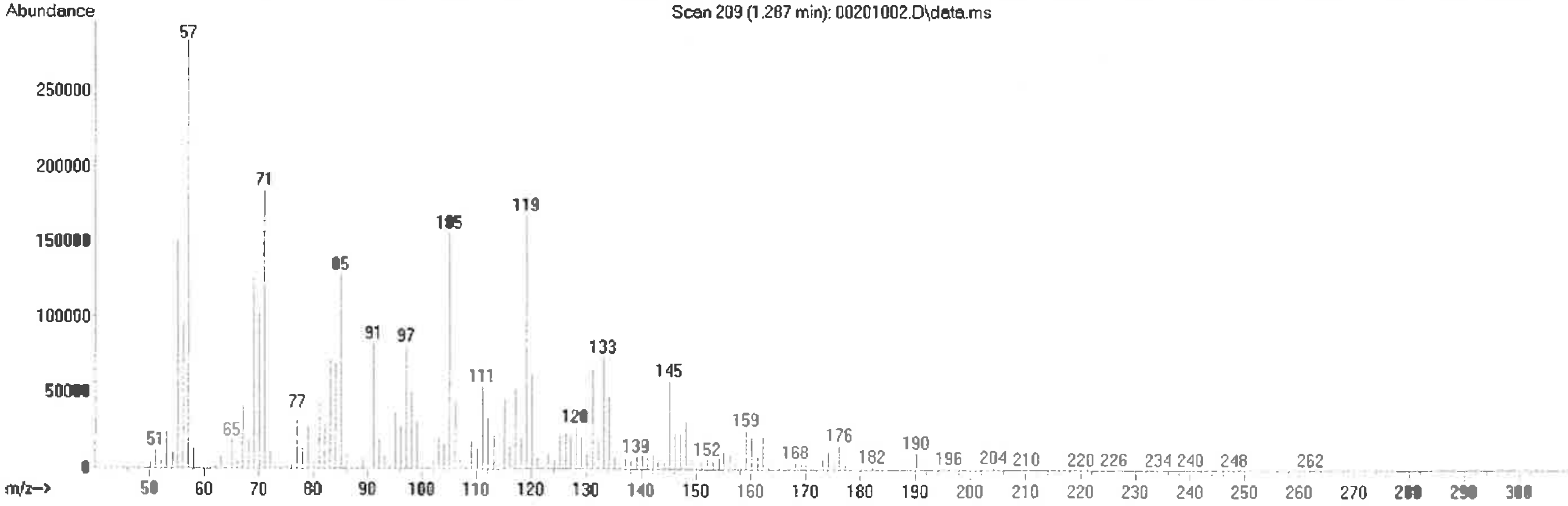

D





Analysis of the Polar Fraction

Before I released the original data, some additional work was performed.

Using another GCMS, with a typical GC column that would allow the separation of the compounds, the polar fraction was analyzed.

Page E is the chromatogram, obviously containing many peaks and most all of them non-polar (saturates). (All these peaks are present and lumped together in the D2425 lump-a-gram)

There is a technique with the software where we can look for the suspected ions of 91 and 105 (106 was also added) throughout the chromatogram. This tells us where to look for suspected aromatic material.

Page F contains two pictures. The top is a zoomed-in picture ~7 minutes into the chromatogram. The front shoulder of the indicated peak is one location where we found these ions. The bottom picture is the mass fragment pattern of that shoulder of the peak (notice the prominent ions).

The ion pattern is submitted to the library. The search matches the pattern (as best as it can) to entries in the library.

Page G are results from the mass spec library for this peak at ~7 minutes. The top picture is the pattern from our sample (the same pattern as the lower picture on page F). The next picture is Hit 1. Notice the 91 and 106 ions. The ID and match factor are highlighted. A match factor of > than 900 is indication of a very, very close match.

Page H is another example at ~12 minutes. The ion pattern for the indicated peak is in the lower box. That same pattern is the top picture on page I. The next picture is the mass speck Hit 1. Another aromatic that would be considered a "alkyl benzene."

Same with page J at ~13.8 minutes, with the mass spec ID highlighted on page K.

Page L is another time fragment from the polar fraction. The mass spec identified these 3 as additional C9 aromatics, other versions of the type of aromatic called "alkyl benzenes."

NOTE: These are not the only aromatics in this sample, just an example of a few to demonstrate the possibility that the aromatic content could be >0.5%.

Also, the expectation is that the most likely type of aromatics found in these type of fuels would be of the class noted as "alkyl benzenes." Generally referred as the more simple, less complexed, type of aromatics.

E

```
File         :D:\1\DATA\13FEB13XB\00101001.D
Operator     : KC
Acquired     : 13 Feb 2013  13:41      using AcqMethod ZEBGENM.M
Instrument   :   GCMS01
Sample Name: OBBD 15438 P frctn
Misc Info    :
Vial Number: 1
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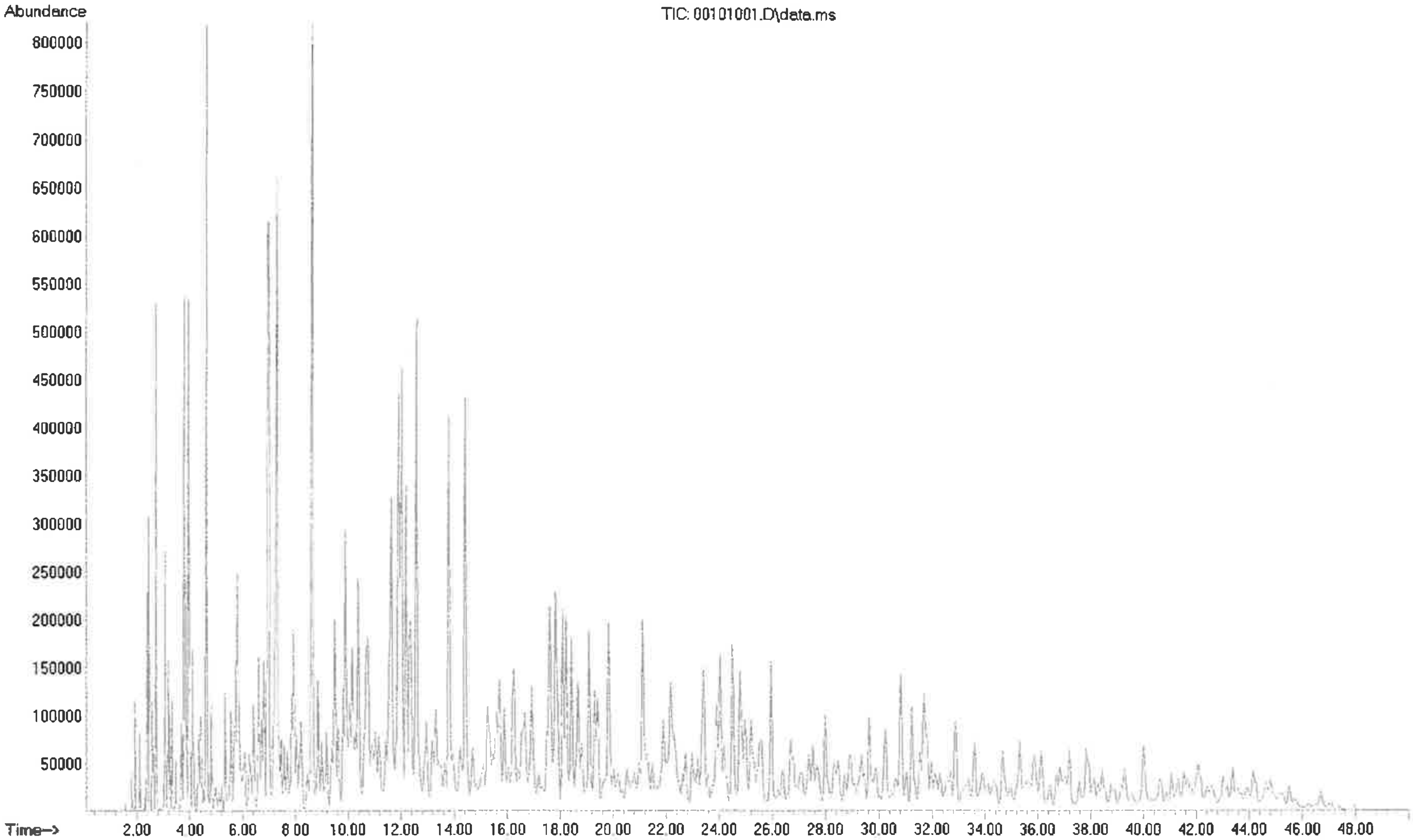

F

```
File        :D:\1\DATA\13FEB13XB\00101001.D
Operator    : KC
Acquired    : 13 Feb 2013  13:41      using AcqMethod ZEBGENM.M
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Sample Name: OBBD 15438 P frctn
Misc Info   :
Vial Number: 1
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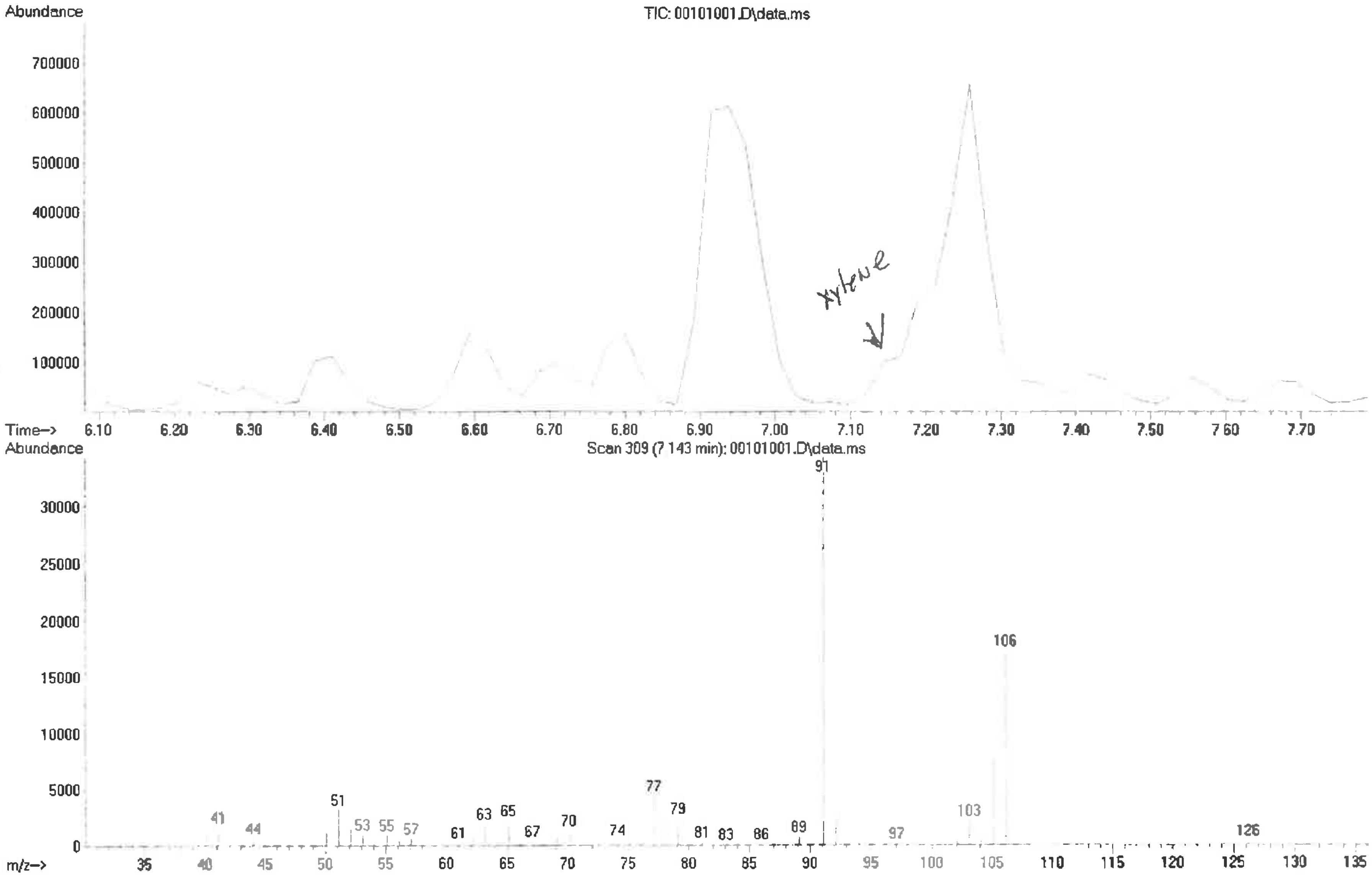

G

** Search Report Page 1 of 1 **

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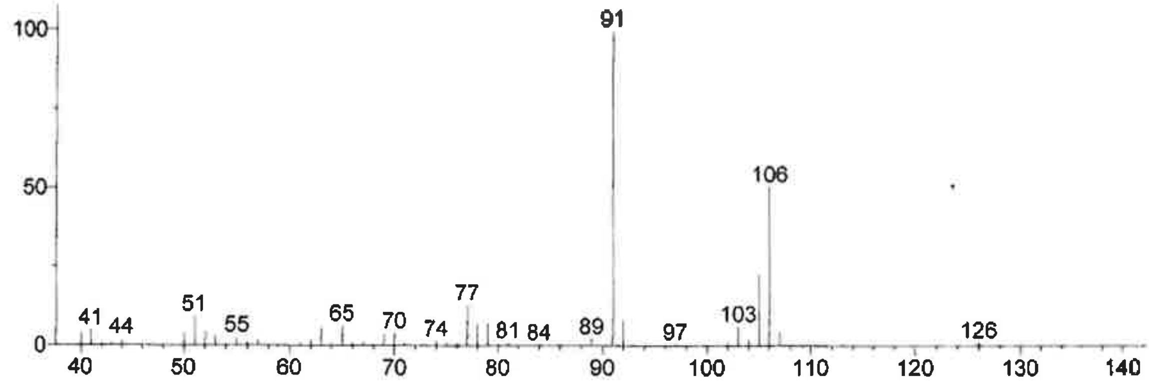
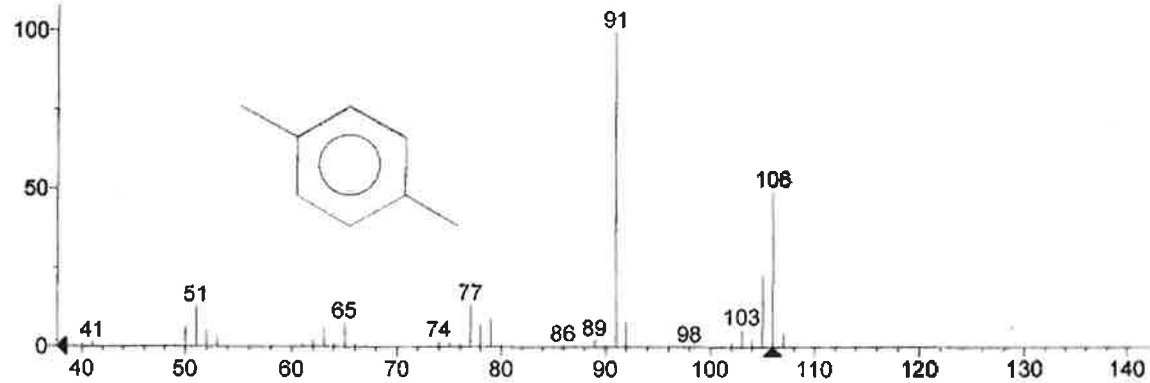
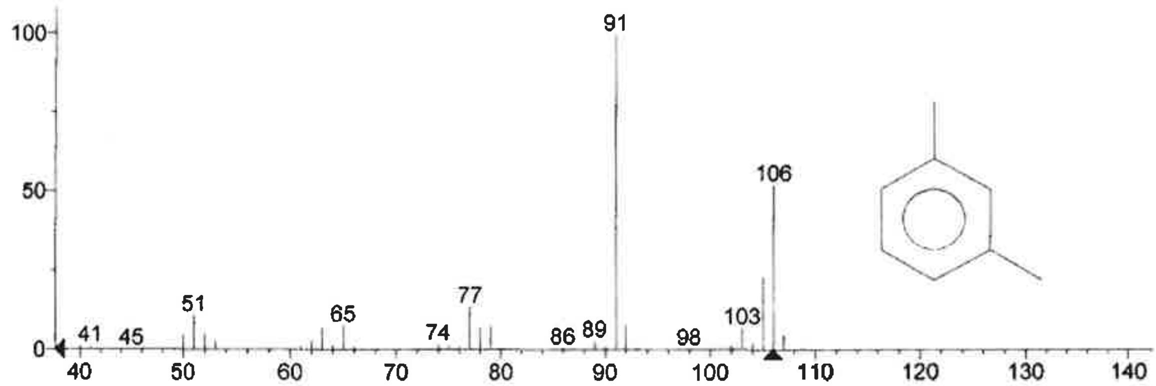
Compound in Library Factor = 138



Hit 1 : p-Xylene
C8H10; MF: 933; RMF: 967; Prob 38.3%; CAS: 106-42-3; Lib: replib; ID: 11408.

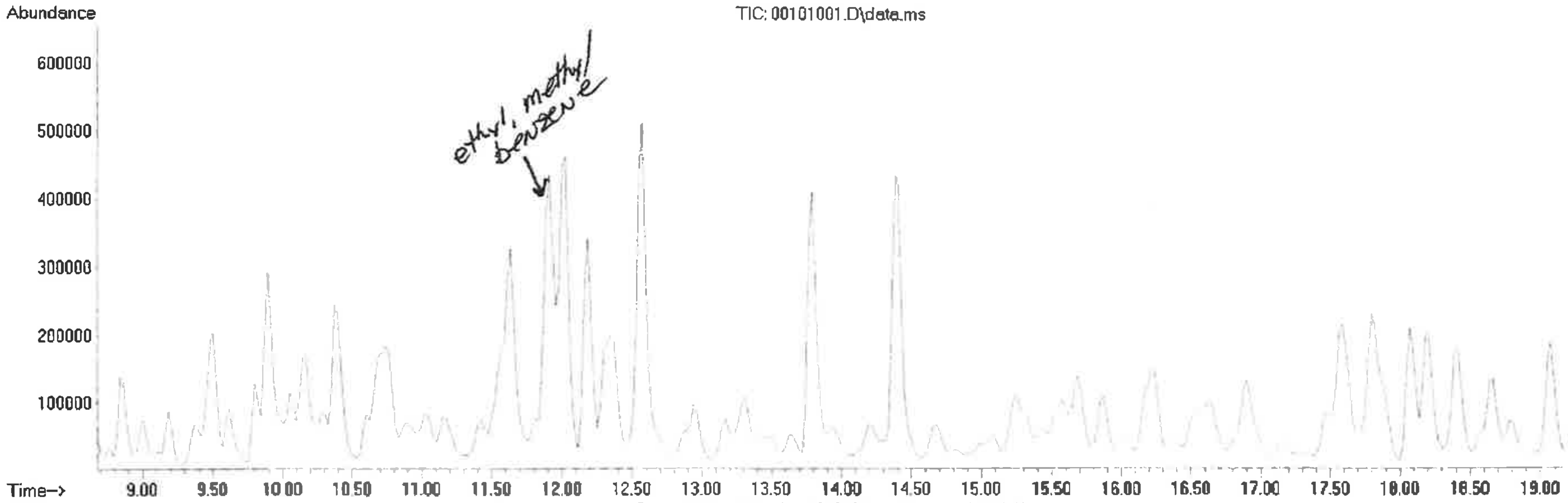


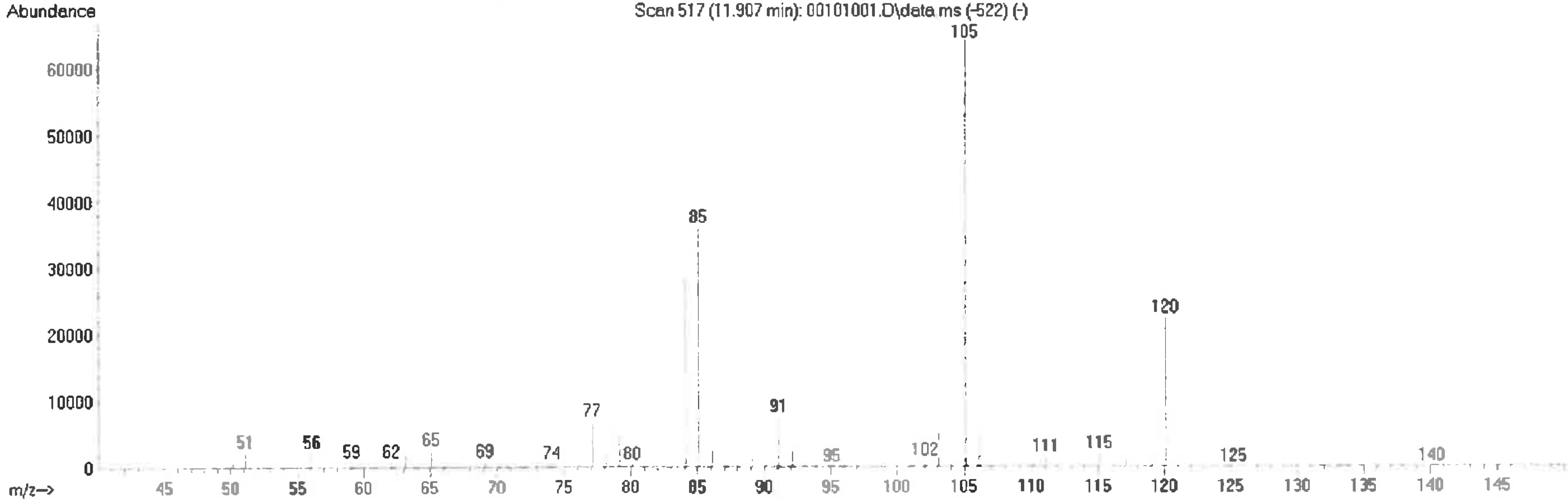
Hit 2 : Benzene, 1,3-dimethyl-
C8H10; MF: 922; RMF: 950; Prob 26.3%; CAS: 108-38-3; Lib: replib; ID: 11401.



File :D:\1\DATA\13FEB13XB\00101001.D
Operator : KC
Acquired : 13 Feb 2013 13:41 using AcqMethod ZEBGENM.M
Instrument : GCMS01
Sample Name: OBBD 15438 P frctn
Misc Info :
Vial Number: 1

(H)

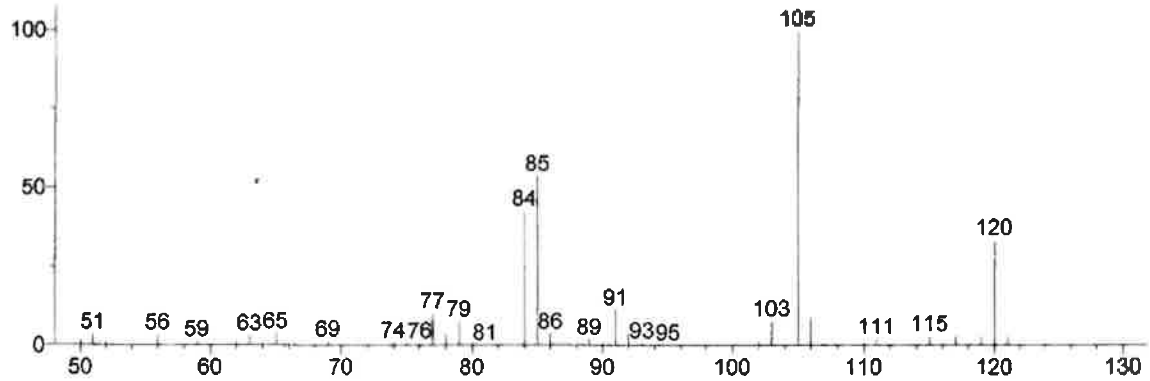




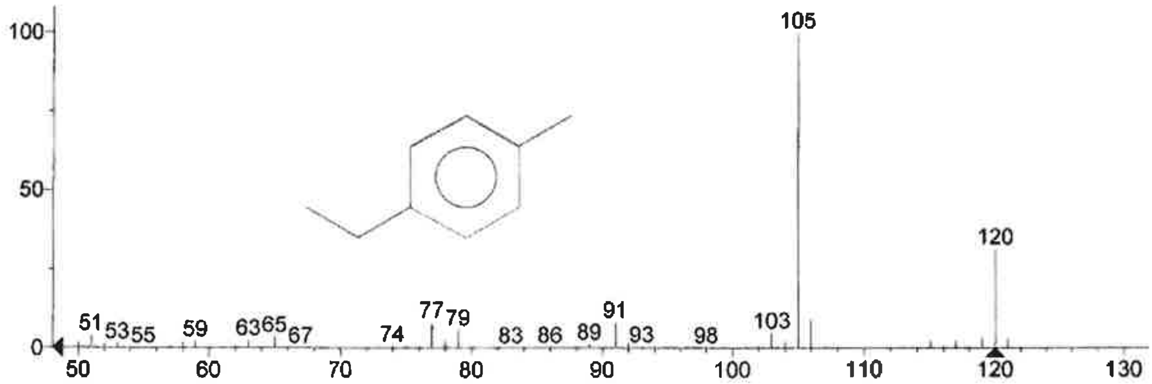
(I)

** Search Report Page 1 of 1 **

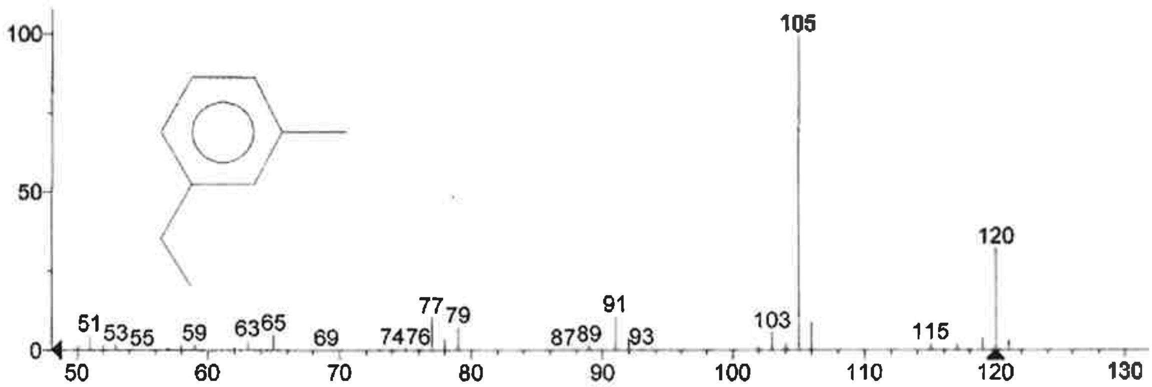
Unknown: Scan 517 (11.907 min): 00101001.D\data.ms (-522)
Compound in Library Factor = -324



Hit 1 : [redacted]
C9H12; [redacted] RMF: 806; Prob [redacted]%; CAS: 622-96-8; Lib: replib; ID: 13925.

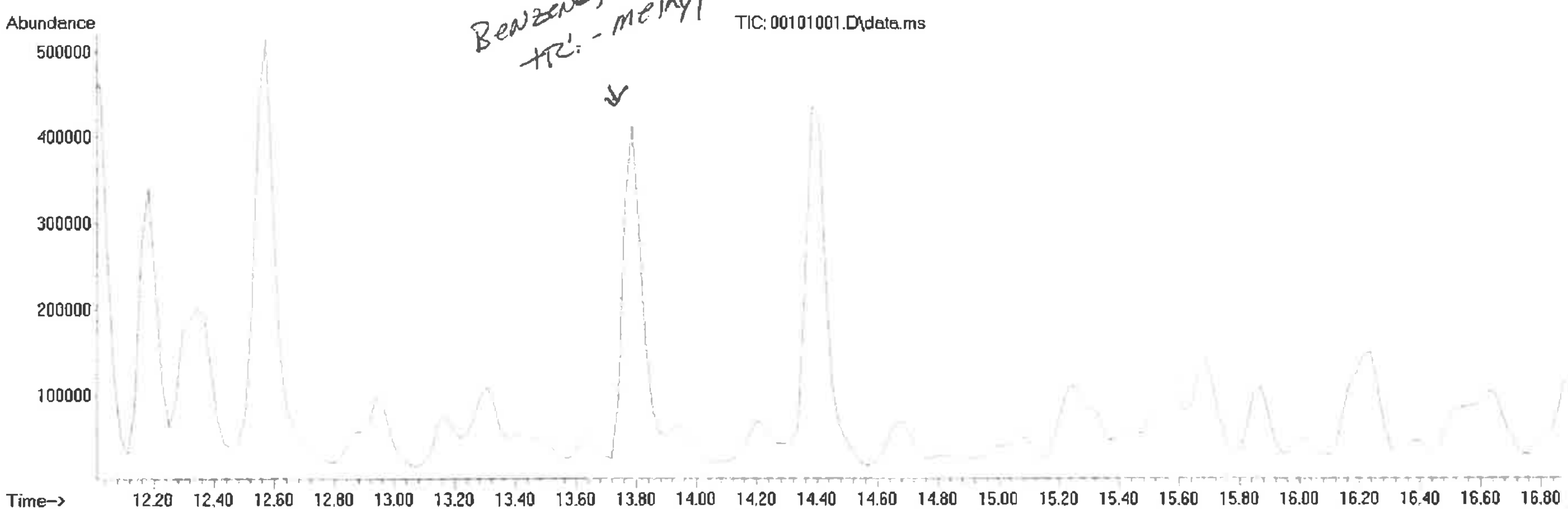


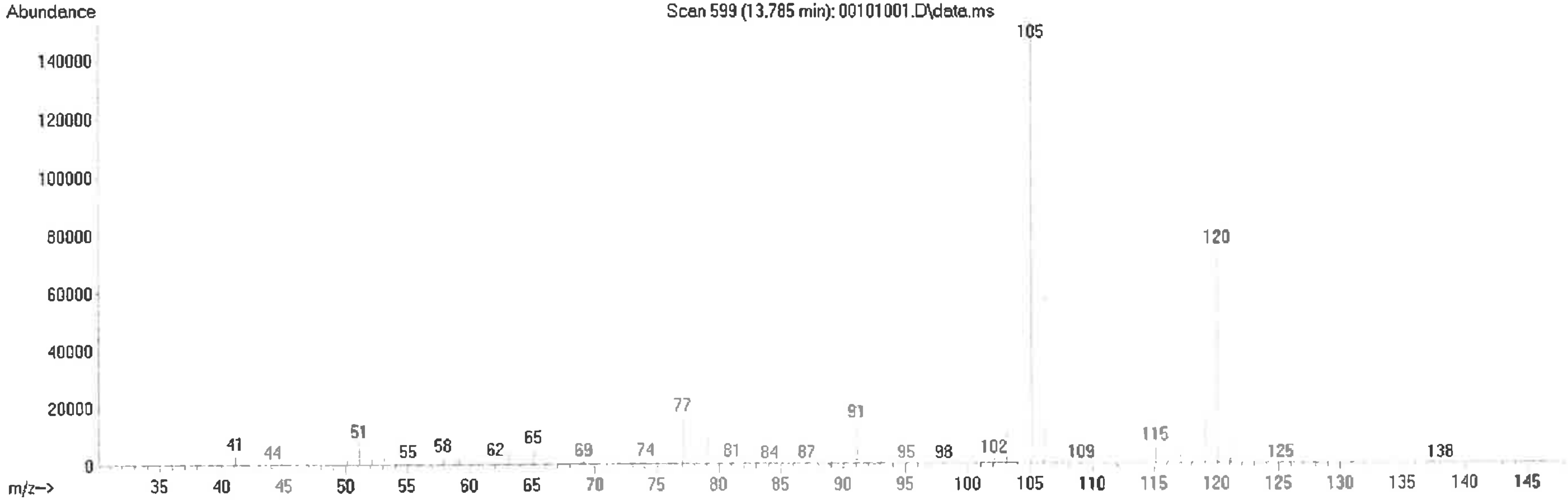
Hit 2 : Benzene, 1-ethyl-3-methyl-
C_9H_{12}; MF: 789; RMF: 933; Prob 21.0%; CAS: 620-14-4; Lib: mainlib; ID: 61559.



File :D:\1\DATA\13FEB13XB\00101001.D
Operator : KC
Acquired : 13 Feb 2013 13:41 using AcqMethod ZEBGENM.M
Instrument : GCMS01
Sample Name: OBBD 15438 P frctn
Misc Info :
Vial Number: 1

J





(K)

** Search Report Page 1 of 1 **

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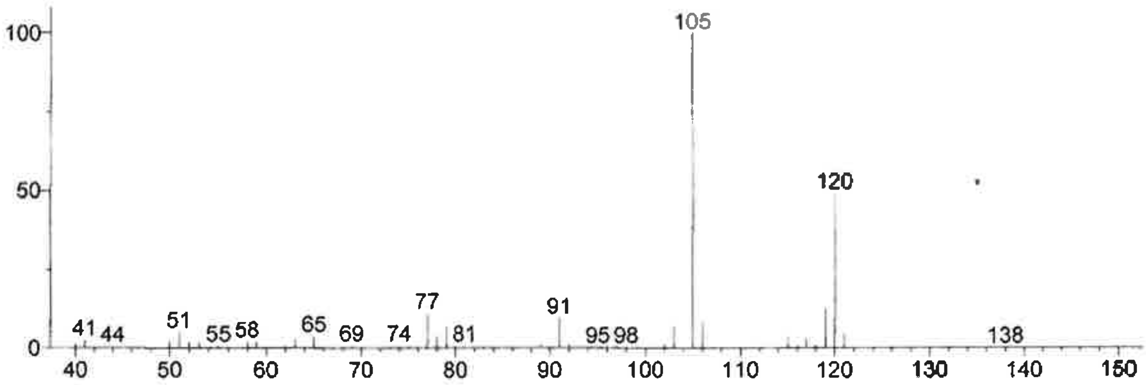
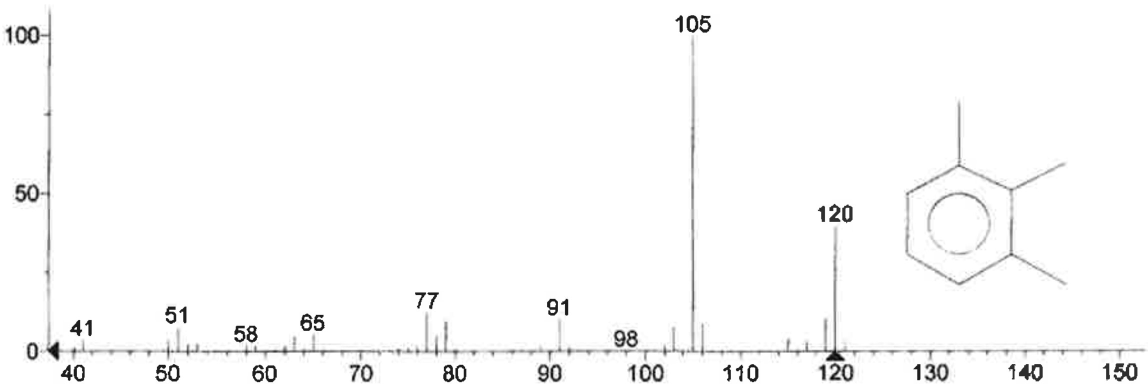
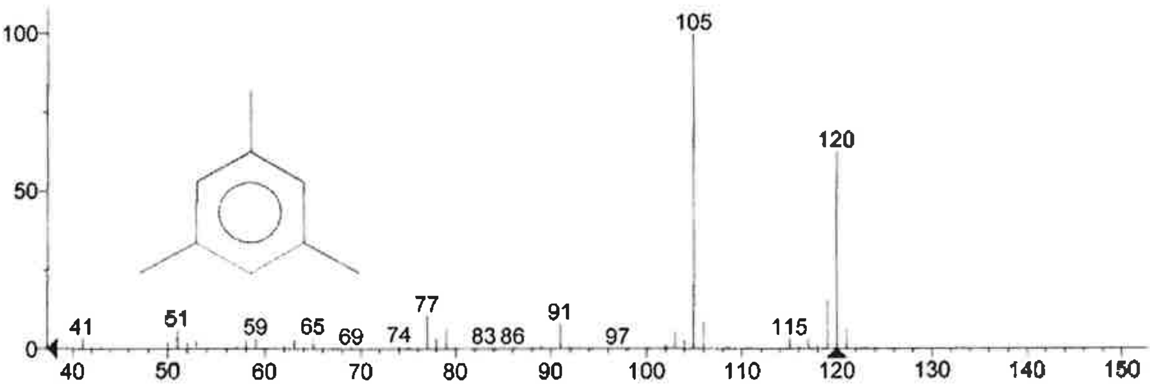
Compound in Library Factor = 138



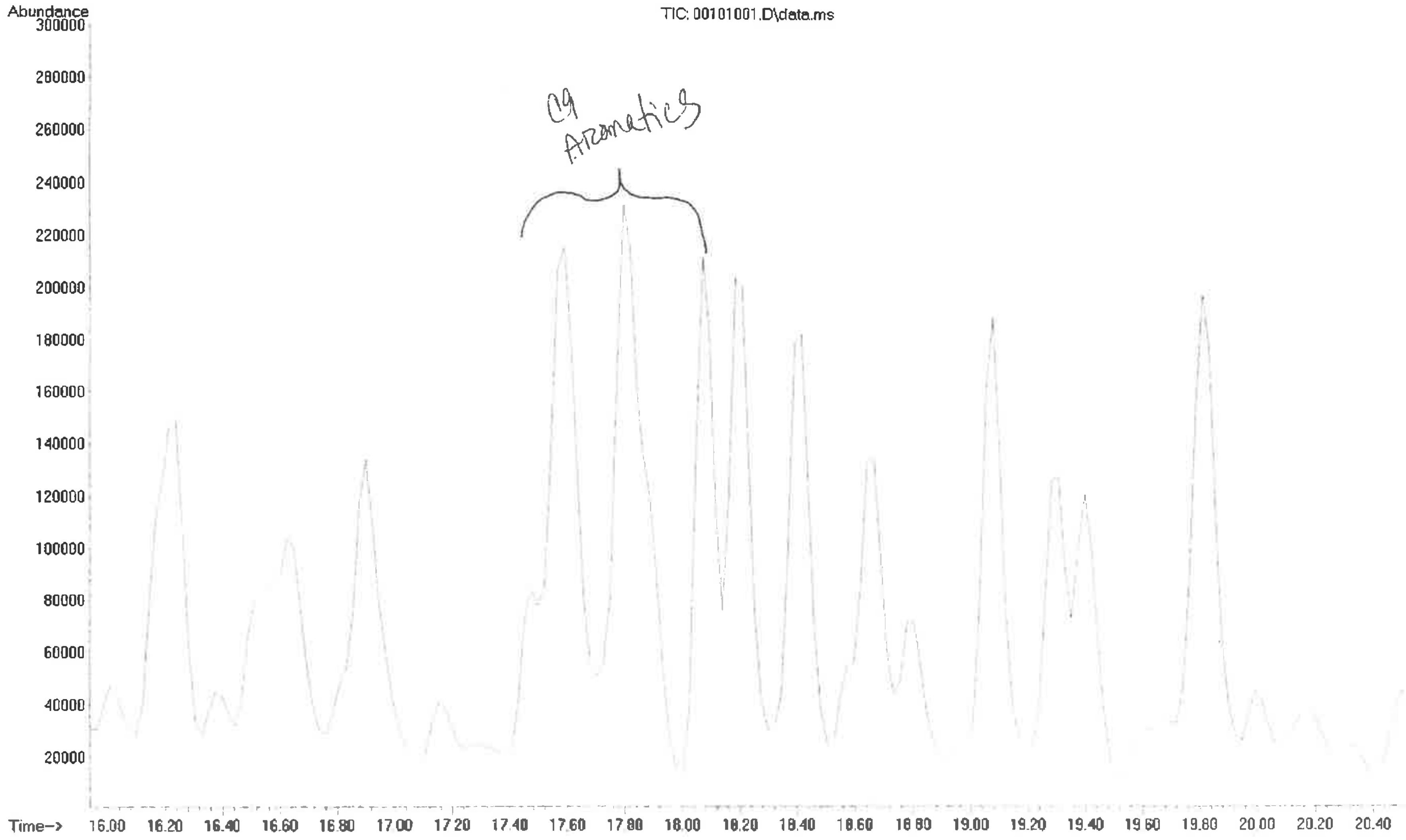
Hit 1 : Benzene, 1,2,3-trimethyl-
C9H12; MF: 935; RMF: 943; Prob 24.6%; CAS: 526-73-8; Lib: mainlib; ID: 61548.



Hit 2 : Benzene, 1,3,5-trimethyl-
C9H12; MF: 930; RMF: 932; Prob 19.8%; CAS: 108-67-8; Lib: replib; ID: 13927.



File : D:\1\DATA\13FEB13XB\00101001.D
Operator : KC
Acquired : 13 Feb 2013 13:41 using AcqMethod ZEBGENM.M
Instrument : GCMS01
Sample Name: OBBD 15438 P frctn
Misc Info :
Vial Number: 1



ANNEX 2

INTERTEK BELGIUM HEFA ANALYSIS BE100-0068443

Intertek

Certificate Of Quality

Your reference : JFK Green Lane project
Product : HEFA
Marked : Shtk 1208
UML samples ex HEFA tank
Date : 4/02/2013
Location : Monument Chemicals
Report n° : BE100- 0068443 / 1
Sample n° : 3755006 / 3796172

Test	Method	Unit	Spec	Result
Table A2.1 Detailed Batch requirements: SPK from Hydroprocessed Esters and Fatty Acids				
COMBUSTION				
Total Acidity	ASTM D 3242	mg KOH/g	max 0.015	0,002
VOLATILITY				
Physical Distillation	ASTM D 86			
Initial Boiling Point		°C	to report	151,3
Fuel recovered at 10% Vol.		°C	max 205.0	169,6
Fuel recovered at 50% Vol.		°C	to report	197,8
Fuel recovered at 90% Vol.		°C	to report	233,4
End point		°C	max 300.0	243,9
T90 - T10		°C	Min 22	63,8
Residue		% Vol	max 1.5	1,2
Loss		% Vol	max 1.5	1,1
Simulated Distillation	ASTM D 2887			
Fuel recovered at 10% Vol.		°C	to report	157,0
Fuel recovered at 50% Vol.		°C	to report	215,9
Fuel recovered at 90% Vol.		°C	to report	275,5
End point		°C	to report	374,1
Flashpoint	ASTM D 56	°C	min 38.0	42,0
Density at 15°C	ASTM D 4052	kg/m3	730.0 - 770.0	759,9
Freezing Point	ASTM D 5972	°C	max -40.0	-54,3
Existent Gum	ASTM D 381	mg/100ml	max 7	4,0
FAME content	IP 585	ppm	max 5	<4,5
THERMAL STABILITY				
2,5 hrs at control temp.	ASTM D 3241			
Temperature		°C	min 325	325
Filter pressure differential		mm Hg	max 25.0	0,1
Tube deposit rating	visual		Less than 3 (No 'peacock' or 'abnormal' colour deposits)	1
ADDITIVES				
Antioxidants *		mg/l	17 - 24	20

A : Accredited analysis by BELAC

Comments : Checked against ASTM D 7566 - 11a TABLE A2.1 dd. July 2011 — Yes / NOK
* info received from supplier

Date of issue : 27/02/2013

Approved by
Peter Gysen
Laboratory Manager
Intertek Belgium

Stephane De Wolf
Shift Supervisor
Intertek Belgium nv

Intertek Belgium NV
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B-2040 ANTWERPEN
Tel.: +32 3 544 10 50 - Fax: +32 3 542 23 44
e-mail : management.belgium@intertek.com

Intertek

Certificate Of Quality

Your reference	:	JFK Green Lane project
Product	:	HEFA
Marked	:	Shtk 1208 UML samples ex HEFA tank Date : 4/02/2013
Location	:	Monument Chemicals
Report n°	:	BE100- 0068443 / 2
Sample n°	:	3755006 / 3796172

Test	Method	Unit	Spec	Result
Table A2.2 Other Detailed requirements: SPK from Hydroprocessed Esters and Fatty Acids				
HYDROCARBON COMPOSITION				
Cycloparaffins *	ASTM D 2425	mass %	max 15	12,2 / 12,3²
Aromatics *	ASTM D 2425	mass %	max 0.5	1.4 / 1,4²
Paraffins *	ASTM D 2425	mass %	to report	86.3 / 86,3²
Carbon & Hydrogen	ASTM D 5291	mass %	min 99.5	98.05 / 99,3²
NON-HYDROCARBON COMPOSITION				
Nitrogen	ASTM D 4629	mg/kg	max 2	<1
Water	ASTM D 6304	mg/kg	max 75	18
Sulphur	ASTM 5453	mg/kg	max 15	<3,0
Sulphur	ASTM D2622	mg/kg	max 15	<3
Metals *	UOP 389	mg/kg		
Al			max 0.1	<0.05
Ca			max 0.1	<0,05
Co			max 0.1	<0.05
Cr			max 0.1	<0.05
Fe			max 0.1	<0.02
K			max 0.1	<0.10
Li			max 0.1	<0.02
Mg			max 0.1	<0.02
Mn			max 0.1	<0.02
Mo			max 0.1	<0.05
Na			max 0.1	0,1
Ni			max 0.1	<0.10
P			max 0.1	<0.10
Pb			max 0.1	<0.10
Pd			max 0.1	<0.05
Pt			max 0.1	<0.05
Sn			max 0.1	<0.20
Sr			max 0.1	<0.02
Ti			max 0.1	<0.20
V			max 0.1	<0.02
Zn			max 0.1	<0.02
Halogens *	ASTM D 7359	mg/kg	max 1	<1

A : Accredited analysis by BELAC

Comments : Checked against ASTM D 7566 - 11a TABLE A2.2 dd. July 2011 — Yes / NOK
* outside lab results.
² Resultaten op tweede staal: 3796172

Date of issue : 27/02/2013

Approved by
Peter Gysen
Laboratory Manager
Intertek Belgium

Stephane De Wolf
Shift Supervisor
Intertek Belgium nv

Precision complies with the precision mentioned in the reference method.
This analytical report is only issued in the name and for account of the principal who recognises that this report purely represents the situation at a given moment. The principal commits himself to disclose each time the complete report and not parts of the report. Fax and E-mail copies have no legal force.
This analytical report can only be used within the specific context of the order and is only valid for the samples analysed and for the company that gave the order.

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SkyNRG

Sky Energy · The Fuel Future

ANNEX 3

EMAIL INTERTEK BELGIUM FEBRUARY 15th, 2013 CONCERNING C+H CONTENT

Bart Rosendaal

Van: Kurt Tyssen Intertek [Kurt.Tyssen@intertek.com]
Verzonden: Friday, February 15, 2013 9:23 PM
Aan: Bart Rosendaal; bart@skynrg.com
CC: Patrick Van Den Berge Intertek
Onderwerp: FW: BE100-0068443 / Tank 1208 HEFA
Bijlagen: 20130215155554.pdf

Urgentie: Hoog

Bart,

Blijkbaar was je email adres verkeerd gespeld in de email van Patrick deze namiddag.
Aromaten werden volgens ASTM D2425 uitgevoerd. Ik heb hieronder de commentaar van het labo die deze test heeft uitgevoerd als achtergrond informatie.

Quote
"The specification for synthetic jet is for the aromatics to be <0.5%.

We are coming up with a result that is >0.5%, which is a cause of concern for us.

This sample was analyzed more than once, with similar results.

Additionally, I analyzed the sample without the pre-separation (neat), which provided us with an even higher aromatic value.

D2425 is a very "vague" analysis which is always a concern when unexpected results happen.

However, I looked into the results in more detail and found evidence that the aromatic content appears to be there. There may be a debate on exactly how much (D2425 is not a precise method (my opinion)), but the aromatic evidence I noticed is usually not present with previous synthetic jet fuels we have analyzed in the past.

If needed, I can pass that information upon request."

Unquote

Zie ook commentaar van Patrick in onderstaande email over C/H gehalte

Mvg,
Kurt

From: Patrick Van Den Berge Intertek
Sent: Friday, February 15, 2013 4:12 PM
To: eline@skynrg.com; bart@skynrgy.com
Cc: Kurt Tyssen Intertek; Peter Gysen Intertek; *OCA EMEA BEL Antwerp Shift Supervisor
Subject: BE100-0068443 / Tank 1208 HEFA

Dear,
Please find attached the preliminary certificate of quality.

Please note that the product is out of limits for:
Aromatics : 1.4 (max 0.5), also after retests.
Carbon & Hydrogen : 98.05% => note: the combined reproducability for C & H of ASTM D 5291 = 3.27%, the uncertainity of the method is to big for the very high specification.
Sodium is just out of the limits but we can report this as 0.1 mg/kg (rounded to the specification digital places (according ASTM E29)).
For Pt & Ti I have asked to our lab in Deerpark to report to the scope of the method instead of <0.20 ppm.

Best regards

Patrick Van Den Berge
Quality Coördinator Laboratory
Intertek Commodities
Cargo Inspection & Analytical Assessment.

kruisschansweg 11
Haven 505
2040 Antwerpen

Tel : +32 3 543 90 83
Fax : +32 3 543 90 77
e-mail : Patrick.Van.Den.Berge@intertek.com
Group-mail : Quality.belgium@intertek.com
web : http://www.intertek.com

Valued Quality. Delivered.

Request to modify D7566 for HEFA jet fuel

Based on operational experience SkyNRG

Bart Rosendaal

December 10th, 2013

KLM NORTH SEA GROUP spring associates

SkyNRG
Sky Energy | The Fuel Future

Brief Introduction to SkyNRG

- Founded in 2009 by KLM aviation, Argos & Spring Ass.
- Mission: Accelerating the global transition to sust. jet fuel
- Active in refining, blending & distribution, incl. into-plane delivery

Partner	KLM	Argos	spring associates
Description	▸ Dow Jones Sustainability Index leader in aviation for the last 5 years ▸ KLM's sustainability ambitions remain high (1% of bio jet fuel consumption in 2015 , and 17% net carbon emission reductions by 2020)	▸ Argos is a down-stream fuel logistics player in Western Europe ▸ Focus on oil storage, transportation, sales and trade of oil products and bio fuel blending and distribution for road transport	▸ Spring Associates is a strategy consulting firm in the clean-tech and sustainability space. ▸ Venturing is an integral part of Spring Associates' proposition
Contribution	▸ Aviation network ▸ Air-side fuel logistics ▸ Contracts ▸ Launching customer	▸ Oil trading/hedging ▸ Downstream fuel supply ▸ Operational biofuel expertise ▸ Legal support ▸ Working capital	▸ Business development ▸ Strategic decisions ▸ Sustainability knowledge ▸ Technology knowledge ▸ Office support

SkyNRG has global experience and a broad customer base

Current customers | **Close friends & partners**

Americas

North America
Boeing
Alaska Airlines
porter
Air Canada

South America
AeroMexico
LAN
TAM

EMEA

Europe
KLM
Airbus
Air France
Finnair
Thomson Airways
Virgin

Middle East
Etihad Airways

APAC

Asia
ANA
Thai
Jetstar

Australia
Virgin
Qantas

Global

Aviation
Airbus
Boeing
IATA

Supply
Greenergy
Dynamic Fuels
Neste Oil
LanzaTech
heliae

Distribution
EPIC
China National Aviation Fuel

NGOs'
Climate Solutions
RSB Roundtable on Sustainable Biofuels
Carbon War Room
WWF
NRDC
National Wildlife Federation

"The biofuel blend was supplied by SkyNRG, which has virtually cornered the market availability of sustainable jet fuel just now, with involvement already in commercial biofuel flights by carriers in Europe, Asia, the United States and now the Middle East" (GreenAirOnline)

Operational Experience with HEFA

- Refining
  - Toll manufacturing HEFA jet fuel
  - Partnering with Dynamic Fuels, Monument Chemicals and KMTEX
  - Feedstocks used: Used Cooking Oil (UCO) & Jatropha
  - Total volume pure HEFA-jet produced so far: approx. 200,000 gallons

- Blending
  - Blending of HEFA jet with fossil jet A and jet A-1 in various concentrations
  - Full recertification to D1655

- Distribution
  - Partnering with Epic Aviation in US
  - Distribution of on-spec D7566-table 1 / D1655 jet fuel to airports
  - Setting up separate supply systems at airports for into-plane fuelings

Overview of modification request for D7566

1. Cancel aromatics content analysis in table 2.2
2. Find & allow alternative method to D2425 (hydrocarbon comp.)
3. Replace D5291 method for C+H content for Oxygen cont.
4. Change trace metals specification from individual limits to overall limit
5. Allow alternative method D7111 to determine trace metals

1. Cancel aromatics content

- Aromatics are required for jet fuel
  - Minimum density requirement
  - Energy content
  - Sufficient swell of gaskets and seals
- D1655 says maximum, but no minimum
  - Fossil jet fuel always has aromatics
  - Lowest we have seen in fossil jet is about 10%
  - HEFA has neglectible aromatics level
- D7566 has conflicting specs
  - Pure HEFA is not allowed to have any aromatics (process control?)
  - Blended HEFA is required to have minimum of 8%
- Experience: Aging catalyst produces more aromatics
  - We have seen aromatics as high as 1.4% in pure HEFA
  - Very expensive and time-consuming to get aromatics out
  - If it has no technical function, why not cancel the aromatics specification?

2. Find & allow alternative method to D2425

- D7566 requires D2425 for Hydrocarbon analysis
  - To determine type and composition of hydrocarbons in pure HEFA jet
  - Important for final jet quality and to verify HEFA-process used
  - Different from D1655 or D7566-table 1 where D1319 and D6379 are required
- Problem: D2425 is not a widely-used standard
  - All our samples so far have been sent to South West Research Institute
  - Our partner Intertek says they cannot offer this method in-house
  - SWRI has only 2 machines running
- Very long and costly analysis path
  - Usually it take 7-10 days for results to come in
  - Very costly
  - Long waiting time prior to and after blending (first HEFA test, then blend test)
- is D2425 fit-for-purpose?
  - Some of our surveyors question the method's preciseness and suitability

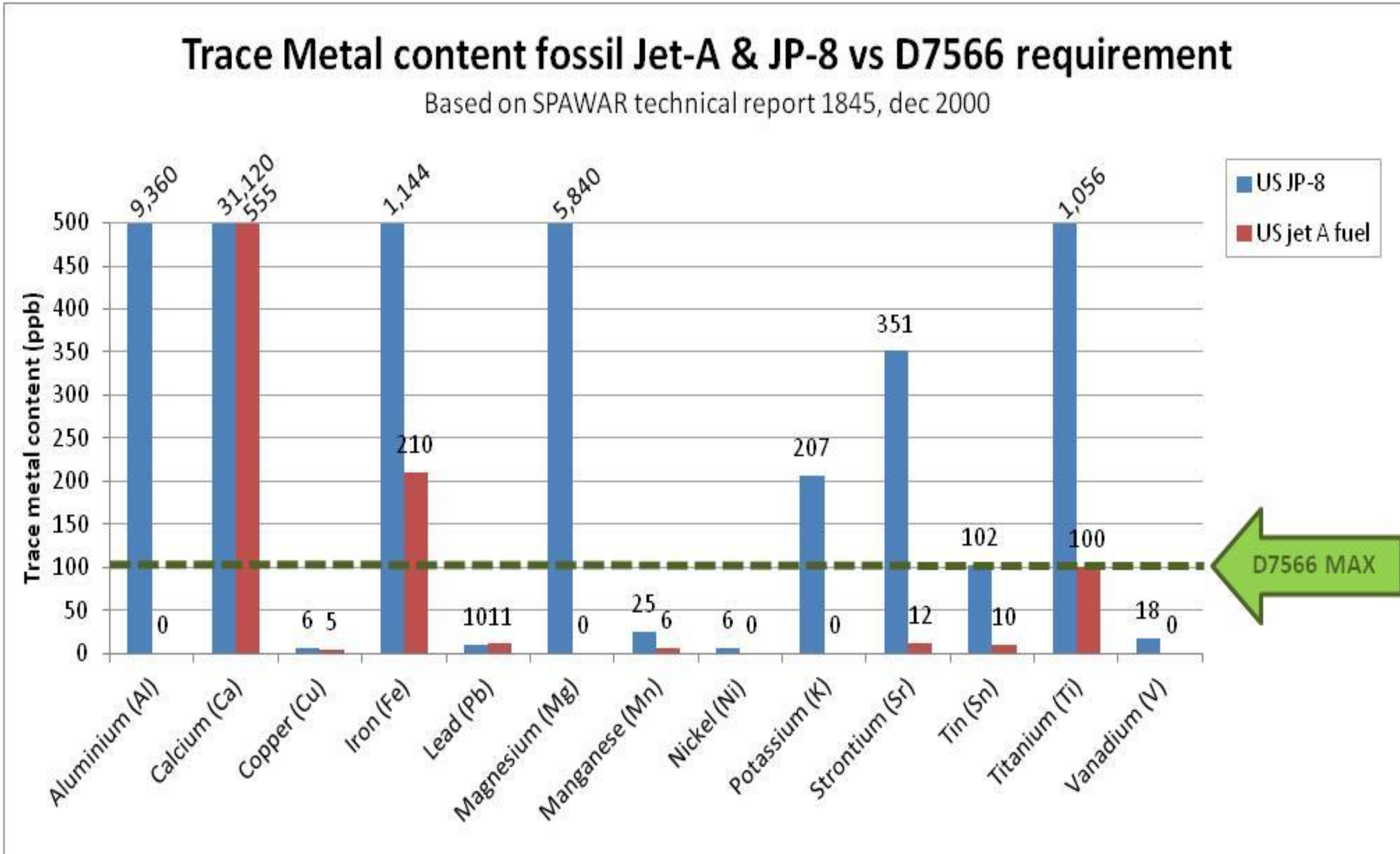
3. Replace D5291 method for oxygen content

- D7566 requires total amount of hydrocarbons to be known
  - Probably done to exclude presence of oxygen
  - Has to be >99.5 wt.-%
- Problem: D5291 is not (by far) accurate enough
  - Accuracy according Intertek is ±3.5 wt.-%, impossible to use for >99.5% !!!
  - Our results on same HEFA have been fluctuating between 98% and almost 100%
  - Has to be >99.5 to be able to get a certified product, so we keep testing until we hit it
- If oxygen is the target, measure it directly instead of indirectly
  - Total oxygen is common in gasoline (in EU max 2.7 wt.%)
  - EN-238, EN 12177 or EN-ISO-22854 is used in Europe
  - Or allow other method to determine oxygen

4. Change trace metals to overall limit of 25 ppm

- D7566 requires individual testing for 21 metals
  - Al, Ca, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, Pd, Pt, Sn, Sr, Ti, V and Z
  - Individual levels are set to < 0.1 ppm-wt
- Issue 1: Levels are very low and touching the detection limit
  - Sampling is very important, ASTM method for sampling has to be used
  - Off-spec readings can occur easily, requiring re-sampling and re-analysis
  - Using clay filters to clean-up HEFA fuel can already create off-spec situation
  - According Intertek, the 0.1 ppm-wt is close to detection limit for at least some metals
- Issue 2: Fossil jet appears to have much more metals in them
  - See SPAWAR report of December 2000
  - Jp-8: Al, Ca, Fe, Mg, K, Sr, Sn and Ti all seem to be off-spec (used for Jet A-1 blending)
  - Jet A: Ca and Fe seem off-spec
  - Why have such stringent specs on metals for HEFA if next step involves blending with fossil fuel?

4. Change trace metals to overall limit of 25 ppm



5. Allow D7111 next to UOP389

- D7566 requires UOP-389 method for trace metal testing
  - Reason for choosing UOP389 is ability to test for phosphorous?

- Issue: UOP389 is very uncommon method
  - Long analysis times (sample has to travel to specialised lab)
  - high costs
  - Usually D7111 is used and this is very common and quick for petroleum testing

- Allow D7111 next to UOP389 and add phosphorous detection