

Page 1 of 26

CPG Quote #: 1585B  Client PO #: 6295967  Report Review (all reviewers must be RSI or above):  Sign:	Print: Van Buren  Role (RS1, RS2, Exec): RS2  Report sign:	Thursday, October 13, 2011	
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Page 2 of 26

Consultation, Testing, and Instrumentation for Polymeric Materials

Thursday, October 13, 2011

To: Dan Burrill,

Boston Scientific Corporation

Re: Comparison of polypropylene samples

Project Manager: Turner

Project Scientist: Adams, Braithwaite, Nugent, Svirkin, Turner, Wilson-Hill

Report Written by: Braithwaite, Nugent, Turner, Wilson-Hill

Report prepared on: 9/29/2011

Report Reviewed by: Spiegelberg, van Buren

Samples: Polypropylene pellets

CPG sample #	Date received	Client sample description
11440-1	9/9/2011	Sample 1, Lot# PP0353133-02
11440-2	9/9/2011	Sample 2, Lot# 6120105
11440-3	9/20/2011	Sample 3, Lot# 2980058

Equipment: Q1000 DSC; Biorad FTS3000 FTIR with UMA500 microscope; Melt flow die; Shodex: Three Linear GPC Column; HP 6890 GC with an HP 5972A MS; Olympus SZ40 stereo microscope with a 0.5X lens and a Jenoptik SpeedCore 5XT digital camera

Summ	nary	3
Sampl	les	3
Exper	imental	3
3.1	Differential Scanning Calorimetry (DSC)	3
3.2		
3.3		
3.4	Melt Flow Index (MFI)	
3.5	Gel Permeation Chromatography (GPC)	4
3.6		
3.6.1	Extraction for Elutables	
3.6.2	Gas Chromatography - Mass Spectroscopy (GC-MS)	5
3.7	Inductively Coupled Plasma (ICP) Spectroscopy	
3.8	Optical Microscopy	5
Result		
4.1	Differential Scanning Calorimetry (DSC)	5
4.2	Oxidation Induction Time (OIT)	····· ·´
4.3		
4.4	Melt Flow Index (MFI)	9
4.5	Gel Permeation Chromatography (GPC)	9
4.6		
4.6.1		
4.6.2		
4.7		
4.8	Optical Microscopy	22
	Exper 3.1 3.2 3.3 3.4 3.5 3.6 3.6.1 3.6.2 3.7 3.8 Result 4.1 4.2 4.3 4.4 4.5 4.6 4.6.1 4.6.2 4.7	3.2 Oxidation Induction Time (OIT) 3.3 Fourier Transform Infrared Spectroscopy (FTIR) 3.4 Melt Flow Index (MFI) 3.5 Gel Permeation Chromatography (GPC) 3.6 Contamination Detection 3.6.1 Extraction for Elutables 3.6.2 Gas Chromatography – Mass Spectroscopy (GC-MS) 3.7 Inductively Coupled Plasma (ICP) Spectroscopy 3.8 Optical Microscopy Results 4.1 Differential Scanning Calorimetry (DSC) 4.2 Oxidation Induction Time (OIT) 4.3 Fourier Transform Infrared Spectroscopy (FTIR) 4.4 Melt Flow Index (MFI) 4.5 Gel Permeation Chromatography (GPC) 4.6 Contamination Detection 4.6.1 Extraction for Elutables 4.6.2 Gas Chromatography – Mass Spectroscopy (GC-MS) Inductively Coupled Plasma (ICP) Spectroscopy

56 Roland Street Suite 310, Boston MA 02129

7-11 Client Report (11/3/2010)



Page 3 of 26

### Summary

A series of tests were conducted on the three poly(propylene) samples received. No difference was detected between the three samples for DSC, FTIR, MFI, and GPC. The OIT for sample 11440-2 was slightly higher than the other two, although all oxidation induction times were within minutes of each other. More residue was extracted from sample 11440-2, and the GC-MS indicated that more compounds were present in the extracted residue of sample 11440-2. The survey scan using ICP showed sample 11440-1 with a wider array of trace metals than the other two samples. Optically, there were also some differences between the samples. Samples 11440-1 and 11440-3 showed more occlusions within the pellet, but samples 11440-2 and 11440-3 were more similar in pellet size.

Three samples of polypropylene pellets were received for analysis. The samples were each assigned a unique CPG number.

#### 3 Experimental

### Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) measurements were run using a Q1000 DSC (TA Instruments). The instrument was set up according to CPG SOP0044 (rev. 11/8/2010). One specimen from each sample was tested for thermal analysis. Specimens were prepared in standard aluminum pans according to CPG SOP0043 (rev. 10/1/2009). A clean razor blade was used to section a disc specimen for each sample, which was then weighed on a precision balance (AND GR-202, ±0.01mg). Each specimen was then crimped between a standard aluminum pan and lid. An empty aluminum pan and lid were used as a reference. A 50 mL/min nitrogen purge was used. The test procedure is listed below:

Equilibrate: -50 °C Isothermal: 5.00 min

Ramp Rate: 20.00 °C/min to 200.00 °C

Mark End Cycle 1 Isothermal: 5.00 min

Ramp Rate: 20.00 °C/min to -50.00 °C

Mark End Cycle 2 Isothermal: 5.00 min Ramp 20.00 °C to 200.00 °C Mark End Cycle 3

Both the first and second heat curves of all samples were analyzed in TA Universal Analysis 2000 software. A glass transition temperature, Tg [°C], was not observed for any of the three samples in either heat cycle. A linear baseline was constructed to analyze the melt peaks from 40 to 190 °C to determine the onset melt temperature,  $Tm_o$  [°C], and peak melt temperature,  $Tm_p$  [°C], and by integrating the endotherm to determine the mass-normalized heat of fusion,  $\Delta H$  [J/g].

### Oxidation Induction Time (OIT)

Oxidation induction time (OIT) was determined using a TA Instruments Q1000 differential scanning calorimeter (DSC) according to ASTM D3895-98. One specimen for each of the three samples was tested. A clean razor blade was used to section each specimen to a mass between 5 and 10 mg. The sample was placed into a standard aluminum pan bottom. An empty aluminum pan bottom was used as a reference. Preparation, instrument set-up, and testing were conducted according to CPG SOP0044 (rev. 11/08/2010).

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7-11 Client Report (11/3/2010)

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Page 4 of 26

Consultation, Testing, and Instrumentation for Polymeric Materi

The testing parameters are listed below:

Set Point: 200.00 °C Purge gas: Nitrogen Purge rate: 50 mL/min Oxygen rate: 50 mL/min

The test procedure is listed below: Equilibrate: 40 °C

Ramp Rate: 20.00 °C/min to 200.00 °C

Isothermal: 5.00 min Mark End of Cycle 1 Select Gas 2: Oxygen Isothermal: 120.00 min Select Gas 1: Nitrogen Mark End of Cycle 2

Ramp Rate: 20.00 °C/min to 40 °C

### 3.3 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy was performed with a Biorad FTS3000 FTIR with UMA500 microscope. The instrument was aligned and calibrated according to CPG SOP0009 (rev. 10/12/2010). A small section was removed from each sample using a clean razor blade to get a section thin enough for analysis. The spectra were collected in transmission mode with the following parameters:

Aperture size: 200 µm # scans/location: 32 Purge: Nitrogen

# 3.4 Melt Flow Index (MFI)

Testing was conducted per ASTM D1238-10 Procedure A. The samples did not require drying prior to testing. The melt flow analysis was performed with the following conditions:

Temperature: 230 °C Plunger weight: 2.16 kg Orifice: 0.0825 inches

Two runs were conducted for each sample.

### 3.5 Gel Permeation Chromatography (GPC)

Gel permeation chromatography (GPC) was performed on all three samples to determine the molecular weight. The samples were filtered prior to analysis. The test parameters for the samples are shown below, with three injections for each sample:

Solvent: TCB, 135 °C Column: Shodex: Three Linear Flow Rate: 1.0 mL/min Injection volume: 150 μL Detector: M-150-C (64/25)

Calibration standards: Polypropylenes

### 3.6 Contamination Detection

The extractable residues from the polypropylene were analyzed using gas chromatography with mass spectroscopy (GC-MS), with comparisons made to the library database. Any differences in components were noted. The procedure is described in detail below.

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Page 5 of 26

### **Extraction for Elutables**

All glassware used was cleaned according to CPG SOP0055 (rev. 8/11/2011); the depyrogenation step was not conducted since pyrogen free glassware was not needed. Each sample was added to a 100 mL flask as received. The total mass of the material added was measured and recorded. Hexane (VWR, ACS grade, ≥98.5%) was then added to the flask to cover the sample material, but no more than half the capacity of the flask. The flask was attached to a condenser cooled with circulated water at 5 °C, and then lowered into a stirred oil bath between 68 and 70 °C. The temperature was adjusted to achieve desired refluxing. After refluxing for 24 hours, the flask was removed from the extraction apparatus. The solvent was poured into a clean beaker of known mass; the sample remained in the flask. This process was repeated twice more for a total reflux time of 72 hours, with fresh hexane replacement every 24 hours. The solvent collection beakers were left open to allow the solvent to evaporate. The remaining cumulative extraction residue was weighed daily until the difference between the consecutive masses was less than 5 %. The amount of extracted residue was then determined as function of the total mass. The mass of the extracted residue was divided by the initial mass of the films and converted to a percent.

## Gas Chromatography – Mass Spectroscopy (GC-MS)

Gas chromatography-mass spectrometry (GC-MS) was performed on the extracted residue. For each sample, approximately 20 mL of methylene chloride was added to the residue in the beaker. A portion of the dissolved residue was then placed into GC autosampler vials and analyzed. A spectral library database was used for compound identifications. The chromatographic conditions are listed below:

Instrument: HP 6890 GC with an HP 5972A MS

Column: Rxi-5MS, 30 m x 0.25 mm ID x 0.25 µm film thickness

Carrier: Helium; 0.8 mL/min Injector Temperature: 250 °C Injection Mode: Split 10:1 Injection Volume: 1 µL

Oven Temperature Program: Hold 5.0 min at 35 °C, 10 °C/min to 300 °C, hold 5.0 min at 325 °C

#### Inductively Coupled Plasma (ICP) Spectroscopy 3.7

Each sample of polypropylene pellets was analyzed. All of the samples were dry ashed at 600 °C; the ash was dissolved in HNO3 and HCl. After the acid digestion, the total metals content was measured with inductively coupled plasma/mass spectrometry (ICP/MS). An Agilent Model 7700 ICP/MS system was used for the analysis.

### Optical Microscopy

Optical analysis was performed using an Olympus SZ40 stereo microscope with a 0.5X lens and a Jenoptik SpeedCore 5XT digital camera. No special preparation was performed on the samples, with five individual pellets selected at random from the container.

#### 4 Results

#### Differential Scanning Calorimetry (DSC) 4.1

The data analysis for the DSC test is summarized Table 1. The overlay of the first heat cycle of all samples is shown in Figure 1. The overlay of the second heat cycle of all samples is shown in Figure 2.

There was no glass transition was observed for any of the samples. All samples have a single melt peak between 150 °C and 160 °C. The onset melt temperature increases slightly from the first heat cycle to the second heat cycle for all the samples. In addition, in samples 11440-2 and 11440-3 a small transition is observed at approximately 50 °C in the first heat cycle only. This is possibly due to different crystal sizes arising from different thermal histories. During the controlled cooling cycle, the samples likely form a more uniform crystalline structure. Therefore, the small transition is not observed in the second heat cycle.

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Page 6 of 26

Consultation, Testing, and Instrumentation for Polymeric Materials

Table 1: Summary of onset melt temperature, peak melt temperature, and enthalpy for all samples.

	Heat Cycle 1			
Sample	Onset Melt Temperature [ °C]	Peak Melt Temperature [°C]	Enthalpy [J/g]	
11440-1	150.48	164.49	104.9	
11440-2	152.26	161.87	112.1	
11440-3	151.69	164.54	111.3	
	Heat Cycle 2			
Sample	Onset Melt Temperature [ °C]	Peak Melt Temperature [°C]	Enthalpy [J/g]	
11440-1	154.14	163.44	106.9	
11440-2	154.60	161.79	109.5	
11440-3	157.52	163.67	113.2	

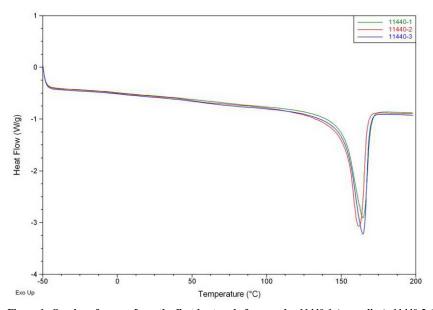


Figure 1: Overlay of curves from the first heat cycle for samples 11440-1 (green line), 11440-2 (red line), and 11440-3 (blue line).

56 Roland Street Suite 310, Boston MA 02129

7-11 Client Report (11/3/2010)

Page 7 of 26

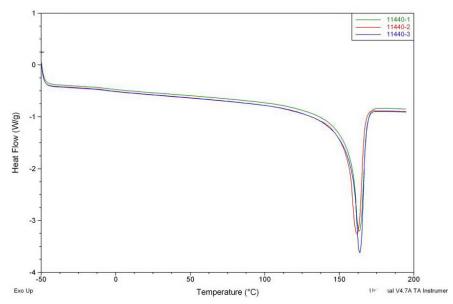


Figure 2: Overlay of curves from the second heat cycle for samples 11440-1 (green line), 11440-2 (red line), and 11440-3 (blue line).

#### Oxidation Induction Time (OIT) 4.2

The oxidation induction times for each sample are shown in Table 2. Following ASTM D3895-98, the OIT of each sample was determined. Using TA Instruments Universal Analysis 2000 Software, the tangent method was employed and each thermal curve was analyzed. As an example, one can see the tangent method applied to the thermal curve of sample 11440-1 in Figure 3. The oxidation induction time for sample 11440-2 is almost twice the time for the other two samples, although all the measurements are within minutes. The larger oxidation induction time for 11440-2 indicates greater oxidative stability under these test conditions.

**Table 2: Oxidation Induction Time results** 

Sample	Oxidation Induction Time [min]
11440-1	5.61
11440-2	8.23
11440-3	4.49

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7-11 Client Report (11/3/2010)

Page 8 of 26

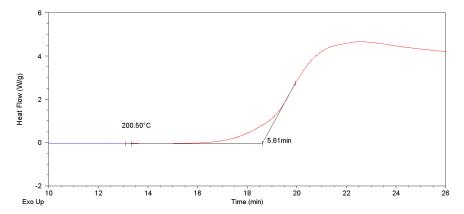
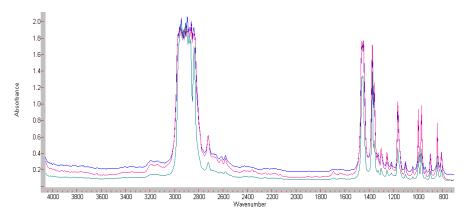


Figure 3: The tangent method employed in determining the OIT of sample 11440-1. The transition from the blue line to the red line indicates the switch from nitrogen to oxygen gas.

# Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR results are shown in Figure 4 and Figure 5. As can be seen, there is no difference between the three spectra.



Figure~4:~FTIR~spectral~comparison~of~sample~11440-1~(green~line),~11440-2~(blue~line),~and~11440-3~(blue~line),~and~11(pink line).

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Figure 5: FTIR spectral comparison of sample 11440-1 (green line), 11440-2 (blue line), and 11440-3 (pink line) (expanded scale).

1100 1050 1000 950 900

## Melt Flow Index (MFI)

Two runs were conducted on each sample for melt flow index. The results are shown in Table 3. The melt flow index, measured as an average of the two runs, is similar for the three samples but there are slight differences between the samples, with sample 11440-3 having the highest melt flow index.

Table 3: Melt flow index results.

Commile	Time [s]	Mass Extruded [g]		Average Melt Flow
Sample		Run 1	Run 2	Rate [g/10 min]
11440-1	60	0.391	0.389	3.90
11440-2	60	0.375	0.370	3.72
11440-3	60	0.473	0.468	4.70

# Gel Permeation Chromatography (GPC)

1400 1350 1300 1250

The molecular weight moments for all samples, relative to poly(propylene) standards, are shown in Table 4. The calibration curve for the samples is shown in Figure 6. Representative refractive index plots are shown in Figure 7 through Figure 9, and the resulting molecular weight distribution plots are provided in Figure 10 through Figure 12. As can be seen from the spectrum, there is a unimodal distribution of molecular weights as only one peak appears. There is little difference between the molecular weights for the three samples. However, the results are consistent with the MFI results. Sample 11440-2 had the lowest MFI, indicating greater resistance to flow. This sample also had the highest Mz and Mw, which would have the greatest influence on MFI.

56 Roland Street Suite 310, Boston MA 02129

7-11 Client Report (11/3/2010)



Boston Scientific Corporation Report # 11440-1 Page 10 of 26

Table 4: Molecular weight moments [g/mol] for all samples.

11440-1				
Run	Mn	Mw	Mz	PDI
1	6.76E+04	2.65E+05	7.78E+05	3.93
2	6.83E+04	2.62E+05	7.80E+05	3.84
3	6.82E+04	2.69E+05	7.73E+05	3.94
Average	6.80E+04	2.65E+05	7.77E+05	3.90
St. Dev.	3,91E+02	3,35E+03	3,33E+03	0.06

		11440-2		
Run	Mn	Mw	Mz	PDI
1	5.92E+04	2.82E+05	9.01E+05	<b>4.</b> 77
2	5.68E+04	2.76E+05	8.36E+05	4.86
3	5.80E+04	2.88E+05	8.86E+05	4.96
Average	5.80E+04	2.82E+05	8.74E+05	4.86
St. Dev.	1.18E+03	5.70E+03	3.39E+04	0.10

		11440-3		
Run	Mn	Mw	Mz	PDI
1	5.87E+04	2.55E+05	7.76E+05	4.35
2	6.14E+04	2.63E+05	8.39E+05	4.28
3	6.24E+04	2.62E+05	8.33E+05	4.19
Average	6.08E+04	2.60E+05	8.16E+05	4.27
St. Dev.	1.89E+03	3.94E+03	3.50E+04	0.08

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Figure 6: Calibration curve for polypropylenes in TCB at 135  $^{\rm o}\text{C}.$ 

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7-11 Client Report (11/3/2010)

Figure 7: Representative raw refractive index signal for sample 11440-1.

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Figure 8: Representative raw refractive index signal for sample 11440-2.

 $Figure \ 9: \ Representative \ raw \ refractive \ index \ signal \ for \ sample \ 11440-3.$ 

RET VOL (mL)

 $${\tt LOG}$\ M$$  Figure 10: Representative molecular weight distribution for sample 11440-1.

 ${\bf Figure~11:~Representative~molecular~weight~distribution~for~sample~11440-2.}$ 

Page 17 of 26

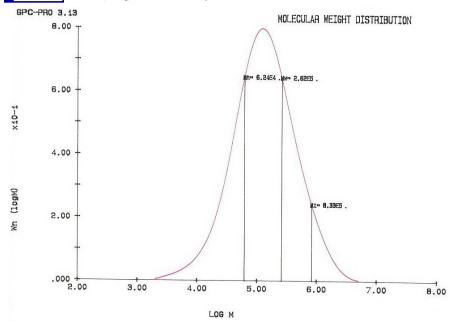


Figure 12: Representative molecular weight distribution for sample 11440-3.

#### 4.6 **Contamination Detection**

### **Extraction for Elutables**

The amount of extractable residue from the three samples is shown in Table 5. The amount of residue extracted for samples 11440-1 and 11440-3 is very similar. However the amount of residue extracted for sample 11440-2 is more than double the amount extracted for the other two samples.

Table 5: Extractable residue results, based on mass.

Sample	Mass of Sample [g]	Extracted Residue [mg]	Percent of Total Mass Extracted [%]
11440-1	5.85	37.6	0.64
11440-2	6.01	86.1	1.43
11440-3	6,21	37.9	0.61

### 4.6.2 Gas Chromatography – Mass Spectroscopy (GC-MS)

The ion chromatograms for the three samples are shown in Figure 13 to Figure 15 with a summary table shown below each figure. The mass spectroscopy identification of the compounds is summarized in Table 6. As shown, the types of fragments detected for the three residue samples are similar but the residue from sample 11440-2 seems to have a higher level of these compounds present. The majority of the extracted compounds are long chain aliphatic hydrocarbons; however there appears to be a few aromatic fragments detected by the GC-MS for sample 11440-2.

It is most likely that two compounds with the same retention time are the same compound. However, it is difficult to tell the difference between the larger alkanes because they undergo extensive fragmentation

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7-11 Client Report (11/3/2010)

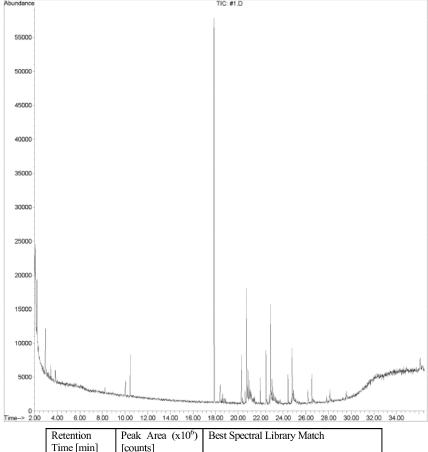
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Page 18 of 26

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during ionization and appear similar afterwards. The best match found for the mass spectra of each peak is chosen from the spectral library, but this assignment is not definitive.



Time [min] [counts] 17.87 0.96 No acceptable spectral match 20.30 0.15 Pentacosane 20.75 0.31 Tetracosane 22.46 Tetracosane 0.14 22.87 0.25 Nonadecane 24,78 0.16 Heptadecane

Figure 13: Total ion chromatogram for sample 11440-1 including chemical species identification through a spectral library match.

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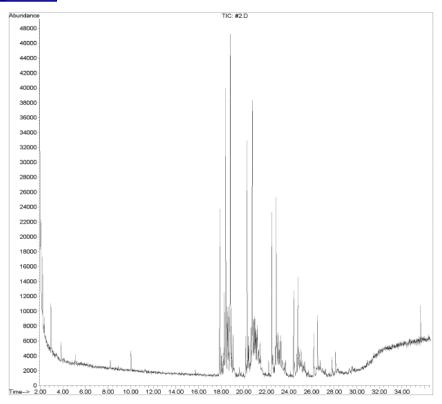
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Boston Scientific Corporation Report # 11440-1
Page 19 of 26

Consultation, Testing, and Instrumentation for Polymeric Materials



Retention Time [min]	Peak Area (x10 <sup>6</sup> ) [counts]	Best Spectral Library Match
17.87	0.41	Heptadecane
18.29	0.21	Bis dimethylethyl phenol
18.40	0.83	Docosane
18.52	0.20	Tetracosane
18.63	0.19	Tetracosane
18.81	1.01	Hydroxy benzoic acid
20.30	0.55	Tetracosane
20.75	0.82	Tetracosane
20.85	0.22	Tetracosane
22.47	0.35	Nonadecane
22.87	0.46	Pentadecane
24.42	0.20	Tetracosane
24.77	0.22	Heptadecane
26.51	0.19	Pentacosane

Figure 14: Total ion chromatogram for sample 11440-2 including chemical species identification through a spectral library match.

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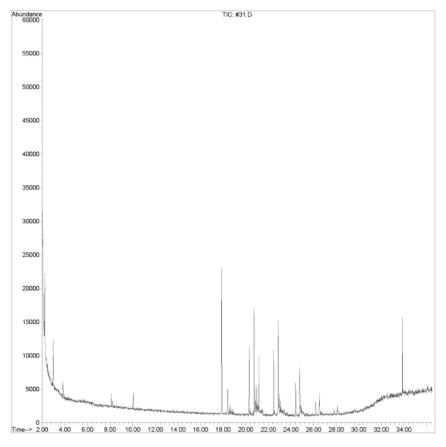
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Boston Scientific Corporation Report # 11440-1 Page 20 of 26



Retention	Peak Area (x10 <sup>6</sup> )	Best Spectral Library Match
Time [min]	[counts]	
17.87	0.31	No acceptable spectral match
20.30	0.17	Pentadecane
20.75	0.31	Tetracosane
22.87	0.21	Tetracosane

Figure 15: Total ion chromatogram for sample 11440-3 including chemical species identification through a spectral library match.

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Page 21 of 26

Consultation, Testing, and Instrumentation for Polymeric Materials

Table 6: Chemical species found in the three samples using GC-MS analysis. The majority of compounds are long chain alkanes, and discretion between chain lengths is challenging. The compounds checked in red are not simple alkanes.

Component	11440-1	11440-2	11440-3
Bis dimethylethyl phenol		✓	
Docosane		✓	
Heptadecane	<b>√</b>	✓	
Hydroxy benzoic acid		✓	
Nonadecane	✓	✓	
Pentacosane	<b>√</b>	✓	
Pentadecane		✓	<b>√</b>
Tetracosane	<b>✓</b>	✓	✓
Unidentified compound	<b>✓</b>		<b>√</b>

# 4.7 Inductively Coupled Plasma (ICP) Spectroscopy

The data for the inductively coupled plasma spectroscopy of all samples is summarized in Table 7. There are some common elements between the three samples, however sample 11440-1 showed a larger array of trace metals than the other two samples. All the samples showed high levels of selenium. As this is a rare and toxic element, this result is unusual and should be verified using another technique, such as SEM-EDS.

56 Roland Street Suite 310, Boston MA 02129

7-11 Client Report (11/3/2010)

(617) 629 4400

Boston Scientific Corporation Report # 11440-1 Page 22 of 26

Table 7: Data summary of all samples for survey scan of metals. Note that ND indicates that the element was not detected within the detection limits.

	Concentration by weight [ppm]					
Element	11440-1	11440-2	11440-3	Detection Limit		
Be	43.0	ND	ND	0.01		
Na	ND	ND	ND	0.05		
Mg	ND	ND	ND	0.05		
Al	106	ND	25.9	0.01		
K	ND	ND	ND	0.10		
Ca	ND	ND	ND	0.01		
Ti	ND	280	ND	0.01		
V	20.9	ND	ND	0.01		
Cr	33.1	ND	ND	0.01		
Mn	6.8	ND	ND	0.01		
Fe	36.1	ND	ND	0.01		
Co	16.4	ND	ND	0.01		
Ni	27.4	ND	ND	0.01		
Cu	14.7	ND	ND	0.01		
Zn	ND	ND	ND	0.01		
As	ND	ND	ND	0.01		
Se	1500	1700	2160	0.01		
Sr	29.2	ND	14.8	0.01		
Mo	ND	ND	ND	0.01		
Cd	ND	ND	ND	0.01		
Sn	ND	ND	ND	0.01		
Sb	ND	ND	ND	0.01		
Ba	10.4	15.7	34.3	0.01		
T1	ND	ND	ND	0.01		
Pb	ND	ND	ND	0.01		

### 4.8 **Optical Microscopy**

Initially, five pellets were drawn from each container and photographed individually. Images for sample 11440-1 are provided in Figure 16, for 11440-2 in Figure 17 and finally 11440-3 in Figure 18. Note that the scale bar should be treated as reference only. In addition, a further 15 specimens (five from each sample group) were removed and photographed at the same time using oblique lighting (see Figure 19). Image analysis was used to determine the long- and short-axis, perimeter and area of the pellets as viewed (see Table 8). Statistically samples 11440-2 and -3 are the same size (p=0.52) with sample 11440-1 statistically smaller. In contrast, samples 11440-1 and 11440-3 visually both exhibit marked internal occlusions, presumably due to different cooling conditions during pelletization. Sample 11440-2 is almost completely devoid of internal occlusions and may have a slightly different surface finish. There was no visible evidence of contaminants in the form of specks or debris, although some images suggest the presence of small fibers. However, given the packaging provided, these are not surprising. Examples are provided in Figure 20.

56 Roland Street Suite 310, Boston MA 02129

7-11 Client Report (11/3/2010)

(617) 629 4400

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CONFIDENTIAL

Page 23 of 26

Table 8: Calculated parameters for images shown in Figure 19

	Axis 1 [mm]	Axis 2 [mm]	Aspect Ratio	Perimeter [mm]	Area [mm <sup>2</sup> ]
11440-1,1	3.87	3.97	1.03	12,24	11.51
11440-1.2	4.27	3.85	1.11	12.62	12.28
11440-1.3	3.87	3.70	1.04	11.88	10.93
11440-1.4	3.70	3.83	1.03	12.07	11.18
11440-1.5	4.19	3.76	1.11	12.45	12.00
Average	3.98	3.82	1.07	12.25	11.58
St. Dev.	0.24	0.10	0.04	0.30	0.56
11440-2.1	4.79	4.19	1.14	14.02	15.23
11440-2.2	4.44	4.35	1.02	13.57	14.31
11440-2.3	4.36	4.19	1.04	13.47	14.08
11440-2.4	4.52	4.22	1.07	13.63	14.23
11440-2.5	4.65	4.31	1.08	14.20	15.56
Average	4.55	4.25	1.07	13.78	14.68
St. Dev.	0.17	0.07	0.05	0.31	0.67
11440-3.1	4.51	4.08	1.10	13.34	13.59
11440-3.2	4.54	4.27	1.06	13.95	15.09
11440-3.3	4.92	4.24	1.16	14.11	15.17
11440-3.4	3.87	4.74	1,23	13.57	14.07
11440-3.5	3.85	4.77	1.24	13.54	14.03
Average	4.34	4.42	1.16	13.70	14.39
St. Dev.	0.47	0.31	0.08	0.32	0.70



Figure 16: Five randomly obtained pellets for 11440-1. Additional image shows single representative zoom of far right pellet.

56 Roland Street Suite 310, Boston MA 02129 7-11 Client Report (11/3/2010)

(617) 629 4400

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Boston Scientific Corporation Report # 11440-1 Page 24 of 26



Figure 17: Five randomly obtained pellets for 11440-2. Additional image shows single representative zoom of far right pellet.



Figure 18: Five randomly obtained pellets for 11440-3. Additional image shows single representative zoom of far right pellet.

56 Roland Street Suite 310, Boston MA 02129 7-11 Client Report (11/3/2010)

(617) 629 4400

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Page 25 of 26



Figure 19: Comparison of all three sample groups with calculated perimeter shown. Other parameters are provided in Table 8.

56 Roland Street Suite 310, Boston MA 02129 7-11 Client Report (11/3/2010)

(617) 629 4400



Boston Scientific Corporation Report # 11440-1 Page 26 of 26

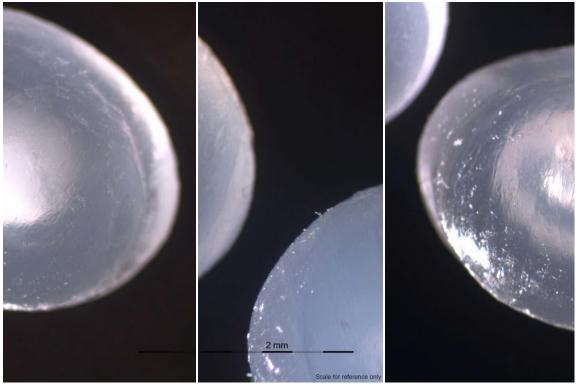


Figure 20: Examples of visible debris on the specimens

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