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Chemical characterization of waxes

Ref : CR08235002
Date : April 4th, 2011

Introduction :

RESCOLL has carried out a chemical screening of several waxes available on the market in order to verify the accordance between real composition of the products and the claims written on the different packages. In a few words, are the so called "bio" or "green" waxes really bio-based and petrochemical free?

6 different waxes have been studied:

- MATUNAS COLD,
- RAMSON COOL FORMULA,
- TERRAWAX 11-17°C/52-62°F,
- FAMOUS GREEN LABEL WARM,
- STICKY BUMPS SOY WAX WARM,
- GREENFIX COLD.

These samples have all been analyzed following a test protocol developed by RESCOLL in order to assess the presence of petrochemical based products in the formulations. According to this protocol, we can identify the major constituents of the waxes (petrochemical or bio-based). However, ingredients with contents under 5%in mass are not detected with this test method.

Analysis methodology

Waxes are constituted by organic compounds (in the sense of carbon based products, synthetic or bio-based) and mineral fillers. This last ingredients may interfere in the characterization, thus extraction of these fillers is mandatory. So, first step of the test protocol is separation of the mineral fillers from the organic ingredients.

Test method is constituted by several steps:

- Extraction of the organic phase with cyclohexane under agitation overnight at room temperature,
- Separation between cyclohexane soluble compounds and other constituents by centrifugation and filtration
- Drying of the cyclohexane solution by solvent evaporation,
- Analysis of the dried mix by Fourier Transform Infra-Red spectroscopy (FTIR), following standard T 51-500 (1989) and Differential Scanning Calorimetry (DSC) following standard NF EN ISO 11357-3 (1999).

Results :

MATUNAS COLD

FTIR spectra and DSC thermogram of this wax are presented in Appendix 1.

The FTIR analysis shows that the analysed portion of the product contains:

- mainly signals that are typical of CH_2 and aliphatic CH functions,
- a band corresponding to the C=O function at 1745 cm^{-1}.

The C=O function band has a very low intensity, which indicates that the sample contains no or very few fatty acids, constitutive compounds of vegetable waxes and oils.

This analysis underlines that **major organic constituents of this wax are from petrochemical origin, for instance paraffins and mineral oils.**

RAMSOM COOL FORMULA

FTIR spectra and DSC thermogram of this wax are presented in Appendix 2.

The FTIR analysis shows that the sample only contains CH_2 and aliphatic CH functions. Bands characteristic to fatty acids are missing on the spectrum.

So, organic constituents of this wax are mainly from petrochemical origin.

TERRAWAX 11-17°C/52-62°F

FTIR spectra and DSC thermogram of this wax are presented in Appendix 3.

Results obtained for this sample are similar to those from RAMSON COOL FORMULA.

Then, organic constituents of TERRAWAX 11-17°C/52-62°F are mainly petrochemicals.

FAMOUS GREEN LABEL WARM

FTIR spectra and DSC thermogram of this wax are presented in Appendix 4.

The FTIR spectrum of this sample contains the bands presented in the following table.

Wave number (cm^{-1})	Mode
2916 – 2848	ν- CH_2 aliphatic
1737 – 1708	ν-C=O ester / acid
1463	d- CH_2 aliphatic
1366	δ- CH aliphatic
1192 – 1169	ν-C-O ester
728 - 719	γ- $(CH_2)_n$ aliphatic n>4

This wax has a more complicated formula compared to the previous references.
The spectrum shows all the bands that are characteristic to vegetable waxes like beeswax, coprah oil and soy butter.

However, DSC does not allow concluding on the absence of paraffin in the formula.

In order to conclude on the paraffin question, we studied the intensity of the FTIR signals. It appears that paraffin, if present in the sample, should correspond to less than a few % in mass.

Thus, organic constituents of this wax are mainly from natural origin.

STICKY BUMPS SOY WAX WARM

FTIR spectra and DSC thermogram of this wax are presented in Appendix 5.

The FTIR spectrum of the sample shows bands that are presented in the table below.

Wave number (cm^{-1})	Mode
2916 – 2848	ν- CH_2 aliphatic
1737	ν-C=O ester
1462	δ- CH_2 aliphatic
1376	δ- CH aliphatic
1171 – 1113	ν-C-O ester
729 - 719	γ- $(CH_2)_n$ aliphatic n>4

According to this spectrum, this wax contains a vegetable oil like coprah oil.

However, given the intensity of the FTIR bands at 1737 and 729cm-1 and the DSC melting temperatures, this sample may probably contain a petrochemical based constituent like paraffin. However, it is difficult to conclude on the presence of this paraffin on the basis of these analyses.

Thus, organic constituents of this wax are mainly from natural origin.

GREENFIX COLD

FTIR spectra and DSC thermogram of this wax are presented in Appendix 6

The FTIR spectrum of this sample shows bands that are presented in the following table.

Wave number (cm^{-1})	Mode
2917 – 2852	ν- CH_2 aliphatic
1730	ν-C=O ester
1497	ν- C=C aromatic
1461	δ- CH_2 aliphatic
1377	δ- CH aliphatic
1235 -1169 – 1104	ν-C-O ester
884 – 822	γ- CH aromatic
719	γ- $(CH_2)_n$ aliphatic n>4

The bands characteristic to aromatic functions seem to indicate the presence of modified rosin.
The ratio between heights of the peaks at 2917 and 1730 cm^{-1} indicates the presence of vegetable waxes (beeswax for instance).

Then, identified constituents of this wax are exclusively from natural origin.

Conclusions

The following table summarizes the results obtained for the 6 tested waxes.

Wax	Identified constituents
MATUNAS COLD	Mainly waxes and oils from **petrochemical origin**
RAMSON COOL FORMULA	Mainly waxes and oils from **petrochemical origin**
TERRAWAX 11-17°C/52-62°F	Mainly waxes and oils from **petrochemical origin**
FAMOUS GREEN LABEL WARM	Mainly waxes and oils from **natural origin**
STICKY BUMPS SOY WAX WARM	Mainly waxe and oils from **natural origin**. Probably very low % of petrochemical waxes
GREENFIX COLD	Waxes and oils from **natural origin exclusively** Modified rosin (**natural origin**)

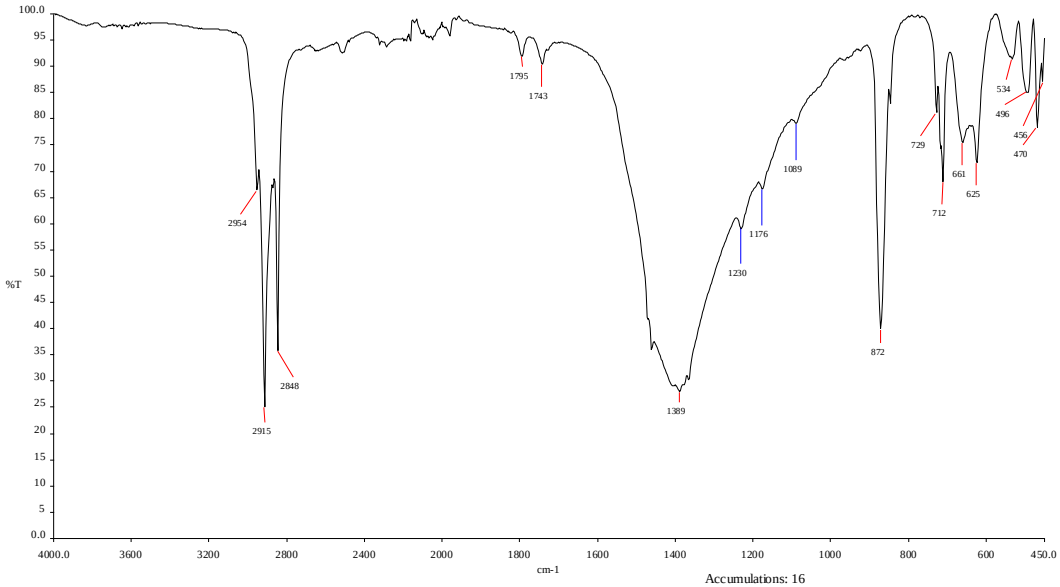
Appendix 1: FTIR spectra and DSC thermograms of MATUNAS COLD

RESCOLL

Spectrum Pathname: C:\pel_data\spectra\6349.003

Date: 03/12/10

Opérateur : BL



Analyst: Admin

Description: RC/08235/MATUNAS ORGANIC COLD

Date Created: fri dec 03 08:54:15 2010

Accumulations: 16

Resolution: 4 cm-1

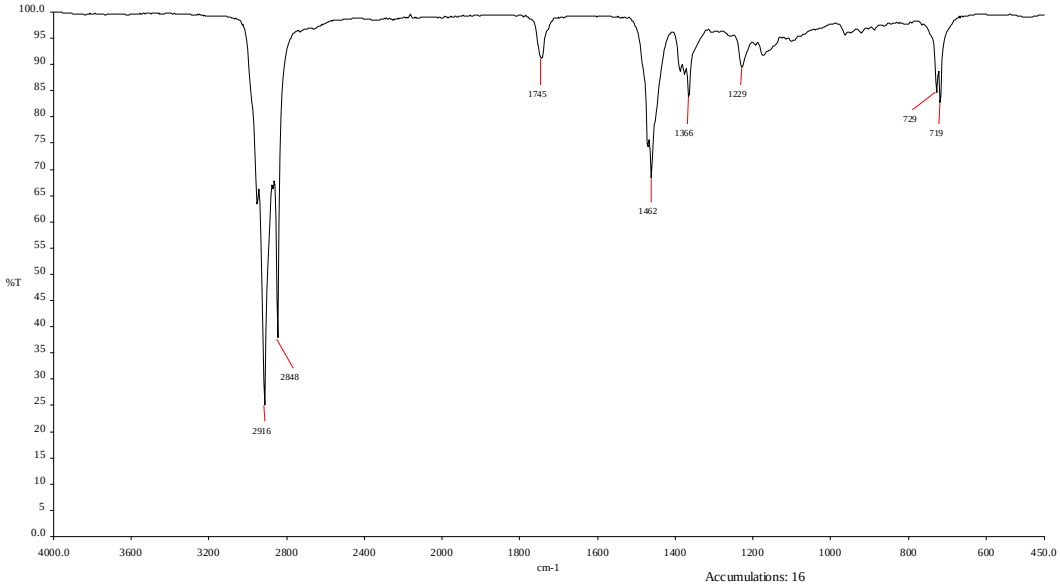
IR Accessory: Golden Gate

RESCOLL

Spectrum Pathname: C:\pel_data\spectra\6381.003

Date: 14/12/10

Opérateur : BL



Analyst: Admin

Description: RC/08235/Extrait Cyclo MATUNAS ORGANICS COLD

Date Created: tue dec 14 16:23:16 2010

Accumulations: 16

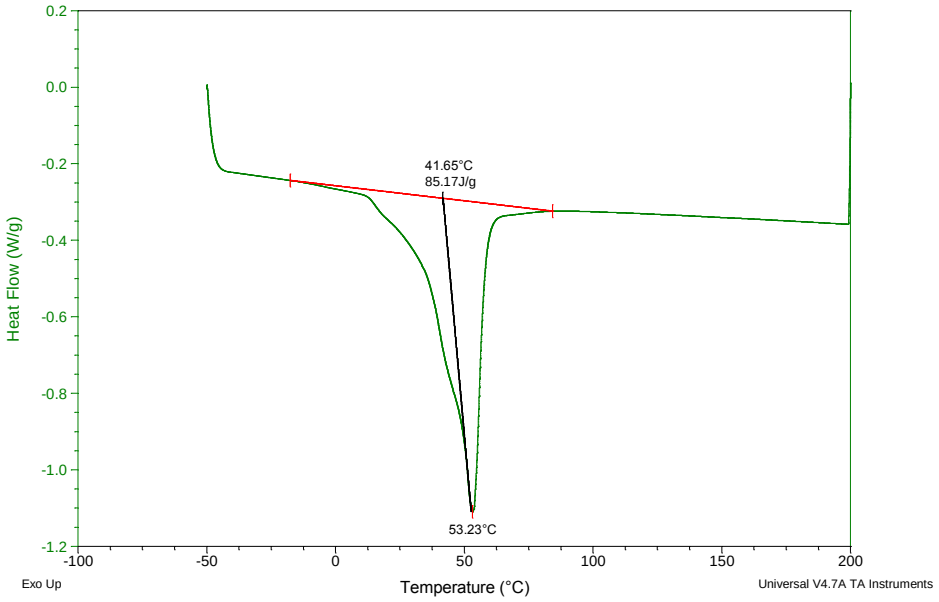
Resolution: 4 cm-1

IR Accessory: Golden Gate

Sample: RC/08235/MATUNAS
Size: 9.5400 mg
Method: ISO 11357

DSC

File: C:...\RC\2008\08235\RC_08235_MATUNAS.00
Operator: BL
Run Date: 12-Jan-2011 00:44
Instrument: DSC Q2000 V24.8 Build 120



Sample: RC/08235/MATUNAS
Size: 9.5400 mg
Method: ISO 11357

DSC

File: C:...\RC\2008\08235\RC_08235_MATUNAS.00
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Run Date: 12-Jan-2011 00:44
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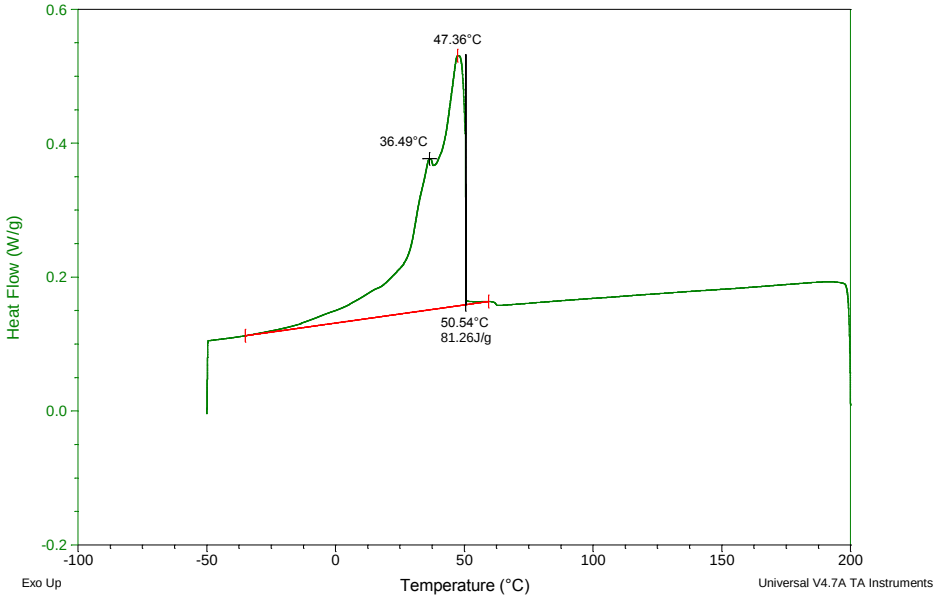
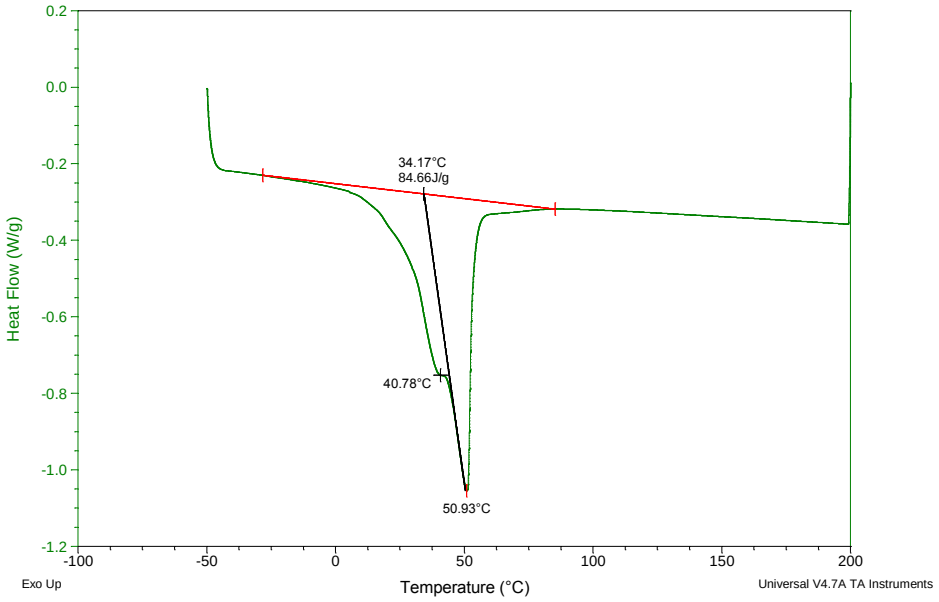


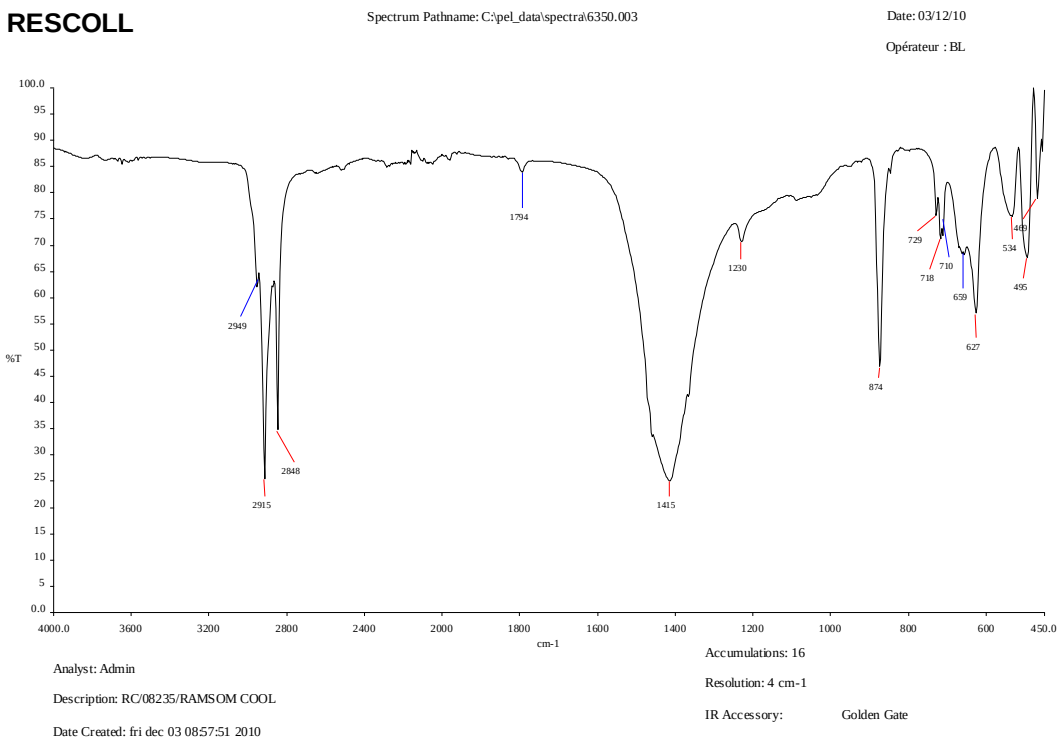
Sample: RC/08235/MATUNAS
Size: 9.5400 mg
Method: ISO 11357

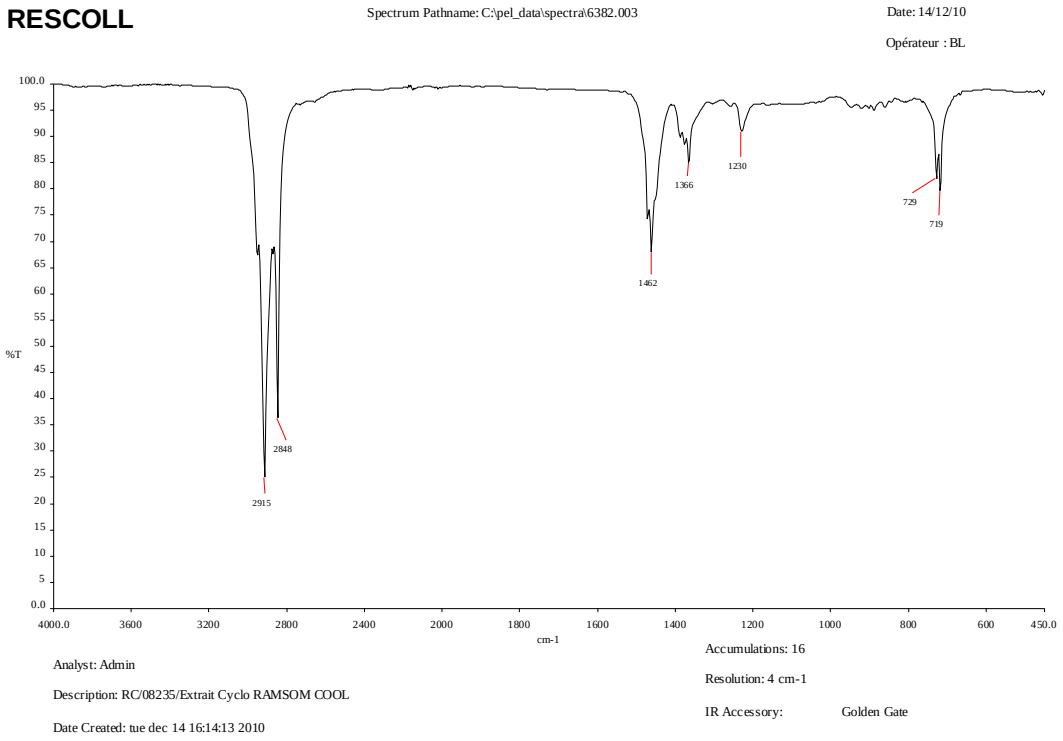
DSC

File: C:...\RC\2008\08235\RC_08235_MATUNAS.00
Operator: BL
Run Date: 12-Jan-2011 00:44
Instrument: DSC Q2000 V24.8 Build 120



Appendix 2: FTIR spectra and DSC thermograms DSC of RAMSON COOL FORMULA

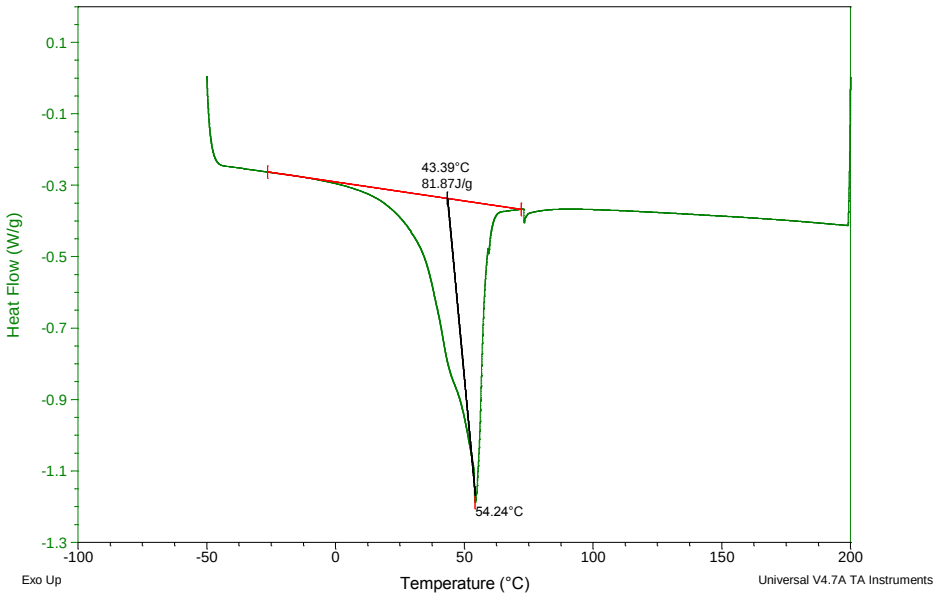




Sample: RC/08235/RAMSON
Size: 7.9400 mg
Method: ISO 11357

DSC

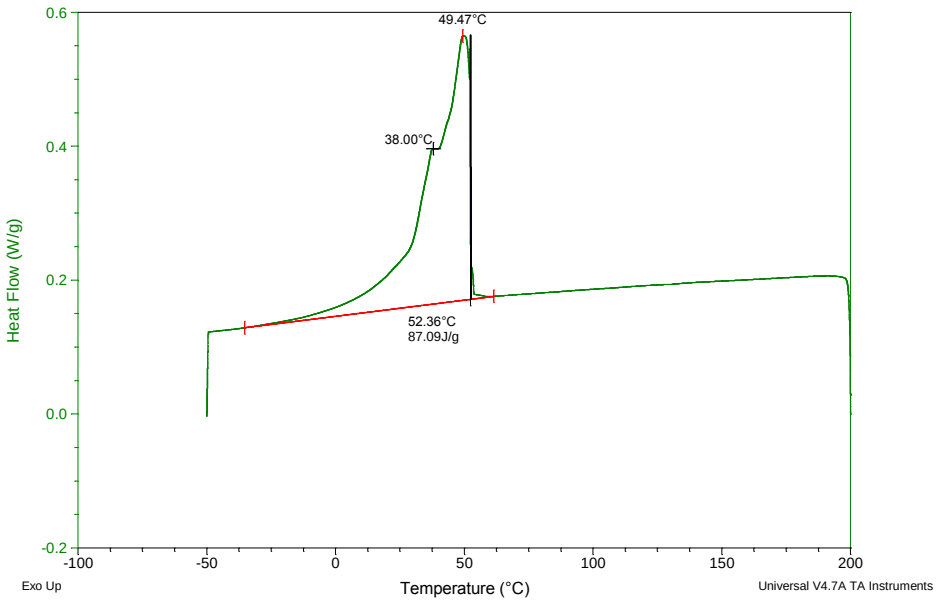
File: C:...\RC\2008\08235\RC_08235_RAMSON.001
Operator: BL
Run Date: 11-Jan-2011 22:34
Instrument: DSC Q2000 V24.8 Build 120



Sample: RC/08235/RAMSON
Size: 7.9400 mg
Method: ISO 11357

DSC

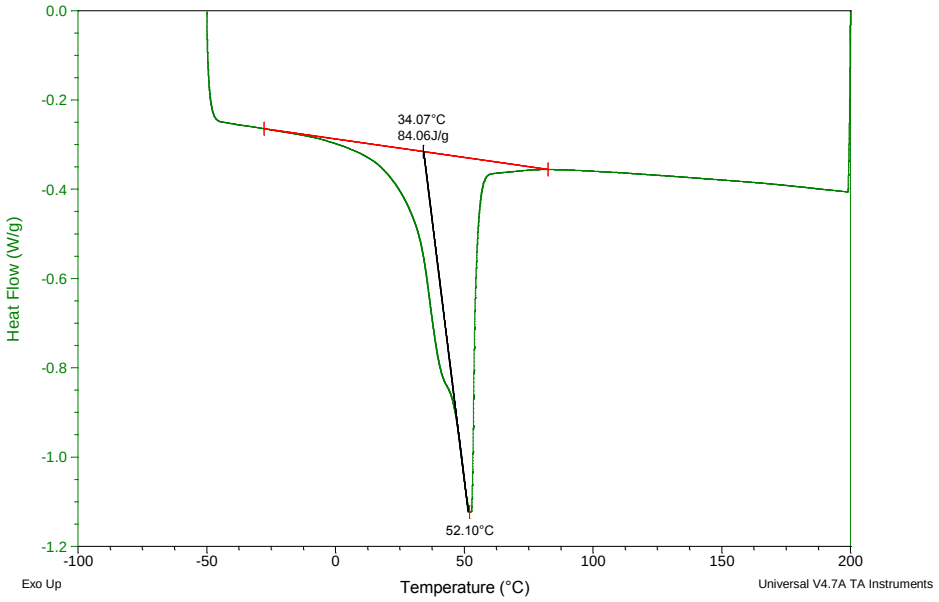
File: C:...\RC\2008\08235\RC_08235_RAMSON.001
Operator: BL
Run Date: 11-Jan-2011 22:34
Instrument: DSC Q2000 V24.8 Build 120



Sample: RC/08235/RAMSON
Size: 7.9400 mg
Method: ISO 11357

DSC

File: C:...\RC\2008\08235\RC_08235_RAMSON.001
Operator: BL
Run Date: 11-Jan-2011 22:34
Instrument: DSC Q2000 V24.8 Build 120



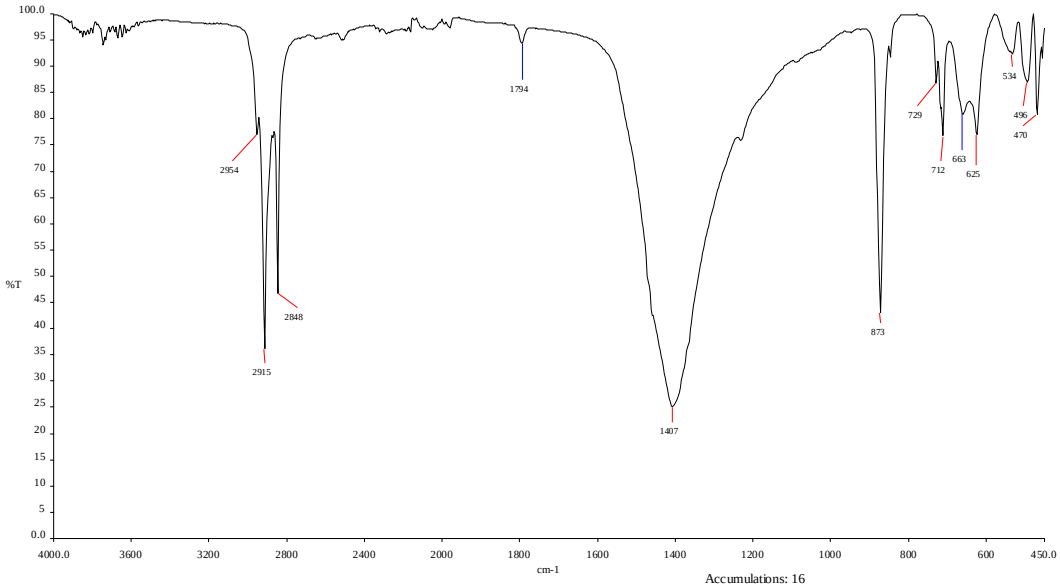
Appendix 3: FTIR spectra and DSC thermograms of TERRAWAX 11-17°C/52-62°F

RESCOLL

Spectrum Pathname: C:\pel_data\spectra\6348.003

Date: 03/12/10

Opérateur : BL



Analyst: Admin

Description: RC/08235/TERRA WAX 11-17

Date Created: fri dec 03 08:48:45 2010

Accumulations: 16

Resolution: 4 cm-1

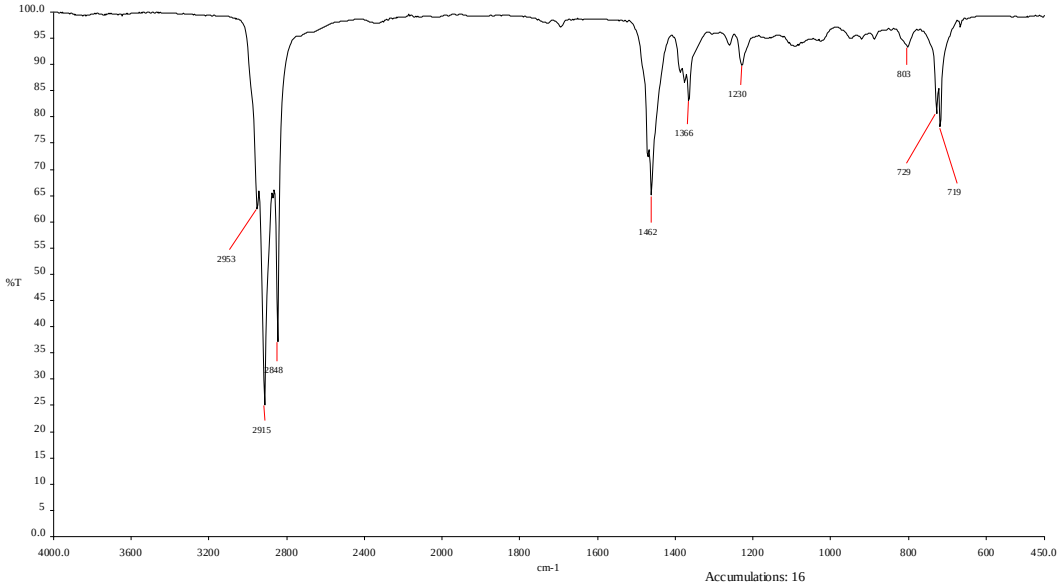
IR Accessory: Golden Gate

RESCOLL

Spectrum Pathname: C:\pel_data\spectra\6380 .003

Date: 14/12/10

Opérateur : BL



Analyst: Admin

Description: RC/08235/Extrait Cyclo TERRA WAX 11-17

Date Created: tue dec 14 16:20:09 2010

Accumulations: 16

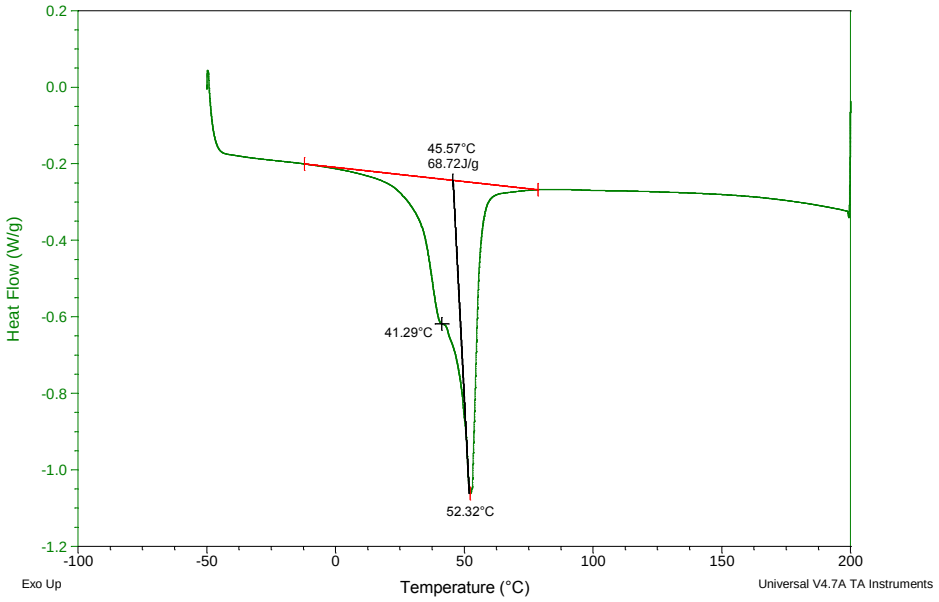
Resolution: 4 cm-1

IR Accessory: Golden Gate

Sample: RC/08235/TERRA WAX
Size: 7.1800 mg
Method: ISO 11357

DSC

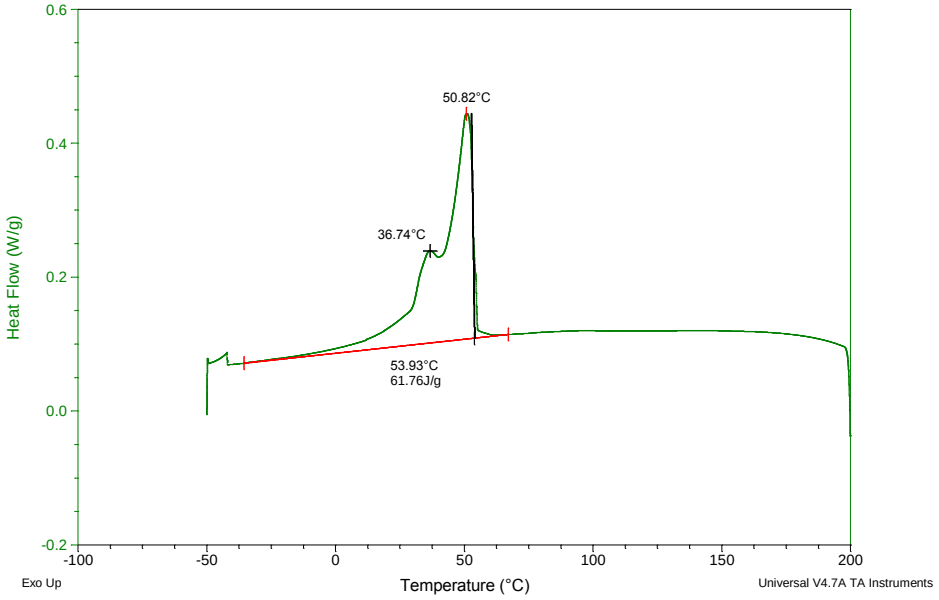
File: C:...\2008\08235\RC_08235_TERRA WAX.001
Operator: BL
Run Date: 12-Jan-2011 05:04
Instrument: DSC Q2000 V24.8 Build 120



Sample: RC/08235/TERRA WAX
Size: 7.1800 mg
Method: ISO 11357

DSC

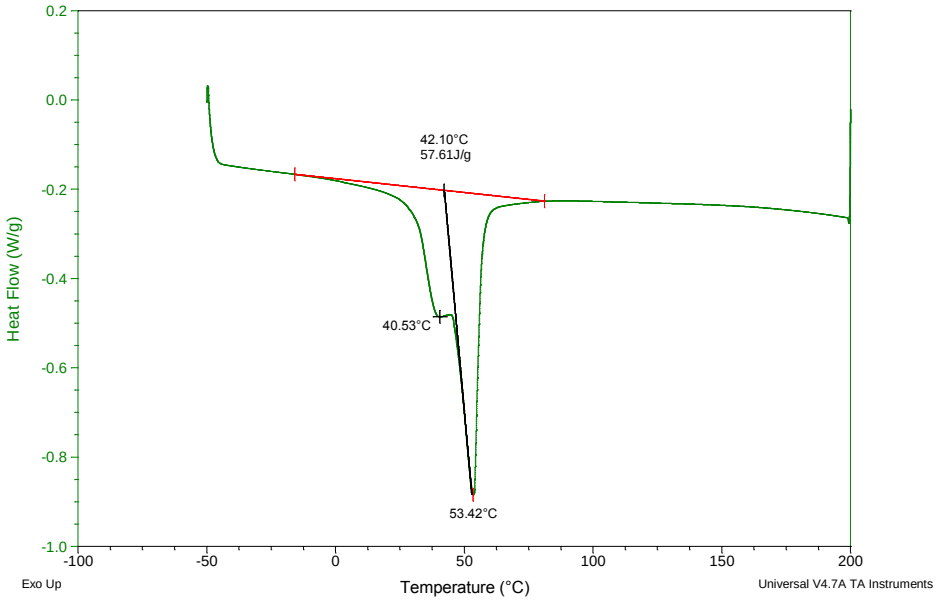
File: C:...\2008\08235\RC_08235_TERRA WAX.001
Operator: BL
Run Date: 12-Jan-2011 05:04
Instrument: DSC Q2000 V24.8 Build 120



Sample: RC/08235/TERRA WAX
Size: 7.1800 mg
Method: ISO 11357

DSC

File: C:...\2008\08235\RC_08235_TERRA WAX.001
Operator: BL
Run Date: 12-Jan-2011 05:04
Instrument: DSC Q2000 V24.8 Build 120



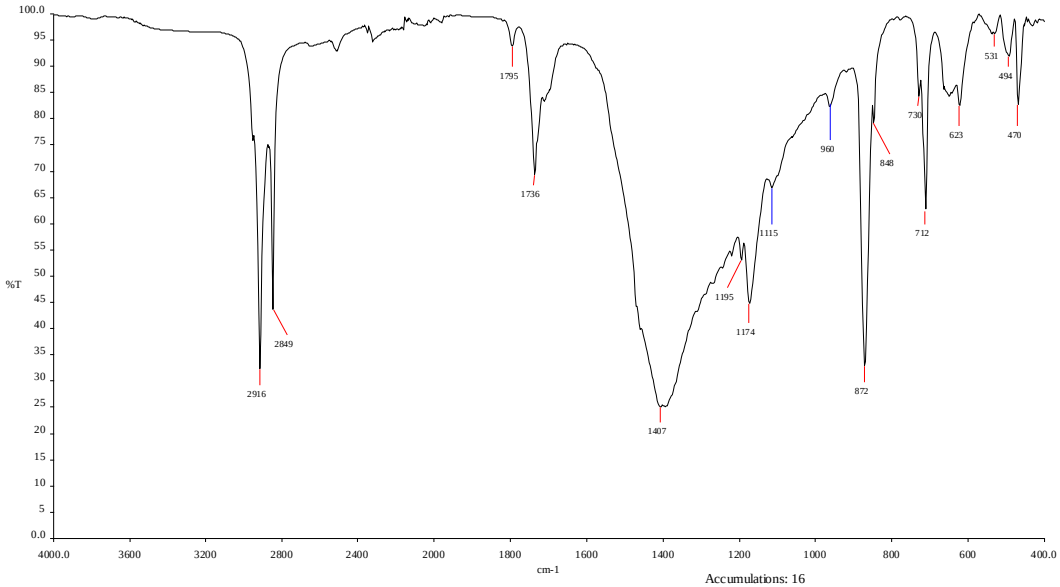
Appendix 4: FTIR Spectra and DSC thermograms of FAMOUS GREEN LABEL WARM

RESCOLL

Spectrum Pathname: C:\pel_data\spectra\6468.003

Date: 25/01/11

Opérateur : BL



Analyst: Admin

Description: RC/08235/FAMOUS Warm

Date Created: tue jan 25 08:10:49 2011

Accumulations: 16

Resolution: 4 cm-1

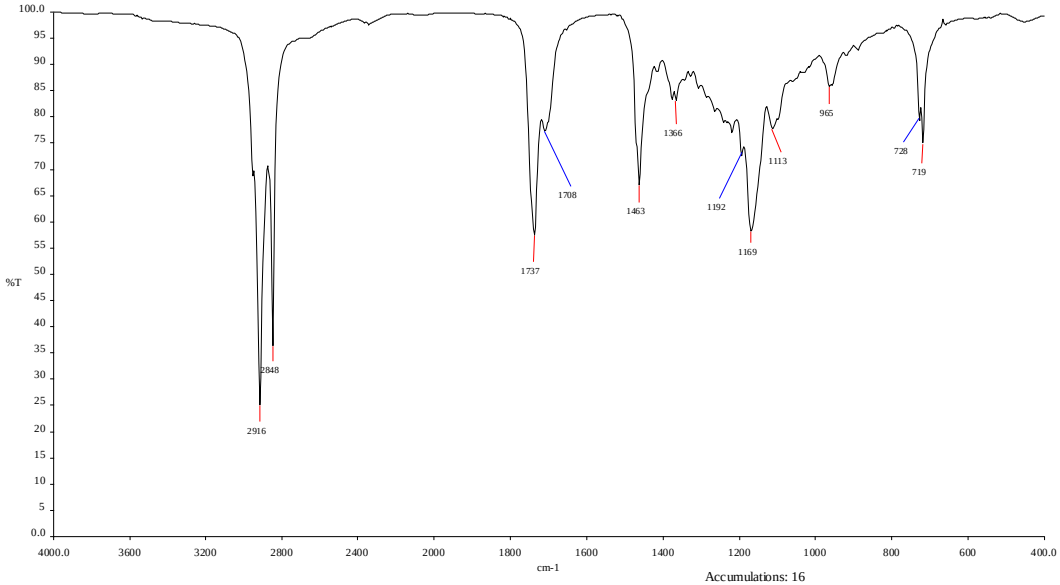
IR Accessory: Golden Gate

RESCOLL

Spectrum Pathname: C:\pel_data\spectra\6469.003

Date: 25/01/11

Opérateur : BL



Analyst: Admin

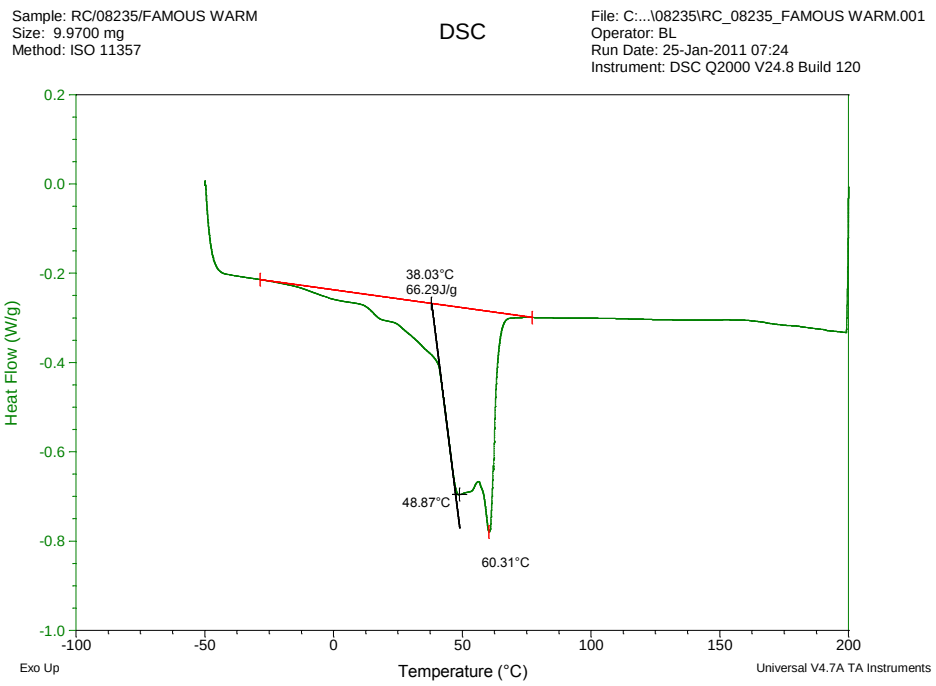
Description: RC/08235/FAMOUS Warm - extrait Cyclohexane

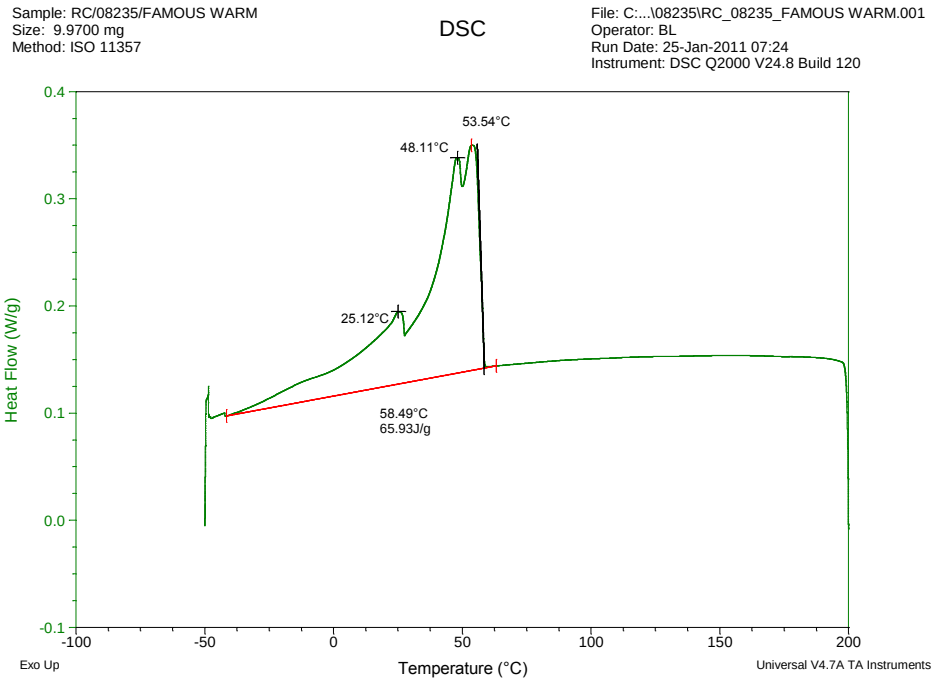
Date Created: tue jan 25 08:13:37 2011

Accumulations: 16

Resolution: 4 cm-1

IR Accessory: Golden Gate

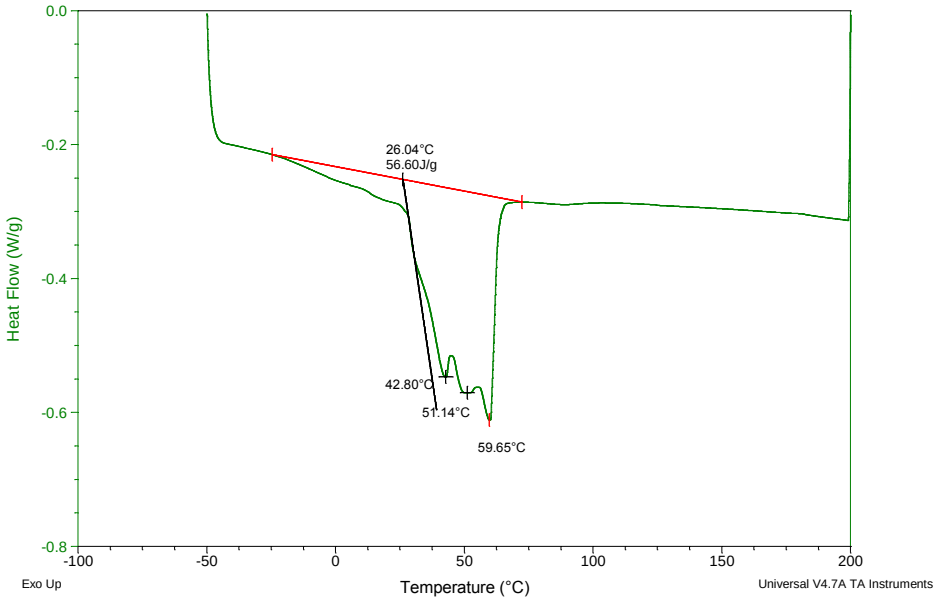

Sample: RC/08235/FAMOUS WARM
Size: 9.9700 mg
Method: ISO 11357
DSC
File: C:...\08235\RC_08235_FAMOUS WARM.001
Operator: BL
Run Date: 25-Jan-2011 07:24
Instrument: DSC Q2000 V24.8 Build 120
38.03°C
66.29J/g
48.87°C
60.31°C
Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.7A TA Instruments


Sample: RC/08235/FAMOUS WARM
Size: 9.9700 mg
Method: ISO 11357
DSC
File: C:...\08235\RC_08235_FAMOUS WARM.001
Operator: BL
Run Date: 25-Jan-2011 07:24
Instrument: DSC Q2000 V24.8 Build 120
53.54°C
48.11°C
25.12°C
58.49°C
65.93J/g
Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.7A TA Instruments

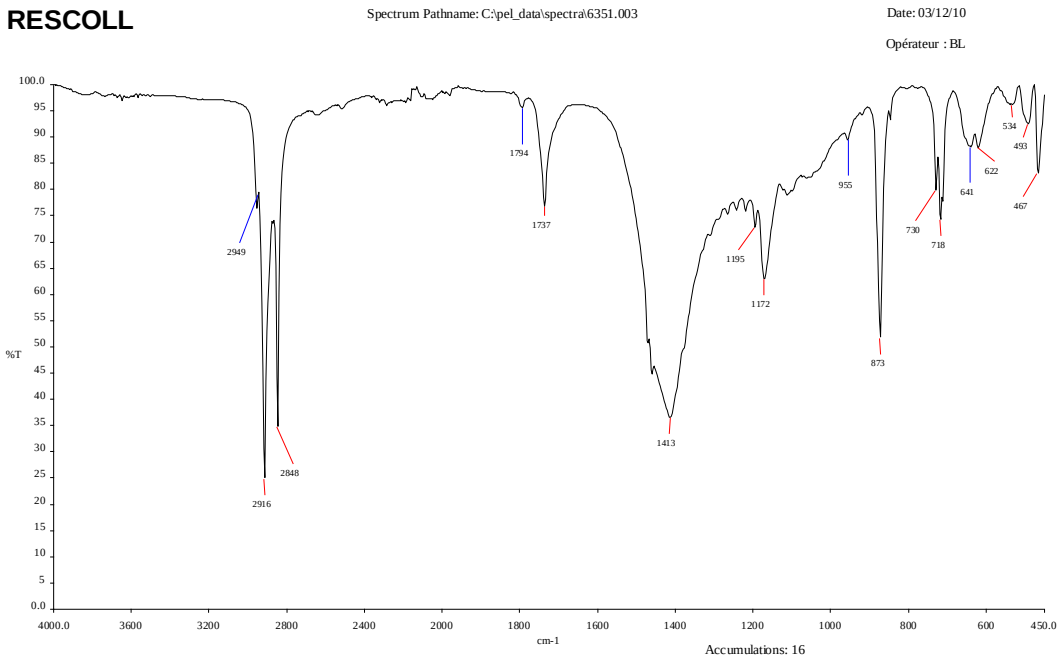
Sample: RC/08235/FAMOUS WARM
Size: 9.9700 mg
Method: ISO 11357

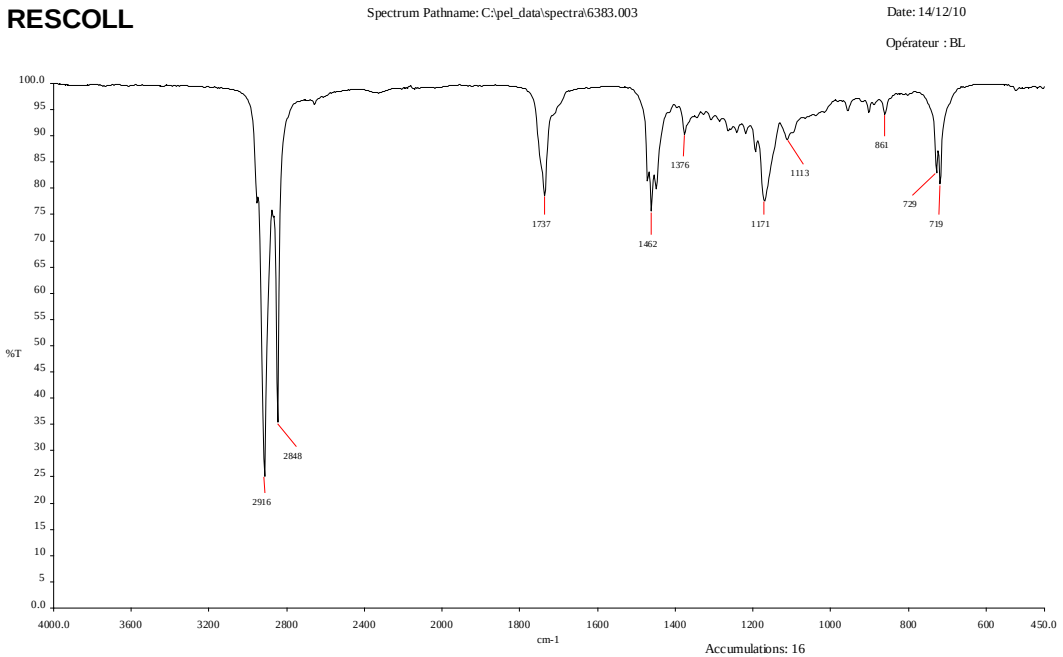
DSC

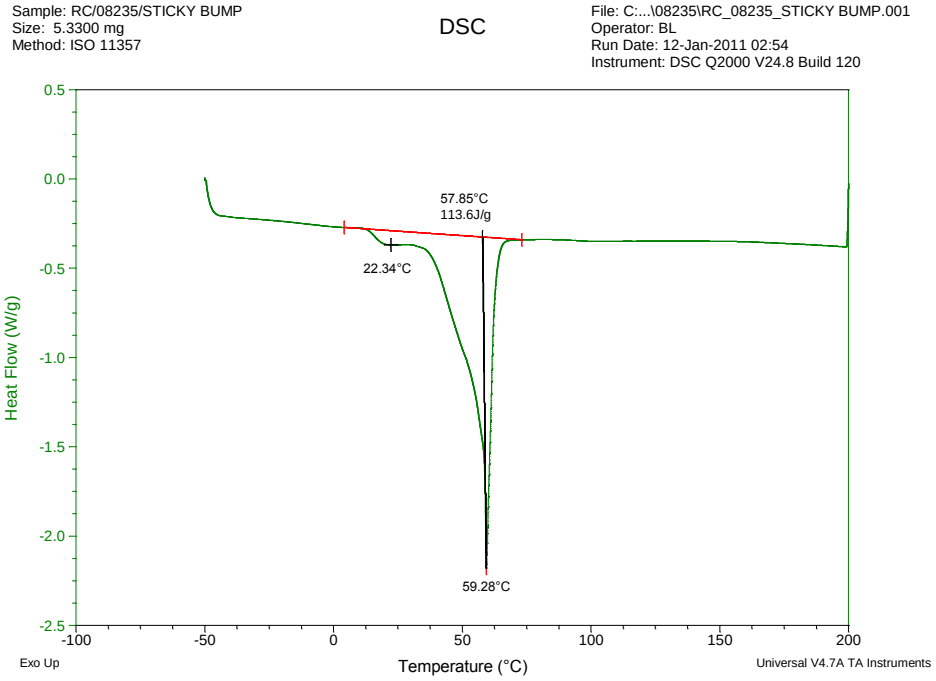
File: C:...\08235\RC_08235_FAMOUS WARM.001
Operator: BL
Run Date: 25-Jan-2011 07:24
Instrument: DSC Q2000 V24.8 Build 120

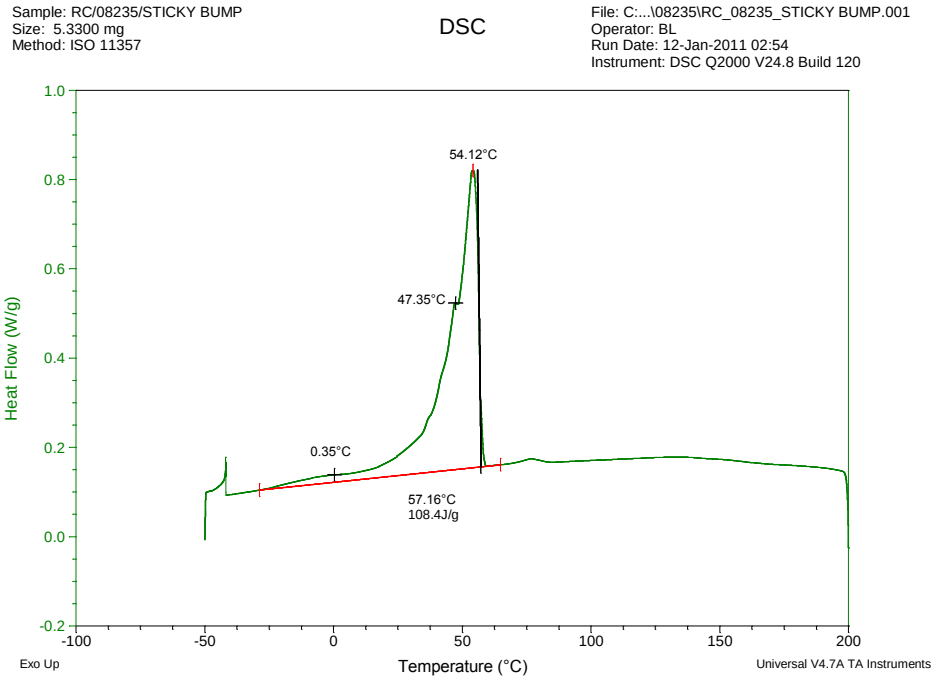


Appendix 5: FTIR Spectra and DSC thermograms DSC of STICKY BUMPS SOY WAX WARM





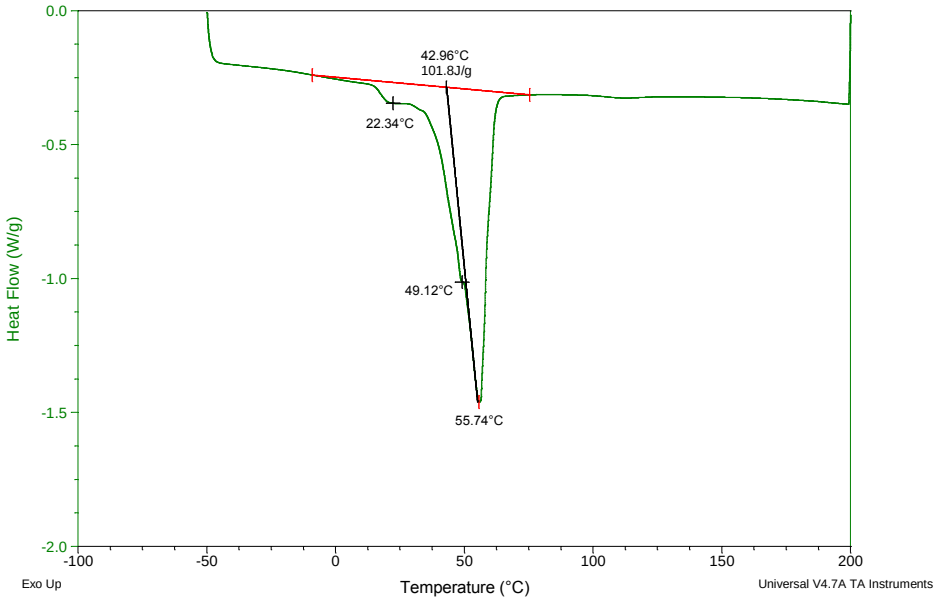

Sample: RC/08235/STICKY BUMP
Size: 5.3300 mg
Method: ISO 11357
DSC
File: C:...\08235\RC_08235_STICKY BUMP.001
Operator: BL
Run Date: 12-Jan-2011 02:54
Instrument: DSC Q2000 V24.8 Build 120
57.85°C
113.6J/g
22.34°C
59.28°C
Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.7A TA Instruments


Sample: RC/08235/STICKY BUMP
Size: 5.3300 mg
Method: ISO 11357
DSC
File: C:...\08235\RC_08235_STICKY BUMP.001
Operator: BL
Run Date: 12-Jan-2011 02:54
Instrument: DSC Q2000 V24.8 Build 120
54.12°C
47.35°C
0.35°C
57.16°C
108.4J/g
Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.7A TA Instruments

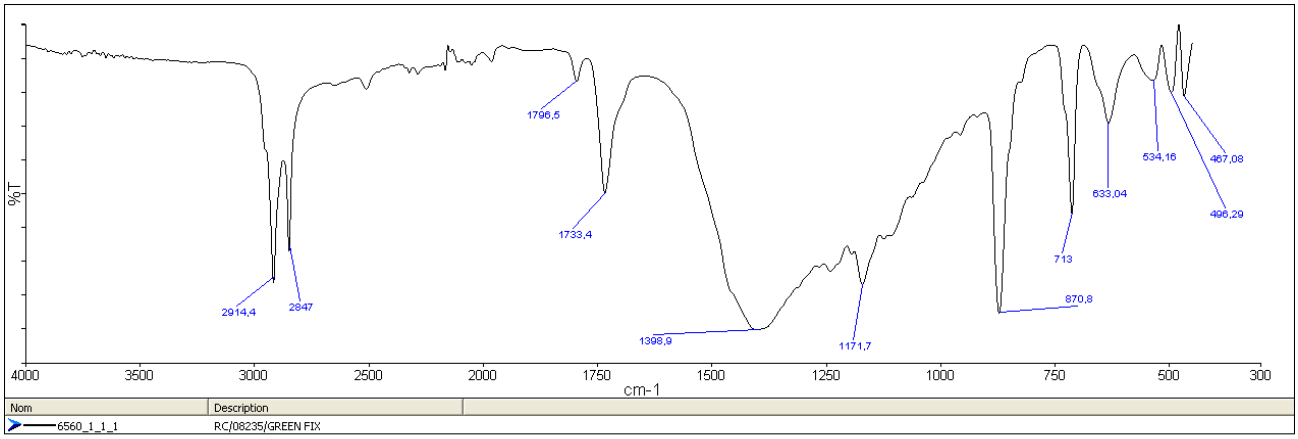
Sample: RC/08235/STICKY BUMP
Size: 5.3300 mg
Method: ISO 11357

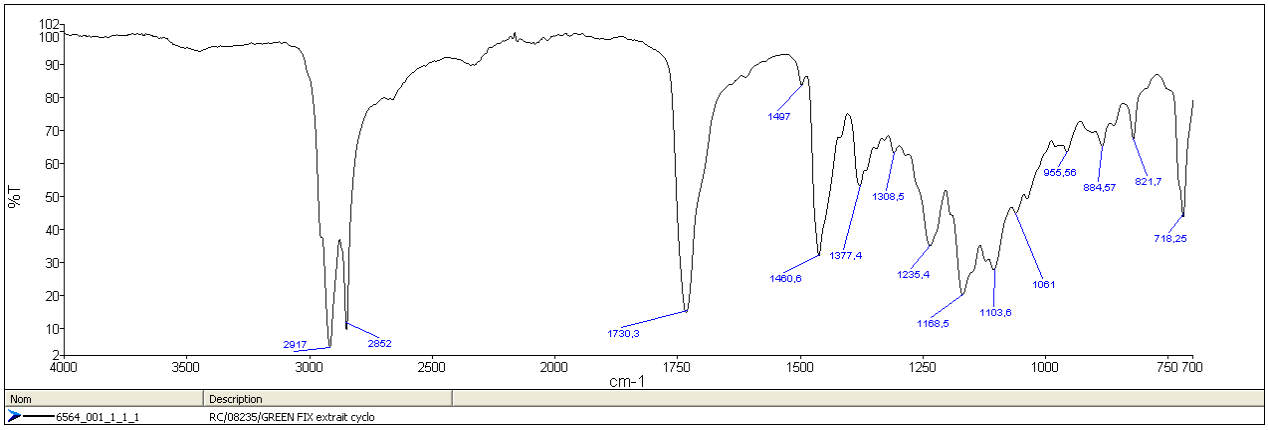
DSC

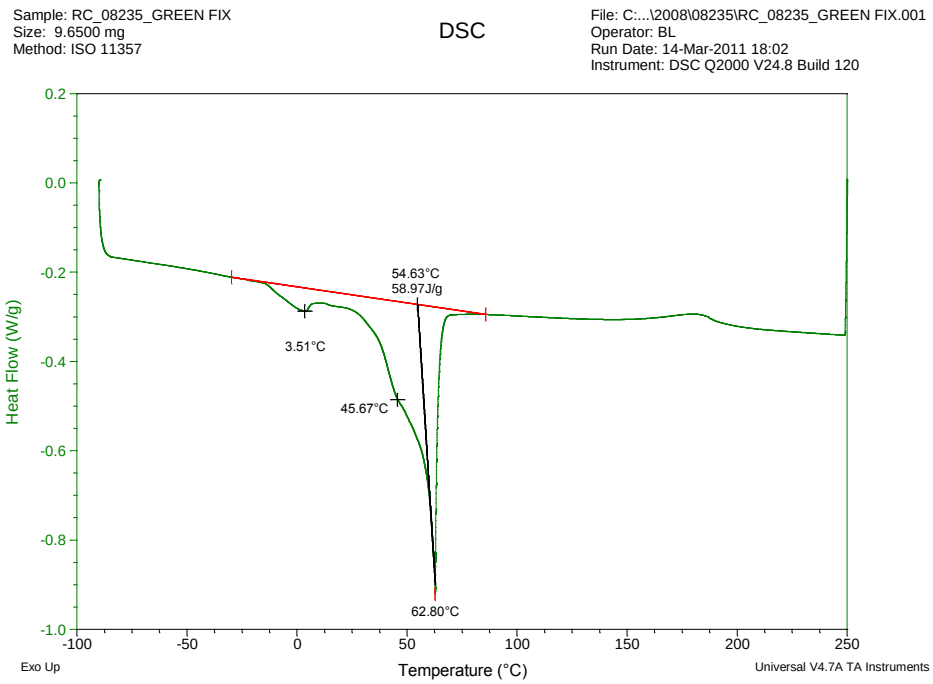
File: C:...\08235\RC_08235_STICKY BUMP.001
Operator: BL
Run Date: 12-Jan-2011 02:54
Instrument: DSC Q2000 V24.8 Build 120

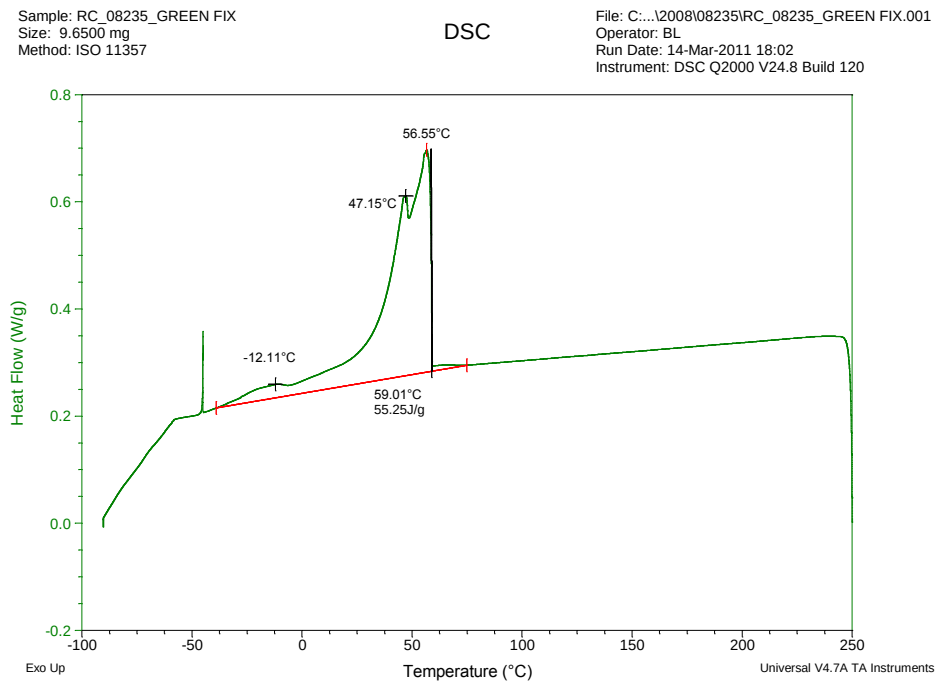


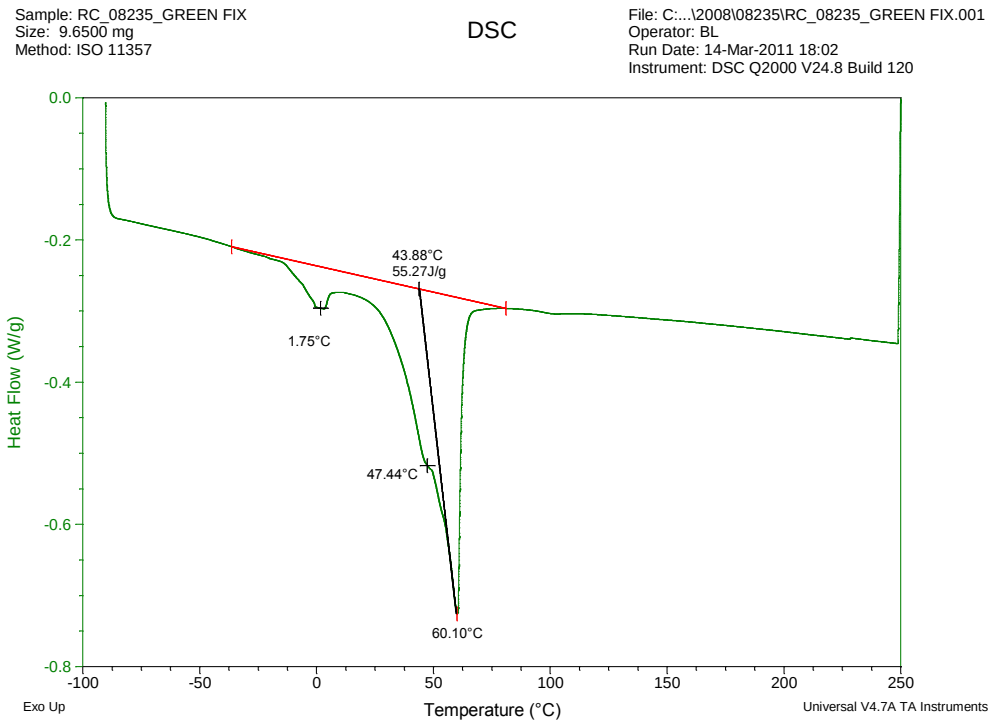
Appendix 6: FTIR Spectra and DSC thermograms of GREENFIX COLD








Sample: RC_08235_GREEN FIX
Size: 9.6500 mg
Method: ISO 11357
DSC
File: C:...\2008\08235\RC_08235_GREEN FIX.001
Operator: BL
Run Date: 14-Mar-2011 18:02
Instrument: DSC Q2000 V24.8 Build 120
56.55°C
47.15°C
-12.11°C
59.01°C
55.25J/g
Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.7A TA Instruments


Sample: RC_08235_GREEN FIX
Size: 9.6500 mg
Method: ISO 11357
DSC
File: C:...\2008\08235\RC_08235_GREEN FIX.001
Operator: BL
Run Date: 14-Mar-2011 18:02
Instrument: DSC Q2000 V24.8 Build 120
43.88°C
55.27J/g
1.75°C
47.44°C
60.10°C
Heat Flow (W/g)
Temperature (°C)
Exo Up
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