

**CASE STUDY – Identification and Quantification of Short Chain Polymer Branching**  
Polyethylene Branching Determination using  $^{13}\text{C}$  Nuclear Magnetic Resonance (NMR).

**OBJECTIVE**

The purpose of this work was to compare the short chain branching structure in a low density and a high density polyethylene sample.

**ANALYTICAL STRATEGY**

$^{13}\text{C}$  Nuclear Magnetic Resonance (NMR) was used to identify branching frequency and type.

**CONCLUSIONS**

The low density polyethylene sample was observed to have ~0.85% of ethyl branches, ~1.59 % of butyl branches and ~1.32% of hexyl branches. No branching was observed in the high density polyethylene sample.



# Final Report

Jordi Labs LLC  
Case Study

Polyethylene Branching Determination using  $^{13}\text{C}$  Nuclear Magnetic Resonance (NMR)

Date: xx/xx/xx

Released by:  
Dr. Mark Jordi  
President  
Jordi Labs LLC

Company Name Confidential





Date

Client Name  
Company Name

P: xxx-xxx-xxx  
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Dear Client,

Please find enclosed the test results for your samples described as:

1. High Density Polyethylene
2. Low Density Polyethylene

The following test was performed:

1. Nuclear Magnetic Resonance Spectroscopy (NMR)

## Objective

The goal of this analysis was to examine the short chain branching type and frequency in a high density polyethylene and a low density polyethylene.

## Summary of Results

Carbon NMR was successfully applied to determining the branching type and the estimated branch frequency for the two polymers. We observed ~0.85% of ethyl branches, ~1.59 % of butyl branches and ~1.32% of hexyl branches in the low density polyethylene. No branching was observed in the high density polyethylene sample.

## Individual Test Results

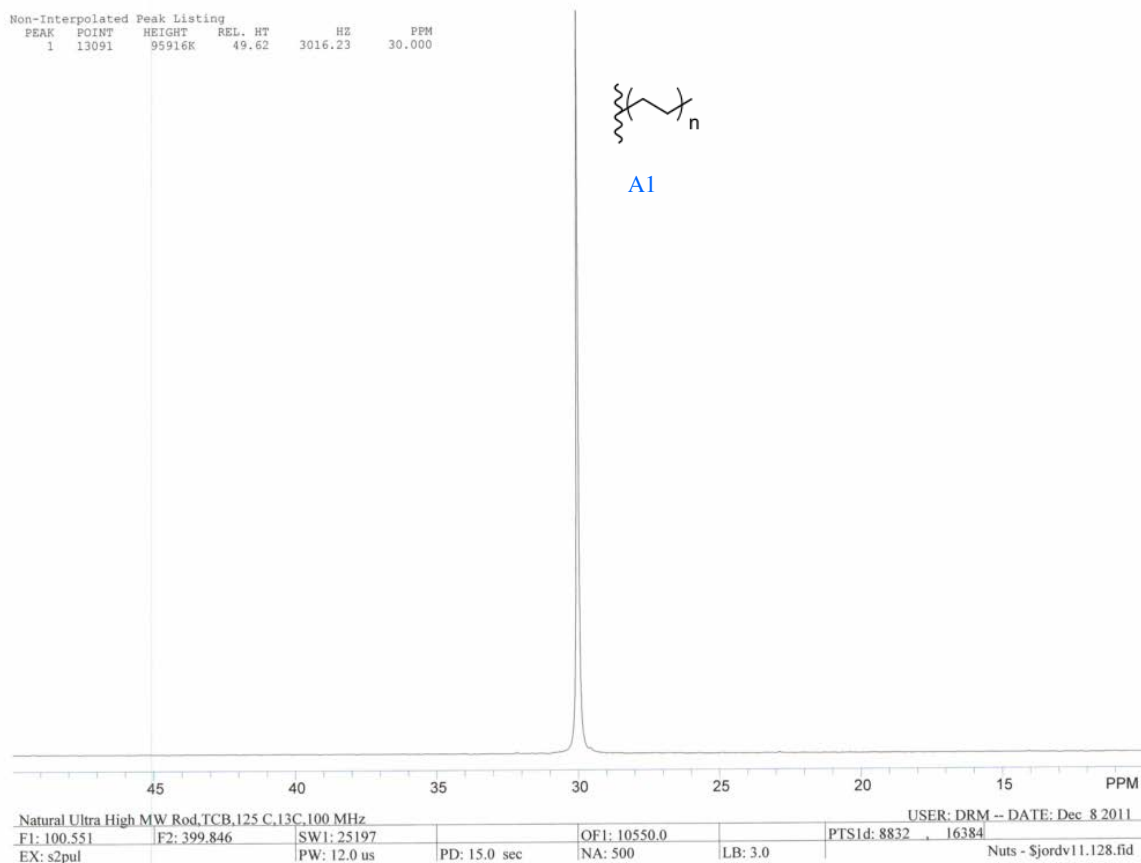
A summary of the individual test results is provided below. All accompanying data, including spectra, has been included in the data section of this report.

### <sup>13</sup>C NMR

<sup>13</sup>C-NMR spectra were acquired to determine the chemical composition of the materials.

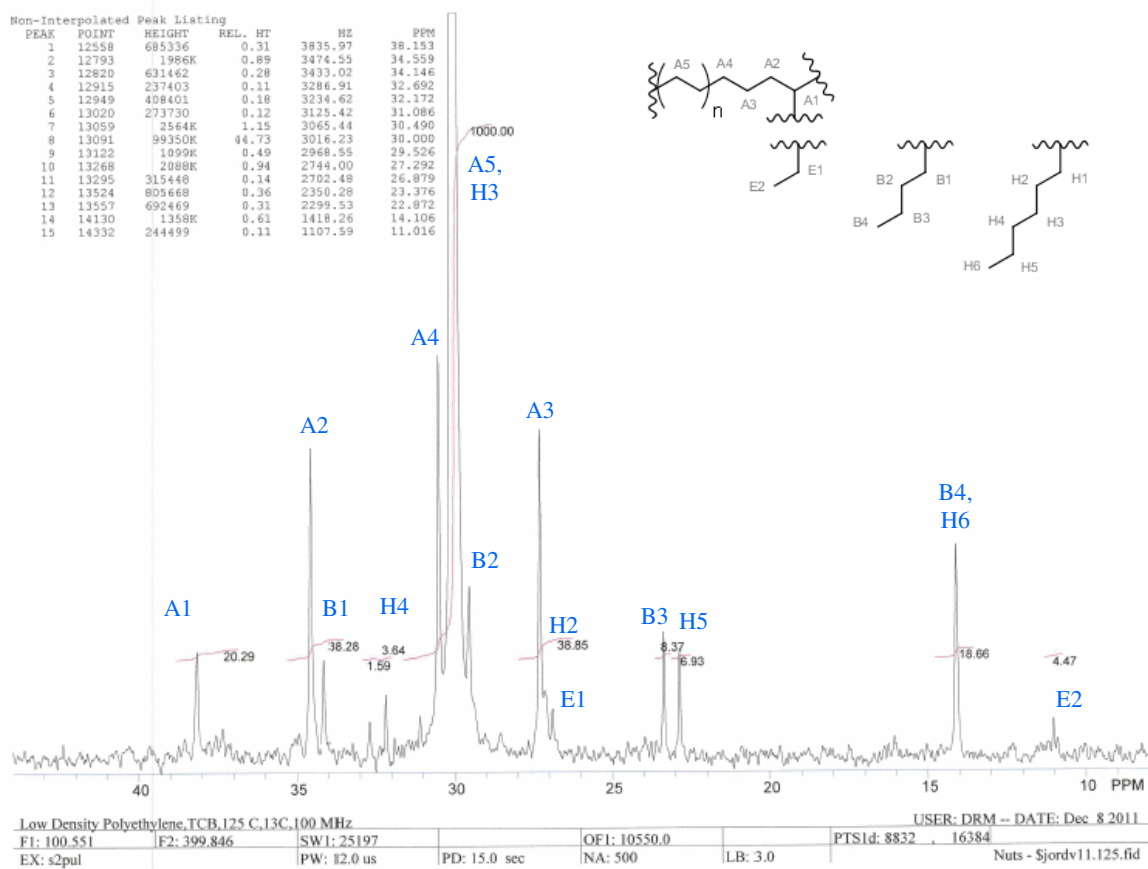
Sample 1: High Density polyethylene (HDPE) A single resonance was observed at 30.00 ppm (**Table 1** and **Figure 1**), which is consistent with a structure that contains only  $-\text{CH}_2-$  carbons, the expected structure for HDPE.

Table 1 High Density Polyethylene NMR Results	
Identification	Chemical Shift (ppm)
A1	30.00



**Figure 1.** Expansion of the aliphatic region for Sample 1, High Density polyethylene (HDPE)

**Sample 2: Low Density Polyethylene (LDPE).** **Table 2** contains the peak identifications for the LDPE sample, and an expansion of the region showing the polymer short chain branching structure is shown in **Figure 2**.



**Figure 2.** Expansion of the aliphatic region for Sample 2, Low Density Polyethylene (LDPE).

The largest resonance was observed at 30.00 ppm, consistent with a structure that contains predominantly internal  $-\text{CH}_2-$  carbons as expected for LDPE. A resonance for the backbone tertiary carbon (A1) is found at 38.15 ppm and integrates to about 2% of the main  $-\text{CH}_2-$  resonance indicating that approximately 4% of the repeat units contain branch points. The peak patterns are most consistent with branching that contains approximately equal parts of butyl ( $\text{C}_4$ ) and hexyl ( $\text{C}_6$ ) side chains, with a small portion of ethyl branches ( $\text{C}_2$ ).

Table 2 Low Density Polyethylene NMR Results		
Identification	Chemical Shift (ppm)	Integration
A1	38.15	20.29
A2, H1	(34.56)*	38.28(+B1)
A3, H2	(27.29)*	38.85 (+E1)
A4	30.49	~25 (1000-A5-H3-B2)
A5, H3	(30.00)*	1000-A4-B2
B1	34.15	38.28- A2-H1
B2	29.52	1000-A4-A5-H3
H4	32.69	3.64
H5, B3	22.87, 23.38	6.93, 8.37
H6, B4	14.11	18.66
E1	26.88	38.85-A3-H2
E2	11.02	4.47

\* overlapping resonances

The integration values for the methyl carbon on the ethyl branches (E2) and the alpha carbon (B3 and H5) on the butyl and hexyl branches can be used to estimate the percentage of each monomer:

The mole percent of each branch type in the polymer would be:

Ethyl = 0.85%

Butyl = 1.59%

Hexyl = 1.32%

The mole percent of ethylene would be:

Ethylene = 96.2%

## Analysis Conditions

### <sup>13</sup>C NMR

Information on the specific conditions used to perform the analysis are listed here.

## Closing Comments

Deformulation of an unknown material is intended to provide a best estimate of the chemical nature of the sample. All chemical structures are supported by the evidence presented but are subject to revision upon receipt of additional evidence. Additional factors such as material processing conditions may also affect final material properties.

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Jordi Labs specializes in polymer testing and has 30 years experience doing complete polymer deformulations. We are one of the few labs in the country specialized in this type of testing. We will work closely with you to help explain your test results and solve your problem. We appreciate your business and are looking forward to speaking with you concerning these results.

Sincerely,

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