Designation: D 5017 – 96 (Reapproved 2003)$^{\epsilon 1}$

Standard Test Method for Determination of Linear Low Density Polyethylene (LLDPE) Composition by Carbon-13 Nuclear Magnetic Resonance[1]

This standard is issued under the fixed designation D 5017; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

ϵ[1] NOTE—Editorially changed content in Sections 2 and 3 in June 2003.

1. Scope

1.1 This test method determines the molar composition of copolymers prepared from ethylene (ethene) and a second alkene-1 monomer. This second monomer can include propene, butene-1, hexene-1, octene-1, and 4-methylpentene-1.

1.2 Calculations of this test method are valid for products containing units EEXEE, EXEXE, EXXE, EXXXE, and of course EEE where E equals ethene and X equals alkene-1. Copolymers containing a considerable number of alkene-1 blocks (such as, longer blocks than XXX) are outside the scope of this test method.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See Section 8 for a specific hazard statement.

NOTE 1—There is no equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

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[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods .

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This revision includes the addition of an ISO equivalency statement and an update of model numbers of referenced NMR instruments.

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[2]
E 380 Practice for Use of the International System of Units (SI)[2]
E 386 Practice for Data Presentation Relating to High-Resolution Nuclear Magnetic Resonance (NMR) Spectroscopy[3]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]
IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern System[4]

3. Terminology

3.1 Some units, symbols, and abbreviations used in this test method are summarized in Practices E 380 IEEE/ASTM SI-10 and Practice E 386. Other abbreviations are listed as follows:

3.2 *Abbreviations:Abbreviations:*

3.2.1 ^{13}C—carbon 13,

3.2.2 LLDPE—linear low-density polyethylene,

3.2.3 ODCB—*ortho*-dichlorobenzene,

3.2.4 F1—transmitter frequency,

3.2.5 T1—relaxation

3.2.3 T1—relaxation time, and

3.2.64 TR—pulse repetition time.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 With a few modifications, terms used to designate different carbon types were suggested by Carman.[5] Methine carbons are identified by CH and branch carbons are labeled according to branch type as summarized in Table 1. Branch carbons are numbered starting with the methyl as number one.

3.3.2 Backbone methylene carbons are designated by a pair of Greek letters that specify the location of the nearest methine carbon in each direction. For example, α,α-methylene carbon is between two methine carbons or an α,δ^{+} methylene carbon has one immediate methine neighbor and the second methine carbon is located at least four carbons away.

4. Summary of Test Method

4.1 Polymer samples are dispersed in hot solvent and analyzed at high temperatures using Carbon-13 nuclear magnetic resonance (NMR) spectroscopy.

4.2 Spectra are recorded under conditions such that the response of each chemically different carbon is identical. Integrated responses for carbons originated from the different comonomers are used for calculation of the copolymer composition.

5. Significance and Use

5.1 Performance properties are dependent on the number and type of short chain branches. This test method permits measurement of these branches for ethylene copolymers with propylene, butene-1, hexene-1, octene-1, and 4-methylpentene-1.

6. Apparatus

6.1 *NMR Spectrometer*, ^{13}C pulse-Fourier transform with field strength of at least 2.35 T. Typical instruments include the Jeol GSX-400, Bruker Avance O DPX-300, and Varian 400 Unity PLUS spectrometers.

NOTE 2—The system should have a computer size of at least 32 K for 50-MHz carbon frequency with digital resolution of at least 0.5 Hz/point in the final spectrum.

6.2 *Sample Tubes,*[6] 10-mm outside diameter.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.
[3] *Annual Book of ASTM Standards*, Vol 14.01.
[4] Carman, C. J., Harrington, R. A., and Wilkes, C. E., *Macromolecules* 1977, Vol 10, p. 536.
[4] Available from ASTM International Headquarters, 100 Barr Harbor Drive, C700, West Conshohocken, PA 19428.
[5] Available from Wilmad Scientific Glass Co.
[5] Carman, C. J., Harrington, R. A., and Wilkes, C. E., *Macromolecules* 1977, Vol 10, p. 536.
[6] Vidrime, D. W., and Peterson, P. E., *Analytical Chemistry* , Vol 48, 1976, p. 1301.
[6] Available from Wilmad Scientific Glass Co.

TABLE 1 Designations for Different Carbon Types

Monomer		Branch Type	Label
Propane	(P)	methyl	M1
Butene-1	(B)	ethyl	E1–E2
Hexene-1	(H)	butyl	B1–B4
4-Methylpentene-1	(MP)	isobutyl	IB1–IB3
Octene-1	(O)	hexyl	H1–H6

NOTE 3—Sample tube size can be varied; however, the sample preparation procedure described in 10.1 may need to be altered to maintain the minimum signal-to-noise requirement of 9.4.

7. Reagents and Materials

7.1 *Ortho-dichlorobenzene or 1,2,4-trichlorobenzene* , reagent grade

7.2 *Deuterated o-dichlorobenzene or p-dichlorobenzene*. This material is used at a concentration up to 20 % with the reagent specified in 7.1 as an internal lock.

8. Hazards

8.1 **Precaution:** Solvents should be handled in a wellventilated fume hood.

9. Instrument Parameters

9.1 Pulse angle, 90°
9.2 Pulse repetition, 10 s
9.3 Sample temperature, 130°C

NOTE 4—The precise temperature should be measured using the NMR thermometer (cyclooctane/methylene iodide).[7]

9.4 Minimum signal-to-noise, 5000:1

NOTE 5—The signal-to-noise ratio is defined as 2.5 times the signal intensity of the 30.0-ppm peak (isolated methylenes) divided by the peak to peak noise for the region from 50 to 70 ppm. Calculation of signal-to-noise is permitted using an equivalent software procedure.

9.5 Sweep width, 175 ppm
9.6 Transmitter frequency (F1), 50 to 55 ppm
9.7 Apodisation, 2 (exponential) Hz
9.8 Pulse width, $<[4 \times \text{sweep width Hz}]^{-1}$
9.9 Decoupling, complete

NOTE 6—The nuclear overhauser enhancement for the carbons used for quantitative analysis have been shown to be full.[4, 7, 8]

10. Procedure

10.1 Weigh a 1.2-g sample into a 10-mm NMR tube. Add 1.5 mL of solvent (7.1) and 1.3 mL deuterated solvent (7.2) to the tube. Cap the tube.

NOTE 7—Solution concentration can be varied with instruments of different field strength as long as one meets the minimum signal-to-noise requirement of 9.4.

10.2 Homogenize the sample in an oven at 150°C for 3 to 4 h. Keep the tube in ~~an almost horizontal~~ a vertical position during the heating step.

10.3 Set spectrometer parameters as detailed in Section 9.

10.4 Transfer the tube to the NMR spectrometer and equilibrate 10 to 15 min at 130°C.

10.5 Scan the sample with complete broadband decoupling using the parameters of Section 9.

10.6 Record the spectrum and the accurate full-scale integral from 10 to 50 ppm. Adjust partial integrals so that integral of the second largest peak in the spectrum is at least 50 % of full-scale. This partial integral must be flat before and after the area to be measured.

NOTE 8—The combination of sample preparation time and acquisition time necessary to obtain the signal-to-noise requirement of 9.4 can lead to prohibitively long experiments if samples are run multiplicatively. It is acceptable to perform sample determinations using a single analysis. Duplicate runs in accordance with 13.1 were performed for the round-robin exercise.

11. Calculation

11.1 Measure the area between the appropriate integration limits outlines in Annex A1.

11.2 Substitute the integrals into the appropriate equations from Annex A2 to calculate the mole percent alkene-1.

11.3 Annex A3 gives a sample calculation for an ethylene-octene copolymer using integrals and equations in accordance with 11.1 and 11.2.

NOTE 9—With the prescribed repetition time (10 s) and pulse angle (90°), the maximum allowable relaxation time (T_1) for carbons used for quantitative analysis is 2 s. To shorten the analysis time, a shorter pulse repetition time can be used if one accounts for the relaxation time differences. Relaxation

~~[7] Randall, J. C., “NMR~~

[7] Vidrime, D. W., and ~~Macromolecules,” Chapter 9,~~ Peterson, P. E., ~~*American Chemical Society Symposium Series 247*~~*Analytical Chemistry* , ~~1984.~~ *Vol 48, 1976, p. 1301.*

~~[8] Farrar, T.~~

[8] Randall, J. C., “NMR and ~~Becker, E. D.,~~ Macromolecules,” Chapter 9, ~~*Pulse and Fourier Transform NMR*~~*American Chemical Society Symposium Series 247*, ~~*Chapter 2, Academic Press, New York, 1971.*~~ *1984.*

times of carbons for the five copolymers were determined at a carbon frequency of 50 MHz using the inversion recovery method.[9, 10] Annex A4 summarizes these relaxation times and correction factors (reciprocal of the relative intensities) for a 4-s repetition time (T_R). With the shorter T_R, multiply integrals by these correction factors before using the equations in Annex A2. The T_1 values would have to be remeasured for analyses performed at spectrometer frequencies other than 50 MHz.

11.4 If desired, convert results from mole percent alkene-1 to branches per 1000 carbons (br/1000C) using the equations in Annex A5.

12. Report

12.1 Report the mole percent alkene from 11.2 or branches/1000C from 11.4, or both.

13. Precision and Bias

13.1 Table 2 is based on a round robin[11] conducted in 1988 in accordance with Practice E 691, involving nine materials tested by six laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each "test result" was the average of two individual determinations. Each laboratory obtained one test result for each material.

NOTE 10—**Caution:** The following explanations of *r* and *R* (13.2-13.2.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 2 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 13.2-13.2.3 would then be valid for such data.

13.2 *Concept of r and R*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing two specimens:

13.2.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

13.2.2 *Reproducibility Limit, R (Comparing Two Test Results for the Same Material, Obtained by Different Operators Using Different Equipment in Different Laboratories)*—The two test results should be judged not equivalent if they differ by more than the " *R*" value for that material.

13.2.3 Any judgment in accordance with 13.2.1 or 13.2.2 would have an approximate 95 % (0.95) probability of being correct.

13.3 There are no recognized standards by which to estimate bias of this test method.

~~[9] Cheng, H. N.,~~

[9] Farrar, T. C., and ~~Bennet, M. A.,~~ Becker, E. D., ~~*Macromolecule Chemistry*~~ *Pulse and Fourier Transform NMR*, ~~*Vol 188, 1987, pp. 2665–2677.*~~ *Chapter 2, Academic Press, New York, 1971.*

~~[10] Supporting data are available from ASTM Headquarters. Request RR: D-20-1192.~~

[10] Cheng, H. N., and Bennet, M. A., *Macromolecule Chemistry* , Vol 188, 1987, pp. 2665–2677.

[11] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods .

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This revision includes the addition of an ISO equivalency statement and an update of model numbers of referenced NMR instruments.

TABLE 2 Precision Statistics for Determination of Mole Percent Branching in LLDPE Copolymers by Carbon-13 NMR Spectroscopy

Sample	Comonomer	Average Mole,%	Expressed as % of the Average			
			V_r[A]	V_R[B]	r[C]	R[D]
A	butene	4.72	11.1	11.5	31.1	32.2
B	butene	4.22	11.9	11.9	33.3	33.3
C	hexene	3.64	17.1	18.2	47.9	51.0
D	hexene	4.03	14.3	14.3	40.0	40.0
E	octene	5.18	10.3	10.3	28.8	28.8
F	octene	0.76	27.5	40.6	77.0	113.7
G	4-methyl-pentene	5.00	14.0	14.8	39.2	41.4
H	4-methyl-pentene	1.26	37.4	38.2	104.7	107.0
I	propene	15.96	7.3	7.6	20.4	21.3

[A] V_r = within laboratory coefficient of variation for the indicated material. It is obtained by pooling the within laboratory standard deviations of the following test results:

$$Sr = [[\Sigma(s_1)^2 + (s_2)^2 \ldots + (s_n)^2]/n]^{1/2}$$

$$Vr = 100 \times (\text{Sr divided by the overall average for the material}).$$

[B] V_R = between laboratories reproducibility, expressed as coefficient of variation, for the indicated material.

[C] r = within laboratory repeatability limit = 2.8 × V_r .

[D] R = between laboratories reproducibility limit = 2.8 × V_R .

14. Keywords

14.1 carbon-13 NMR; composition; LLDPE; polyethylene

ANNEXES

(Mandatory Information)

A1. AREA BETWEEN THE APPROPRIATE INTEGRATION LIMITS OUTLINES

A2. EQUATIONS FOR CALCULATING MOLE % COMPOSITION

A2.1 Ethene-Propene Copolymers

A2.1.1 *Moles Propene*:

$$P_1 = \alpha - \text{carbons: } (2A + B)/2 \quad \text{(See Note A2.1)} \tag{A2.1}$$

$$P_2 = \text{CH carbons: } 2A + C - H \tag{A2.1}$$

$$P' = \text{average moles propylene: } (P_1 + P_2)/2 \tag{A2.1}$$

A2.1.2 *Moles Ethene*:–
:

$$E' = (C + D + E + F - A)/2 \tag{A2.2}$$

~~Mole % propene = 100 % × P′ + E′)~~
(See Note A2.2) (A2.2)

$$\text{Mole \% propene} = 100\,\% \times (P' + E') \quad \text{(See Note A2.2)} \tag{A2.2}$$

TABLE A1.1 Integration Limits for Ethylene Copolymers[A]

Copolymer	Area	Region, ppm
Ethene-propene	A	47.5 to 44.5
	B	39.8 to 36.8
	C	35.5 to 32.5
	C + D + E	35.5 to 25.8
	F	25.8 to 23.8
	G	22.5 to 18.5
	H	Peak at 21.6
Ethene-butene-1	A	41.5 to 38.5
	A′	Peak at 39.4
	B	37.8 to 36.8
	C	36.0 to 33.2
	D + E	33.2 to 25.5
	F	25.2 to 24.0
Ethene-hexene-1	A	41.5 to 40.5
	B	40.5 to 39.5
	C	39.5 to 37.0
	D	Peak at 35.8
	D + E	36.8 to 33.2
	F + G	33.2 to 25.5
	G	28.5 to 26.5
	H	24.9 to 24.1
Ethene-octene-1	A	41.5 to 40.5
	B	40.5 to 39.5
	C	39.5 to 37.0
	D	Peak at 35.8
	D + E	36.8 to 33.2
	F + G + H	33.2 to 25.5
	H	28.5 to 26.5
	I	25.0 to 24.0
	P	24.0 to 22.0
Ethene-4-methylpentene-1	A	46.5 to 43.5
	B	43.0 to 41.8
	C	41.8 to 40.5
	D	37.5 to 34.2
	E	Peak at 33.7
	F + G	33.2 to 25.2
	G	28.0 to 25.2
	H	Peak at 24.1

[A] Isolated methylene carbons at 30.0 ppm.

A2.2 Ethene-Butene-1 Copolymers

A2.2.1 *Moles Butene-1*:~~–~~

$$B_1 = \alpha - \text{ carbons: } (2A + B)/2 \qquad \text{(See Note A2.1)} \tag{A2.3}$$

$$B_2 = CH \text{ carbons: } (A' + 2C + 2B)/4 \tag{A2.3}$$

$$B' = \text{average moles butene} - 1\text{: } (B_1 + B_2)/2 \tag{A2.3}$$

A2.2.2 *Moles ethene*:~~–~~

$$E' = (2D + 2E + 2F - A' - B)/4 \tag{A2.4}$$

$$\text{Mole \% butene} - 1 = 100\ \% \times B'/(B' + E') \quad \text{(See Note A2.2)} \tag{A2.4}$$

A2.3 Ethene-Hexene-1 Copolymers

A2.3.1 *Moles Hexene-1*:

$$H_1 = \alpha - \text{carbons: } [1.5A + 2B + (D + E) - D]/3 \quad \text{(See Note A2.1)} \tag{A2.5}$$

$$H_2 = \text{CH carbons: } (A + 2C + 2D)/2 \tag{A2.5}$$

$$H' = \text{average moles Hexene} - 1\text{: } (H_1 + H_2)/2 \tag{A2.5}$$

A2.3.2 *Moles ethene*:~~–~~

$$E' = [(F + G) - 3A - 3B - G - H]/2 + H' \tag{A2.6}$$

~~Mole % hexene − 1 = 100 % × E′/(E′ + H′)~~

(See Note A2.2) (A2.6)

$$\text{Mole \% hexene} - 1 = 100\ \% \times H'/(E' + H') \quad \text{(See Note A2.2)} \tag{A2.6}$$

A2.4 Ethene-Octene-1 Copolymers

A2.4.1 *Moles Octene-1*:

$$O_1 = \alpha - \text{carbons: } (A + 2C + 2D)/2 \quad \text{(See Note A2.1)} \tag{A2.7}$$

$$O_2 = CH \text{ carbons: } [1.5A + 2B + (D + E) - D]/3 \tag{A2.7}$$

$$O' = \text{average moles Octene} - 1 = (O_1 + O_2)/2 \tag{A2.7}$$

A2.4.2 *Moles Ethene*:~~–~~

$$E' = [(F + G + H) - (3A + 3B + H + P + I)]/2 + O' \tag{A2.8}$$

$$\text{Mole \% octene} - 1 = 100\ \% \times O'/(O' + E') \quad \text{(See Note A2.3)} \tag{A2.8}$$

A2.5 Ethene-4-Methylpentene-1 Copolymers

A2.5.1 *Moles 4-Methylpentene-1*:~~–~~

$$MP_1 = \alpha - \text{carbons} + \text{CH} - \text{branch carbons:} \tag{A2.9}$$

$$= (2B + C + D + 1.5E)/3 \tag{A2.9}$$

$$MP_2 = CH_2 \text{ branch carbon: } A \tag{A2.9}$$

$$MP' = \text{average moles 4} - \text{Methylpentene} - 1 = (MP_1 + MP_2)/2 \tag{A2.9}$$

A2.5.2 *Moles Ethene*:~~–~~

$$E' = [(F + G) - (2B + 1.5E + G + H)]/2 + 1.5MP' \tag{A2.10}$$

$$\text{Mole 5 4} - \text{methylpentene} - 1 = 100\ \% \times MP'/(MP' + E') \tag{A2.10}$$

NOTE A2.1—"α-carbons" and "CH carbons" mean predominantly peaks originating from α-carbons and CH-carbons, respectively.

NOTE A2.2—As their relaxation times are completely different, end groups are not included in the area measurement. The formal correction for this omission is as follows:

$$\text{End group concentration in mole percent} = (4 \times 1400)/Mn$$

where Mn = number average molecular weight.

NOTE A2.3—In the case of octene-1 copolymers, the end group concentration in mole percent = (6 × 1400)/ *Mn*.

A3. SAMPLE CALCULATION FOR AN ETHYLENE-OCTENE COPOLYMER

A3.1

TABLE A3.1 Sample Calculation for an Ethene-Octene-1 Copolymer[A]

Area	Integral
A	0.0
B	0.2
C	8.1
D	0.6
D + E	26.8
F + G + H	343.6
H	24.8
I	0.6
P	7.9

[A] Calculation for 90° pulse angle and 10-s repetition time.

$$O_1 = \frac{A + 2C + 2D}{2} = \frac{0.0 + 16.2 + 1.2}{2} = 8.70 \quad \text{(A3.1)}$$

A3.2

$$O_2 = \frac{[1.5A + 2B + (D + E) - D]}{3} \quad \text{(A3.2)}$$

$$= \frac{0.0 + 0.4 + 26.8 - 0.6}{3} = 9.27 \quad \text{(A3.2)}$$

A3.3

$$O' = \frac{O_1 + O_2}{2} = 8.98 \quad \text{(A3.3)}$$

A3.4

$$E' = \frac{[(F + G + H) - (3A + 3B + H + P + I)]}{2} + O \quad \text{(A3.4)}$$

A3.5

$$E' = \frac{[343.6 - (0.0 + 0.6 + 24.8 + 7.9 + 0.0]}{2} \quad \text{(A3.5)}$$

$$+ 8.98 = 164.13 \quad \text{(A3.5)}$$

A3.6

$$\text{Mole \% octene}-1 = \frac{(100)O}{O + E} = \frac{898.0}{173.11} = 5.19 \quad \text{(A3.6)}$$

A3.7

$$\text{br/1000C} = \frac{(\text{mole \% octene}-1)(1000)}{2\,(\text{mole \% ethene}) + 4(\text{mole \% octente})} \quad \text{(A3.7)}$$

$$= \frac{5190}{2(94.81) + 8(5.19)} = 22.41 \quad \text{(A3.7)}$$

A4. CONVERSION FROM MOLE % ALKENE-1 TO BRANCHES PER 1000 CARBONS

A4.1

$$\text{br/1000 carbons} = \frac{1000(\text{mole \% comonomer})}{2\ (\text{moles ethene}) + n\ (\text{mole \% comonomer})}$$

where n = number of carbons in the comonomer.

APPENDIX

(Nonmandatory Information)

X1. RELAXATION TIMES AND CORRECTION FACTORS

X1.5. See Tables X1.1- FACTORS

TABLE X1.1 Ethene-Propene Copolymers[A]

Area	Chemical Shift, ppm	EEXEE		EEXEXEE		EEXXEE		EEXXXEE		T1, s	Factor
		Type	B	Type	B	Type	B	Type	B		
A	~45.5					αα	1	ααγ	2	1.38	1.06
B	~37.5–37.9	α	2	α,αγ	4	αγ	2	αγ	2	1.38	1.06
C	33.2	CH	1	CH	2					2.14	1.18
D	30.4–31.5	γ	2	γ	2	CHβ,γ	4	CHβ, γ	4	1.56	1.08
	30.0	δ,δ[C]	3	δ, δ[C]	3	δ,δ[C]	3	δ, δ[C]	3	1.64	1.10
	~28							CHββ	1	2.14	1.18
E	27.4	β	2	β	2	β	2	β	2	1.54	1.08
F	24.8			ββ	1					1.54	1.08
G	20.0–21.6	M1	1	M1	2	M1	2	M1	3	2.96	1.35

[A] Correction factors valid for 90° pulse and 4-s repetition time.

[B] Number of carbons.

[C] Referenced to isolated methylenes at 30.0 ppm.

TABLE X1.2 Ethene-Butene-1 Copolymers[A]

Area	Chemical Shift, ppm	EEXEE		EEXEXEE		EEXXEE		EEXXXEE		T1, s	Factor
		Type	B	Type	B	Type	B	Type	B		
A	39.0–39.6	CH	1	CH	2	αα	2		1.91	1.14	
A	~39.4							ααγ	2	1.23	1.04
B	~37.2					CHβ	2	CHβ	2	1.91	1.14
C	34–35	α	2	α,αγ	4	αγ	2	CHββ, αγ	3	1.23	1.04
D	30.4	γ	2	γ	2	γ	2	γ	2	1.51	1.08
	30.0	δ, δ[C]	3	δ,δ[C]	3	δ, δ[C]	3	δ,δ[C]	3	1.64	1.09
E	27.3	β	2	β	2	β	2	β	2	1.46	1.07
	26.7	E2	1	E2	2	E2	2	E2	3	1.56	1.08
F	24.6			ββ	1					1.46	1.07

[A] Correction factors valid for 90° pulse and 4-s repetition time.

[B] Number of carbons.

[C] Referenced to isolated methylenes at 30.0 ppm.

TABLE X1.1 Ethene-Propene Copolymers[A]

Area	Chemical Shift, ppm	EEXEE		EEXEXEE		EEXXEE		EEXXXEE		T1, s	Factor
		Type	B	Type	B	Type	B	Type	B		
A	~45.5					αα	1	ααγ	2	1.38	1.06
B	~37.5–37.9	α	2	α,αγ	4	αγ	2	αγ	2	1.38	1.06
C	33.2	CH	1	CH	2					2.14	1.18
D	30.4–31.5	γ	2	γ	2	CHβ,γ	4	CHβ, γ	4	1.56	1.08
	30.0	δ,δ[C]	3	δ, δ[C]	3	δ,δ[C]	3	δ, δ[C]	3	1.64	1.10
	~28							CHββ	1	2.14	1.18
E	27.4	β	2	β	2	β	2	β	2	1.54	1.08
F	24.8			ββ	1					1.54	1.08
G	20.0–21.6	M1	1	M1	2	M1	2	M1	3	2.96	1.35

[A] Correction factors valid for 90° pulse and 4-s repetition time.
[B] Number of carbons.
[C] Referenced to isolated methylenes at 30.0 ppm.

TABLE X1.4 Ethene-Octene-1 Copolymers[A]

Area	Chemical Shift, ppm	EEXEE Type	EEXEE [A]	EEXEXEE Type	EEXEXEE [A]	EEXXEE Type	EEXXEE [A]	EEXXXEE Type	EEXXXEE [A]	T1, s	Factor
A	~40.8							ααγ	2	0.79	1.01
B	~40.1					αα	1			0.79	1.01
C	38.1	CH	1	CH	2					1.06	1.02
D	35.8					CHβ	2	CHβ	2	1.06	1.02
E	34.5–35.2	H6, α	3	H6, α, αγ	6	H6,αδ	4	H6, αγ	5	0.79	1.01
	33.9							CHββ	1	1.06	1.02
F	32.2	H3	1	H3	2	H3	2	H3	3	4.24	1.64
G	30.4	γ	2	γ	2	γ	2	γ	2	1.22	1.04
	30.0	H4	1	H4	1	H4	1	H4	1	~2.0[B]	1.15
	30.0	δ,δ[C]	3	δ,δ[C]	3	δ,δ[C]	3	δ,δ[C]	3	1.60	1.09
H	27.3	β	2	β	2	β	2	β	2	0.92	1.01
	27.2	H5	1	H5	2	H5	2	H5	3	1.30	1.05
I	24.5			ββ	1					0.98	1.02
P	22.9	H2	1	H2	2	H2	2	H2	2	6.21	2.11

[A] Number of carbons.
[B] Estimated T1 relaxation.
[C] Reference to isolated methylenes at 30.0 ppm.

TABLE X1.5 Ethene-4-Methylpentene-1 Copolymers[A]

Area	Chemical Shift, ppm	EEXEE Type	EEXEE [A]	EEXEXEE Type	EEXEXEE [A]	EEXXEE Type	EEXXEE [A]	EEXXXEE Type	EEXXXEE [A]	T1, s	Factor
A	44.7	IB3	1	IB3	2	IB3	2	IB3	3	1.09	1.03
B	42.2							ααγ	2	0.92	1.01
C	41.2					αα	1			0.92	1.01
D	35.9	CH	1	CH	2					1.33	1.05
	34.8–35.2	α	2	α,αγ	4	αγ	2	αγ	2	0.92	1.01
E	33.6					CHβ	2	CHβ	2	1.33	1.05
F	31.0							CHββ	1	1.33	1.05
	30.4	γ	2	γ	2	γ	2	γ	2	1.24	1.04
	30.0	δ, δ[B]	3	δ,δ[B]	3	δ, δ[B]	3	δ,δ[B]	3	1.62	1.09
G	27.1	β	2	β	2	β	2	β	2	1.14	1.03
	26.0	IB2	1	IB2	2	IB	2	IB2	3	1.66	1.10
H	24.1			ββ	1					1.14	1.03

[A] Number of carbons.
[B] Reference to isolated methylenes at 30.0 ppm.

TABLE X1.2 Ethene-Butene-1 Copolymers[A]

Area	Chemical Shift, ppm	EEXEE Type	EEXEE [B]	EEXEXEE Type	EEXEXEE [B]	EEXXEE Type	EEXXEE [B]	EEXXXEE Type	EEXXXEE [B]	T1, s	Factor
A	39.0–39.6	CH	1	CH	2	αα	2		1.91	1.14	
A	~39.4							ααγ	2	1.23	1.04
B	~37.2					CHβ	2	CHβ	2	1.91	1.14
C	34–35	α	2	α,αγ	4	αγ	2	CHββ, αγ	3	1.23	1.04
D	30.4	γ	2	γ	2	γ	2	γ	2	1.51	1.08
	30.0	δ, δ[C]	3	δ,δ[C]	3	δ, δ[C]	3	δ,δ[C]	3	1.64	1.09
E	27.3	β	2	β	2	β	2	β	2	1.46	1.07
	26.7	E2	1	E2	2	E2	2	E2	3	1.56	1.08
F	24.6			ββ	1					1.46	1.07

[A] Correction factors valid for 90° pulse and 4-s repetition time.
[B] Number of carbons.
[C] Referenced to isolated methylenes at 30.0 ppm.

~~TABLE X1.3 Ethene-Hexene-1 Copolymers[A]~~

TABLE X1.3 Ethene-Hexene-1 Copolymers[A]

Area	Chemical Shift, ppm	EEXEE Type	A	EEXEXEE Type	A	EEXXEE Type	A	EEXXXEE Type	A	T1, s	Factor
A	~40.8							ααγ	2	0.96	1.02
B	~40.2					αα	1			0.96	1.02
C	38.1	CH	1	CH	2					1.48	1.07
D	~35.8					CHβ	2			1.48	1.07
E	34.5–35.0	α	2	α, αγ	4	B4,αγ	4	B4, αγ	5	0.96	1.02
	34.1	B4	1	B4	2					1.19	1.04
	33.5							CHββ	1	1.48	1.07
F	30.4	γ	2	γ	2	γ	2	γ	2	1.36	1.05
	30.0	δ, δ[B]	3	δ,δ[B]	3	δ, δ[B]	3	δ,δ[B]	3	1.75	1.11
	29.5	B3	1	B3	2	B3	2	B3	3	1.98	1.15
G	27.3	β	2	β	2	β	2	β	2	1.21	1.04
H	24.5			ββ	1					1.21	1.04

[A] Number of carbons.
[B] Referenced to isolated methylenes at 30.0 ppm.

Area	Chemical Shift, ppm	EEXEE Type	A	EEXEXEE Type	A	EEXXEE Type	A	EEXXXEE Type	A	T1, s	Factor
A	~40.8							ααγ	2	0.96	1.02
B	~40.2					αα	1			0.96	1.02
C	38.1	CH	1	CH	2					1.48	1.07
D	~35.8					CHβ	2			1.48	1.07
E	34.5–35.0	α	2	α, αγ	4	B4,αγ	4	B4, αγ	5	0.96	1.02
	34.1	B4	1	B4	2					1.19	1.04
	33.5							CHββ	1	1.48	1.07
F	30.4	γ	2	γ	2	γ	2	γ	2	1.36	1.05
	30.0	δ, δ[B]	3	δ,δ[B]	3	δ, δ[B]	3	δ,δ[B]	3	1.75	1.11
	29.5	B3	1	B3	2	B3	2	B3	3	1.98	1.15
G	27.3	β	2	β	2	β	2	β	2	1.21	1.04
H	24.5			ββ	1					1.21	1.04

[A] Number of carbons.
[B] Referenced to isolated methylenes at 30.0 ppm.

TABLE X1.4 Ethene-Octene-1 Copolymers[A]

Area	Chemical Shift, ppm	EEXEE Type	A	EEXEXEE Type	A	EEXXEE Type	A	EEXXXEE Type	A	T1, s	Factor
A	~40.8							ααγ	2	0.79	1.01
B	~40.1					αα	1			0.79	1.01
C	38.1	CH	1	CH	2					1.06	1.02
D	35.8					CHβ	2	CHβ	2	1.06	1.02
E	34.5–35.2	H6, α	3	H6, α, αγ	6	H6,αδ	4	H6, αγ	5	0.79	1.01
	33.9							CHββ	1	1.06	1.02
F	32.2	H3	1	H3	2	H3	2	H3	3	4.24	1.64
G	30.4	γ	2	γ	2	γ	2	γ	2	1.22	1.04
	30.0	H4	1	H4	1	H4	1	H4	1	~2.0[B]	1.15
	30.0	δ,δ[C]	3	δ,δ[C]	3	δ,δ[C]	3	δ,δ[C]	3	1.60	1.09
H	27.3	β	2	β	2	β	2	β	2	0.92	1.01
	27.2	H5	1	H5	2	H5	2	H5	3	1.30	1.05
I	24.5			ββ	1					0.98	1.02
P	22.9	H2	1	H2	2	H2	2	H2	2	6.21	2.11

[A] Number of carbons.
[B] Estimated T1 relaxation.
[C] Reference to isolated methylenes at 30.0 ppm.

TABLE X1.5 Ethene-4-Methylpentene-1 Copolymers[A]

Area	Chemical Shift, ppm	EEXEE Type	A	EEXEXEE Type	A	EEXXEE Type	A	EEXXXEE Type	A	T1, s	Factor
A	44.7	IB3	1	IB3	2	IB3	2	IB3	3	1.09	1.03
B	42.2							ααγ	2	0.92	1.01
C	41.2					αα	1			0.92	1.01
D	35.9	CH	1	CH	2					1.33	1.05
	34.8–35.2	α	2	α,αγ	4	αγ	2	αγ	2	0.92	1.01
E	33.6					CHβ	2	CHβ	2	1.33	1.05
F	31.0							CHββ	1	1.33	1.05
	30.4	γ	2	γ	2	γ	2	γ	2	1.24	1.04
	30.0	δ, δ[B]	3	δ,δ[B]	3	δ, δ[B]	3	δ,δ[B]	3	1.62	1.09

Area	Chemical Shift, ppm	EEXEE		EEXEXEE		EEXXEE		EEXXXEE		T1, s	Factor
		Type	A	Type	A	Type	A	Type	A		
G	27.1	β	2	β	2	β	2	β	2	1.14	1.03
	26.0	IB2	1	IB2	2	IB	2	IB2	3	1.66	1.10
H	24.1			ββ	1					1.14	1.03

[A] Number of carbons.
[B] Reference to isolated methylenes at 30.0 ppm.

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