

## Supporting Information for

# Determination of Copolymer Compositions in Polyhydroxyalkanoates Using $^1\text{H}$ Benchtop Nuclear Magnetic Resonance Spectroscopy

Juan F. Araneda,<sup>a</sup> Hasitha de Alwis Weerasesera,<sup>b</sup> Matthew C. Leclerc,<sup>\*a</sup> Sharmaine B. Luk,<sup>b</sup> and Susanne D. Riegel<sup>a</sup>

<sup>a</sup>*Nanalysis Corp., 1-4600 5 St NE, Calgary, AB, Canada T2E 7C3*

<sup>b</sup>*Genecis Bioindustries, 1100-700 Bay St, Toronto, ON, Canada M5G 1Z6*

*\*Corresponding author: [matt.leclerc@nanalysis.com](mailto:matt.leclerc@nanalysis.com)*

Table S1. Summary of monomeric ratios obtained for PHBV1 using <sup>1</sup>H NMR at 60 MHz, 100 MHz, and 400 MHz. The ratios are shown both as mol% and wt%. Three assays were prepared and analyzed for each polymer. The averages of triplicate analyses are included for each assay, in addition to the relative standard deviations (RSD).

PHBV1	60 MHz				100 MHz				400 MHz			
	mol%		wt%		mol%		wt%		mol%		wt%	
	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV
Assay 1	94.7	5.3	93.9	6.1	96.4	3.6	95.8	4.2	96.6	3.4	96.1	3.9
	94.8	5.2	94.0	6.0	96.2	3.8	95.6	4.4	96.6	3.4	96.1	3.9
	94.1	5.9	93.2	6.8	96.3	3.7	95.7	4.3	96.6	3.4	96.1	3.9
Average	94.5	5.5	93.7	6.3	96.3	3.7	95.7	4.3	96.6	3.4	96.1	3.9
RSD	0.3%	5.7%	0.4%	5.6%	0.1%	2.2%	0.1%	2.2%	0.0%	0.0%	0.0%	0.0%
Assay 2	94.9	5.1	94.1	5.9	96.9	3.1	96.4	3.6	96.6	3.4	96.1	3.9
	95.2	4.8	94.5	5.5	96.7	3.3	96.2	3.8	96.6	3.4	96.1	3.9
	94.9	5.1	94.1	5.9	96.4	3.6	95.8	4.2	96.6	3.4	96.1	3.9
Average	95.0	5.0	94.2	5.8	96.7	3.3	96.1	3.9	96.6	3.4	96.1	3.9
RSD	0.1%	2.8%	0.2%	2.8%	0.2%	6.2%	0.2%	6.1%	0.0%	0.0%	0.0%	0.0%
Assay 3	93.5	6.5	92.5	7.5	96.2	3.8	95.6	4.4	96.7	3.3	96.2	3.8
	94.2	5.8	93.3	6.7	96.3	3.7	95.7	4.3	96.7	3.3	96.2	3.8
	93.9	6.1	93.0	7.0	96.2	3.8	95.6	4.4	96.6	3.4	96.1	3.9
Average	93.9	6.1	92.9	7.1	96.2	3.8	95.7	4.3	96.7	3.3	96.1	3.9
RSD	0.3%	4.7%	0.4%	4.6%	0.0%	1.3%	0.1%	1.2%	0.0%	1.4%	0.1%	1.4%

Table S2. Summary of monomeric ratios obtained for PHBV2 using <sup>1</sup>H NMR at 60 MHz, 100 MHz, and 400 MHz. The ratios are shown both as mol% and wt%. Three assays were prepared and analyzed for each polymer. The averages of triplicate analyses are included for each assay, in addition to the relative standard deviations (RSD).

PHBV2	60 MHz				100 MHz				400 MHz			
	mol%		wt%		mol%		wt%		mol%		wt%	
	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV
Assay 1	73.7	26.3	70.7	29.3	74.2	25.8	71.2	28.8	74.0	26.0	71.0	29.0
	73.3	26.7	70.3	29.7	74.1	25.9	71.1	28.9	74.0	26.0	71.0	29.0
	74.0	26.0	71.0	29.0	74.0	26.0	71.0	29.0	74.0	26.0	71.0	29.0
Average	73.7	26.3	70.7	29.3	74.1	25.9	71.1	28.9	74.0	26.0	71.0	29.0
RSD	0.4%	1.1%	0.4%	1.0%	0.1%	0.3%	0.1%	0.3%	0.0%	0.0%	0.0%	0.0%
Assay 2	74.0	26.0	71.0	29.0	74.3	25.7	71.3	28.7	74.0	26.0	71.0	29.0
	74.0	26.0	71.0	29.0	73.8	26.2	70.8	29.2	74.0	26.0	71.0	29.0
	73.5	26.5	70.5	29.5	73.9	26.1	70.9	29.1	73.9	26.1	70.9	29.1
Average	73.8	26.2	70.8	29.2	74.0	26.0	71.0	29.0	74.0	26.0	71.0	29.0
RSD	0.3%	0.9%	0.4%	0.9%	0.3%	0.8%	0.3%	0.8%	0.1%	0.2%	0.1%	0.2%
Assay 3	72.3	27.7	69.2	30.8	74.4	25.6	71.4	28.6	74.0	26.0	71.0	29.0
	72.6	27.4	69.5	30.5	74.8	25.2	71.9	28.1	74.0	26.0	71.0	29.0
	73.5	26.5	70.5	29.5	74.4	25.6	71.4	28.6	74.1	25.9	71.1	28.9
Average	72.8	27.2	69.7	30.3	74.5	25.5	71.6	28.4	74.0	26.0	71.1	28.9
RSD	0.7%	1.9%	0.8%	1.8%	0.3%	0.7%	0.3%	0.7%	0.1%	0.2%	0.1%	0.2%

Table S3. Summary of monomeric ratios obtained for PHBV3 using <sup>1</sup>H NMR at 60 MHz, 100 MHz, and 400 MHz. The ratios are shown both as mol% and wt%. Three assays were prepared and analyzed for each polymer. The averages of triplicate analyses are included for each assay, in addition to the relative standard deviations (RSD).

PHBV3	60 MHz				100 MHz				400 MHz			
	mol%		wt%		mol%		wt%		mol%		wt%	
	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV
Assay 1	97.1	2.9	96.6	3.4	92.3	7.7	91.2	8.8	92.6	7.4	91.5	8.5
	97.5	2.5	97.1	2.9	92.4	7.6	91.3	8.7	92.6	7.4	91.5	8.5
	97.5	2.5	97.1	2.9	92.7	7.3	91.6	8.4	92.7	7.3	91.6	8.4
Average	97.4	2.6	97.0	3.0	92.5	7.5	91.4	8.6	92.6	7.4	91.5	8.5
RSD	0.2%	7.2%	0.2%	7.1%	0.2%	2.3%	0.2%	2.2%	0.1%	0.6%	0.1%	0.6%
Assay 2	97.6	2.4	97.2	2.8	92.7	7.3	91.6	8.4	92.6	7.4	91.5	8.5
	97.0	3.0	96.5	3.5	92.3	7.7	91.2	8.8	92.6	7.4	91.5	8.5
	97.2	2.8	96.8	3.2	92.3	7.7	91.2	8.8	92.6	7.4	91.5	8.5
Average	97.3	2.7	96.8	3.2	92.4	7.6	91.3	8.7	92.6	7.4	91.5	8.5
RSD	0.3%	9.1%	0.3%	9.1%	0.2%	2.5%	0.2%	2.5%	0.0%	0.0%	0.0%	0.0%
Assay 3	97.0	3.0	96.5	3.5	92.4	7.6	91.3	8.7	92.5	7.5	91.4	8.6
	97.2	2.8	96.8	3.2	92.4	7.6	91.3	8.7	92.6	7.4	91.5	8.5
	97.1	2.9	96.6	3.4	92.6	7.4	91.5	8.5	92.6	7.4	91.5	8.5
Average	97.1	2.9	96.6	3.4	92.5	7.5	91.4	8.6	92.6	7.4	91.5	8.5
RSD	0.1%	2.8%	0.1%	2.8%	0.1%	1.3%	0.1%	1.2%	0.1%	0.6%	0.1%	0.6%

Table S4. Summary of monomeric ratios obtained for PHBV4 using <sup>1</sup>H NMR at 60 MHz, 100 MHz, and 400 MHz. The ratios are shown both as mol% and wt%. Three assays were prepared and analyzed for each polymer. The averages of triplicate analyses are included for each assay, in addition to the relative standard deviations (RSD).

PHBV4	60 MHz				100 MHz				400 MHz			
	mol%		wt%		mol%		wt%		mol%		wt%	
	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV
Assay 1	94.9	5.1	94.1	5.9	85.2	14.8	83.2	16.8	85.5	14.5	83.5	16.5
	93.8	6.2	92.9	7.1	85.5	14.5	83.5	16.5	85.3	14.7	83.3	16.7
	94.9	5.1	94.1	5.9	85.1	14.9	83.1	16.9	85.5	14.5	83.5	16.5
Average	94.5	5.5	93.7	6.3	85.3	14.7	83.3	16.7	85.4	14.6	83.5	16.5
RSD	0.5%	9.5%	0.6%	9.4%	0.2%	1.2%	0.2%	1.1%	0.1%	0.6%	0.1%	0.6%
Assay 2	95.6	4.4	94.9	5.1	85.6	14.4	83.7	16.3	85.4	14.6	83.4	16.6
	94.0	6.0	93.1	6.9	84.7	15.3	82.7	17.3	85.4	14.6	83.4	16.6
	94.3	5.7	93.4	6.6	85.8	14.2	83.9	16.1	85.4	14.6	83.4	16.6
Average	94.6	5.4	93.8	6.2	85.4	14.6	83.4	16.6	85.4	14.6	83.4	16.6
RSD	0.7%	12.9%	0.8%	12.8%	0.6%	3.3%	0.6%	3.2%	0.0%	0.0%	0.0%	0.0%
Assay 3	93.8	6.2	92.9	7.1	85.3	14.7	83.3	16.7	85.5	14.5	83.5	16.5
	94.4	5.6	93.6	6.4	85.0	15.0	83.0	17.0	85.5	14.5	83.5	16.5
	93.8	6.2	92.9	7.1	84.7	15.3	82.7	17.3	85.5	14.5	83.5	16.5
Average	94.0	6.0	93.1	6.9	85.0	15.0	83.0	17.0	85.5	14.5	83.5	16.5
RSD	0.3%	4.7%	0.3%	4.7%	0.3%	1.6%	0.3%	1.6%	0.0%	0.0%	0.0%	0.0%

Table S5. Summary of monomeric ratios obtained for PHBV5 using <sup>1</sup>H NMR at 60 MHz, 100 MHz, and 400 MHz. The ratios are shown both as mol% and wt%. Three assays were prepared and analyzed for each polymer. The averages of triplicate analyses are included for each assay, in addition to the relative standard deviations (RSD).

PHBV5	60 MHz				100 MHz				400 MHz			
	mol%		wt%		mol%		wt%		mol%		wt%	
	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV
Assay 1	95.6	4.4	94.9	5.1	89.1	10.9	87.6	12.4	89.1	10.9	87.6	12.4
	95.5	4.5	94.8	5.2	88.5	11.5	86.9	13.1	89.2	10.8	87.7	12.3
	95.5	4.5	94.8	5.2	88.2	11.8	86.6	13.4	89.1	10.9	87.6	12.4
Average	95.5	4.5	94.8	5.2	88.6	11.4	87.0	13.0	89.1	10.9	87.6	12.4
RSD	0.0%	1.1%	0.1%	1.0%	0.4%	3.3%	0.5%	3.2%	0.1%	0.4%	0.1%	0.4%
Assay 2	95.1	4.9	94.4	5.6	88.6	11.4	87.0	13.0	89.2	10.8	87.7	12.3
	95.6	4.4	94.9	5.1	88.4	11.6	86.8	13.2	89.2	10.8	87.7	12.3
	95.6	4.4	94.9	5.1	88.6	11.4	87.0	13.0	89.2	10.8	87.7	12.3
Average	95.4	4.6	94.7	5.3	88.5	11.5	86.9	13.1	89.2	10.8	87.7	12.3
RSD	0.2%	5.2%	0.3%	5.1%	0.1%	0.8%	0.1%	0.8%	0.0%	0.0%	0.0%	0.0%
Assay 3	95.7	4.3	95.0	5.0	88.5	11.5	86.9	13.1	89.2	10.8	87.7	12.3
	95.5	4.5	94.8	5.2	88.4	11.6	86.8	13.2	89.1	10.9	87.6	12.4
	95.5	4.5	94.8	5.2	88.3	11.7	86.7	13.3	89.2	10.8	87.7	12.3
Average	95.6	4.4	94.9	5.1	88.4	11.6	86.8	13.2	89.2	10.8	87.6	12.4
RSD	0.1%	2.1%	0.1%	2.1%	0.1%	0.7%	0.1%	0.7%	0.1%	0.4%	0.1%	0.4%

Table S6. Summary of monomeric ratios obtained for PHBV6 using <sup>1</sup>H NMR at 60 MHz, 100 MHz, and 400 MHz. The ratios are shown both as mol% and wt%. Three assays were prepared and analyzed for each polymer. The averages of triplicate analyses are included for each assay, in addition to the relative standard deviations (RSD).

PHBV6	60 MHz				100 MHz				400 MHz			
	mol%		wt%		mol%		wt%		mol%		wt%	
	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV
Assay 1	78.5	21.5	75.9	24.1	81.0	19.0	78.6	21.4	81.0	19.0	78.6	21.4
	80.0	20.0	77.5	22.5	80.6	19.4	78.2	21.8	80.9	19.1	78.5	21.5
	79.8	20.2	77.3	22.7	80.7	19.3	78.3	21.7	80.9	19.1	78.5	21.5
Average	79.4	20.6	76.9	23.1	80.8	19.2	78.3	21.7	80.9	19.1	78.5	21.5
RSD	0.8%	3.2%	0.9%	3.1%	0.2%	0.9%	0.2%	0.9%	0.1%	0.2%	0.1%	0.2%
Assay 2	80.6	19.4	78.2	21.8	81.2	18.8	78.8	21.2	80.8	19.2	78.4	21.6
	79.9	20.1	77.4	22.6	80.7	19.3	78.3	21.7	80.7	19.3	78.3	21.7
	80.8	19.2	78.4	21.6	80.7	19.3	78.3	21.7	80.8	19.2	78.4	21.6
Average	80.4	19.6	78.0	22.0	80.9	19.1	78.4	21.6	80.8	19.2	78.3	21.7
RSD	0.5%	2.0%	0.5%	1.9%	0.3%	1.2%	0.3%	1.2%	0.1%	0.2%	0.1%	0.2%
Assay 3	80.2	19.8	77.7	22.3	80.5	19.5	78.0	22.0	80.9	19.1	78.5	21.5
	79.9	20.1	77.4	22.6	80.9	19.1	78.5	21.5	80.8	19.2	78.4	21.6
	80.0	20.0	77.5	22.5	80.9	19.1	78.5	21.5	80.9	19.1	78.5	21.5
Average	80.0	20.0	77.5	22.5	80.8	19.2	78.3	21.7	80.9	19.1	78.4	21.6
RSD	0.2%	0.6%	0.2%	0.6%	0.2%	1.0%	0.3%	1.0%	0.1%	0.2%	0.1%	0.2%

Table S7. Summary of monomeric ratios obtained for PHBV7 using <sup>1</sup>H NMR at 60 MHz, 100 MHz, and 400 MHz. The ratios are shown both as mol% and wt%. Three assays were prepared and analyzed for each polymer. The averages of triplicate analyses are included for each assay, in addition to the relative standard deviations (RSD).

PHBV7	60 MHz				100 MHz				400 MHz			
	mol%		wt%		mol%		wt%		mol%		wt%	
	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV
Assay 1	76.5	23.5	73.7	26.3	77.8	22.2	75.1	24.9	77.5	22.5	74.8	25.2
	76.2	23.8	73.4	26.6	77.9	22.1	75.3	24.7	77.6	22.4	74.9	25.1
	76.2	23.8	73.4	26.6	77.9	22.1	75.2	24.8	77.6	22.4	74.9	25.1
Average	76.3	23.7	73.5	26.5	77.9	22.1	75.2	24.8	77.6	22.4	74.9	25.1
RSD	0.2%	0.6%	0.2%	0.6%	0.1%	0.3%	0.1%	0.2%	0.1%	0.2%	0.1%	0.2%
Assay 2	75.4	24.6	72.5	27.5	78.1	21.9	75.4	24.6	77.9	22.1	75.2	24.8
	75.2	24.8	72.3	27.7	78.5	21.5	75.9	24.1	77.9	22.1	75.2	24.8
	75.3	24.7	72.4	27.6	78.4	21.6	75.8	24.2	77.8	22.2	75.1	24.9
Average	75.3	24.7	72.4	27.6	78.3	21.7	75.7	24.3	77.9	22.1	75.2	24.8
RSD	0.1%	0.3%	0.1%	0.3%	0.2%	0.8%	0.2%	0.8%	0.1%	0.2%	0.1%	0.2%
Assay 3	75.8	24.2	73.0	27.0	77.9	22.1	75.2	24.8	77.6	22.4	74.9	25.1
	76.2	23.8	73.4	26.6	77.9	22.1	75.2	24.8	77.5	22.5	74.8	25.2
	75.7	24.3	72.8	27.2	77.9	22.1	75.2	24.8	77.6	22.4	74.9	25.1
Average	75.9	24.1	73.1	26.9	77.9	22.1	75.2	24.8	77.6	22.4	74.9	25.1
RSD	0.3%	0.9%	0.3%	0.9%	0.0%	0.0%	0.0%	0.0%	0.1%	0.2%	0.1%	0.2%



Table S8. Summary of monomeric ratios obtained for PHBV8 using <sup>1</sup>H NMR at 60 MHz, 100 MHz, and 400 MHz. The ratios are shown both as mol% and wt%. Three assays were prepared and analyzed for each polymer. The averages of triplicate analyses are included for each assay, in addition to the relative standard deviations (RSD).

PHBV8	60 MHz				100 MHz				400 MHz			
	mol%		wt%		mol%		wt%		mol%		wt%	
	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV
Assay 1	60.4	39.6	56.8	43.2	61.3	38.7	57.7	42.3	61.1	38.9	57.5	42.5
	60.5	39.5	56.9	43.1	61.0	39.0	57.4	42.6	61.2	38.8	57.6	42.4
	60.7	39.3	57.1	42.9	61.2	38.8	57.6	42.4	61.0	39.0	57.4	42.6
Average	60.5	39.5	56.9	43.1	61.2	38.8	57.6	42.4	61.1	38.9	57.5	42.5
RSD	0.2%	0.3%	0.2%	0.3%	0.2%	0.3%	0.2%	0.3%	0.1%	0.2%	0.1%	0.2%
Assay 2	62.9	37.1	59.3	40.7	62.0	38.0	58.4	41.6	61.4	38.6	57.8	42.2
	62.8	37.2	59.2	40.8	61.8	38.2	58.2	41.8	61.4	38.6	57.8	42.2
	62.6	37.4	59.0	41.0	61.8	38.2	58.2	41.8	61.5	38.5	57.9	42.1
Average	62.8	37.2	59.2	40.8	61.9	38.1	58.3	41.7	61.4	38.6	57.8	42.2
RSD	0.2%	0.3%	0.2%	0.3%	0.2%	0.2%	0.2%	0.2%	0.1%	0.1%	0.1%	0.1%
Assay 3	60.6	39.4	57.0	43.0	60.7	39.3	57.1	42.9	60.7	39.3	57.1	42.9
	60.7	39.3	57.1	42.9	61.0	39.0	57.4	42.6	60.7	39.3	57.1	42.9
	59.9	40.1	56.3	43.7	60.8	39.2	57.2	42.8	60.7	39.3	57.1	42.9
Average	60.4	39.6	56.8	43.2	60.8	39.2	57.2	42.8	60.7	39.3	57.1	42.9
RSD	0.6%	0.9%	0.6%	0.8%	0.2%	0.3%	0.2%	0.3%	0.0%	0.0%	0.0%	0.0%

Table S9. Summary of monomeric ratios obtained for PHBV9 using <sup>1</sup>H NMR at 60 MHz, 100 MHz, and 400 MHz. The ratios are shown both as mol% and wt%. Three assays were prepared and analyzed for each polymer. The averages of triplicate analyses are included for each assay, in addition to the relative standard deviations (RSD).

PHBV9	60 MHz				100 MHz				400 MHz			
	mol%		wt%		mol%		wt%		mol%		wt%	
	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV	3HB	3HV
Assay 1	58.9	41.1	55.2	44.8	57.7	42.3	54.0	46.0	57.0	43.0	53.3	46.7
	59.2	40.8	55.5	44.5	57.5	42.5	53.8	46.2	57.0	43.0	53.3	46.7
	59.4	40.6	55.7	44.3	57.2	42.8	53.5	46.5	56.8	43.2	53.1	46.9
Average	59.2	40.8	55.5	44.5	57.5	42.5	53.8	46.2	56.9	43.1	53.2	46.8
RSD	0.3%	0.5%	0.4%	0.5%	0.4%	0.5%	0.4%	0.5%	0.2%	0.2%	0.2%	0.2%
Assay 2	58.4	41.6	54.7	45.3	57.5	42.5	53.8	46.2	57.1	42.9	53.4	46.6
	57.7	42.3	54.0	46.0	57.8	42.2	54.1	45.9	57.1	42.9	53.4	46.6
	58.2	41.8	54.5	45.5	57.6	42.4	53.9	46.1	57.2	42.8	53.5	46.5
Average	58.1	41.9	54.4	45.6	57.6	42.4	53.9	46.1	57.1	42.9	53.4	46.6
RSD	0.5%	0.7%	0.6%	0.7%	0.2%	0.3%	0.2%	0.3%	0.1%	0.1%	0.1%	0.1%
Assay 3	59.3	40.7	55.6	44.4	57.4	42.6	53.7	46.3	57.2	42.8	53.5	46.5
	58.6	41.4	54.9	45.1	57.6	42.4	53.9	46.1	57.2	42.8	53.5	46.5
	59.0	41.0	55.3	44.7	57.6	42.4	53.9	46.1	57.1	42.9	53.4	46.6
Average	59.0	41.0	55.3	44.7	57.5	42.5	53.8	46.2	57.2	42.8	53.5	46.5
RSD	0.5%	0.7%	0.5%	0.7%	0.2%	0.2%	0.2%	0.2%	0.1%	0.1%	0.1%	0.1%

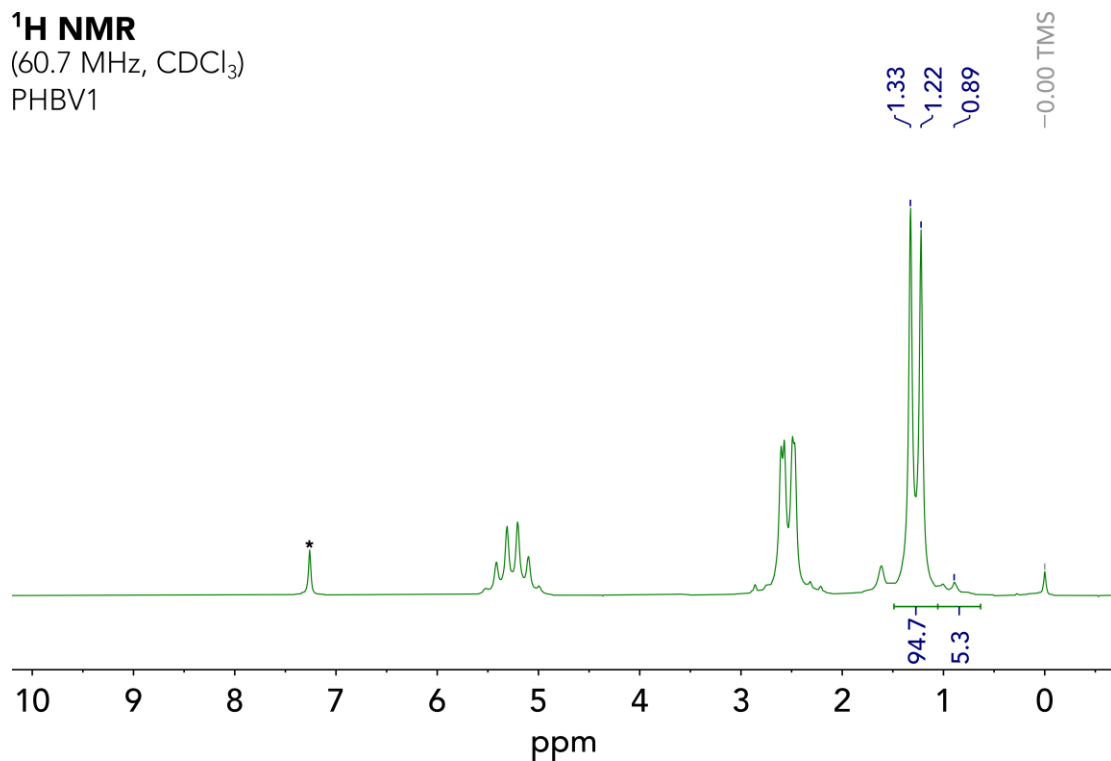


Figure S1. <sup>1</sup>H (60.7 MHz) NMR spectrum of PHBV1 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

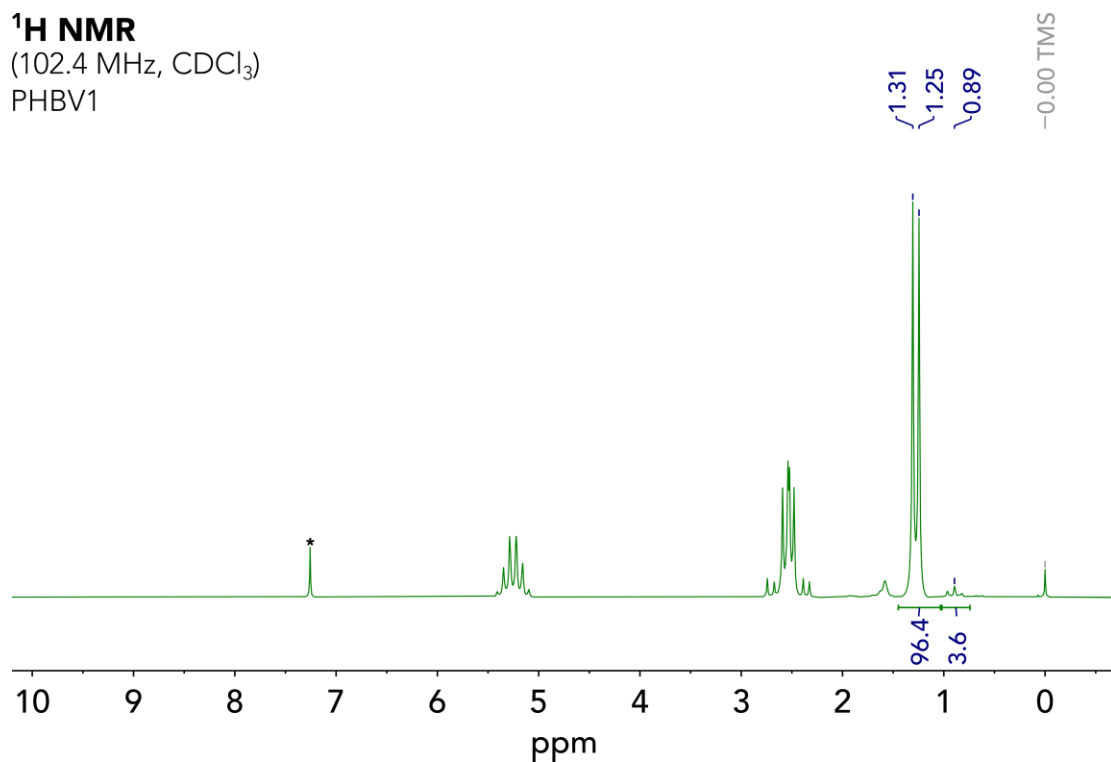


Figure S2. <sup>1</sup>H (102.4 MHz) NMR spectrum of PHBV1 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**(400.1 MHz, CDCl<sub>3</sub>)

PHBV1

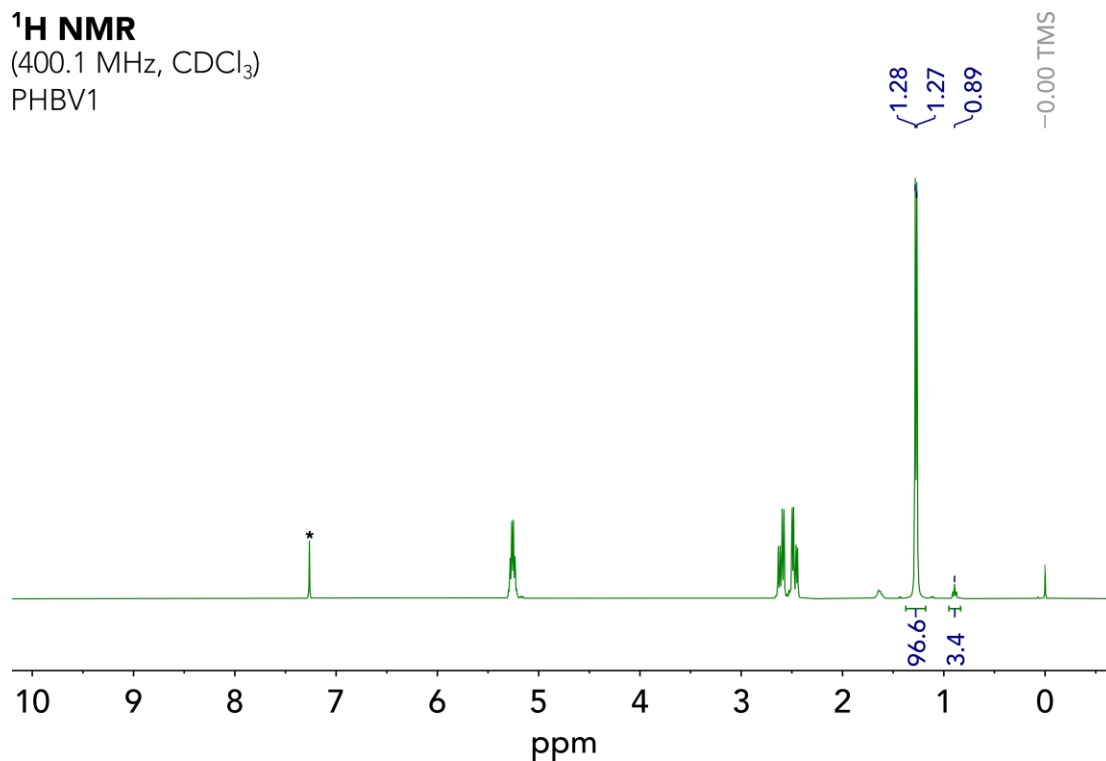


Figure S3. <sup>1</sup>H (400.1 MHz) NMR spectrum of PHBV1 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**(60.4 MHz, CDCl<sub>3</sub>)

PHBV2

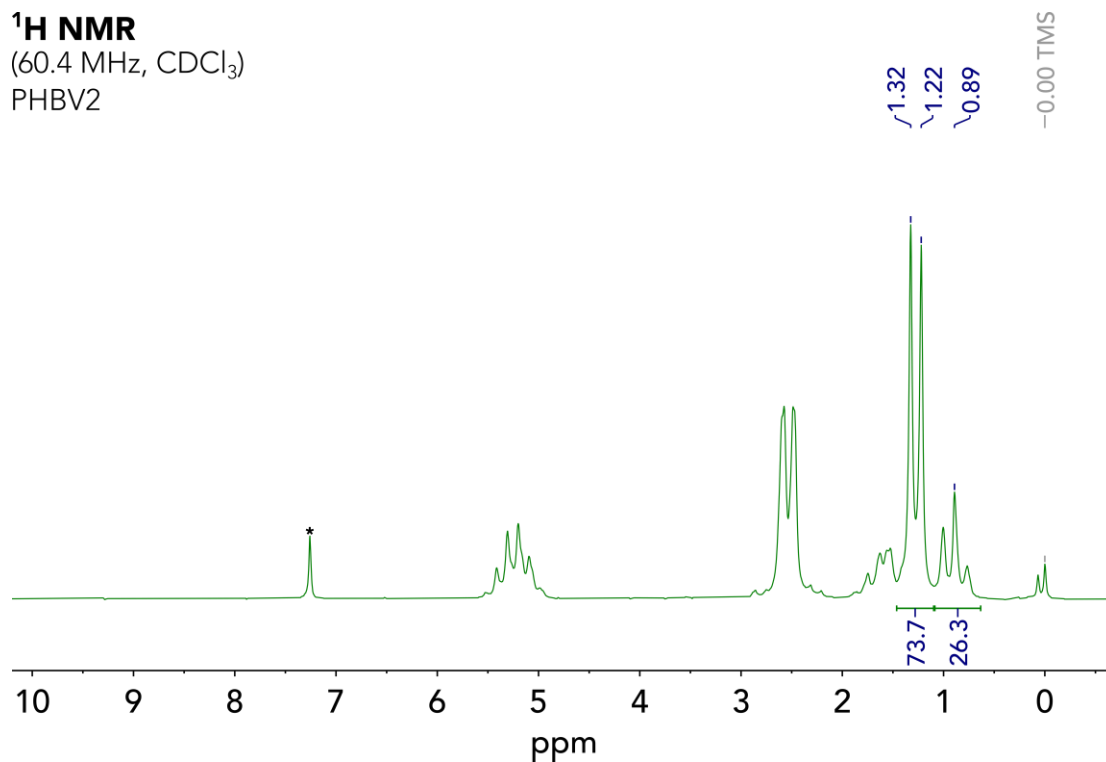


Figure S4. <sup>1</sup>H (60.4 MHz) NMR spectrum of PHBV2 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

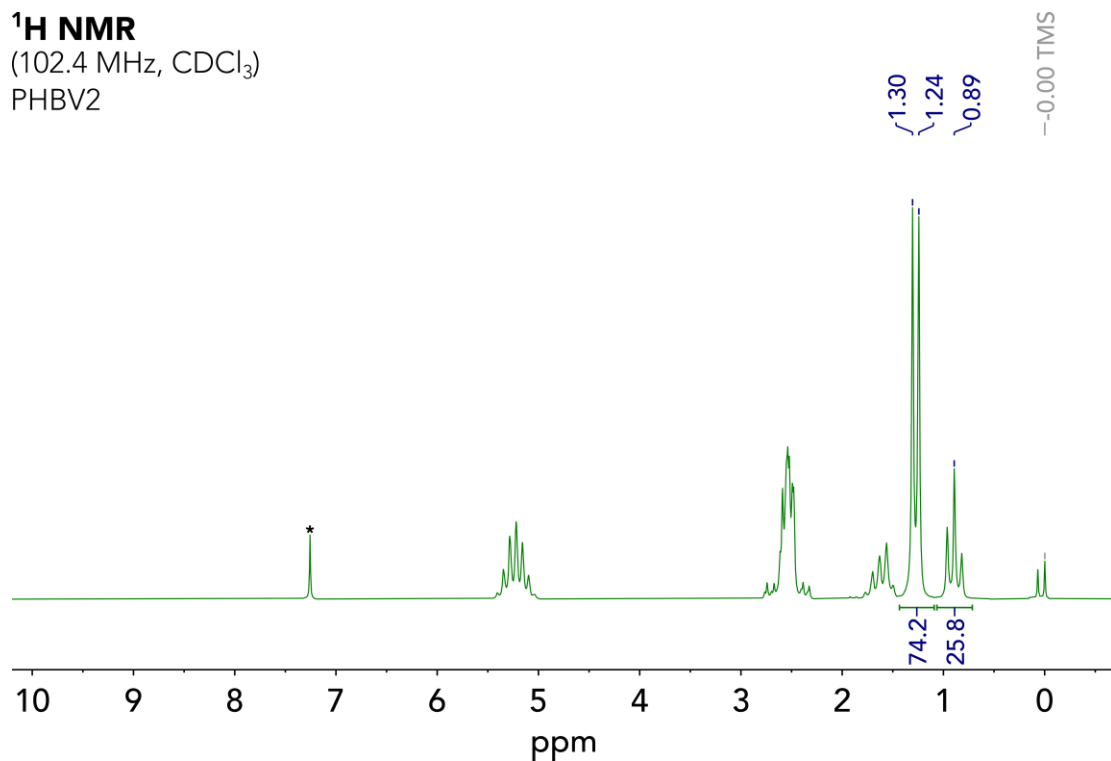


Figure S5. <sup>1</sup>H (102.4 MHz) NMR spectrum of PHBV2 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

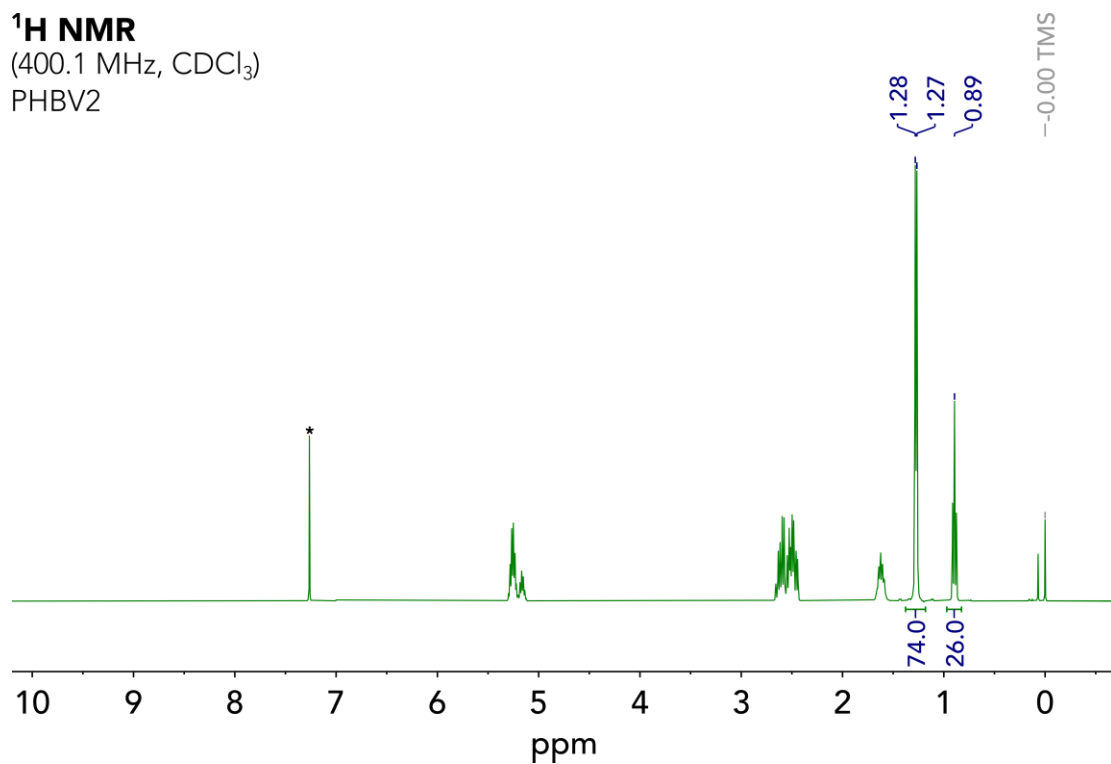


Figure S6. <sup>1</sup>H (400.1 MHz) NMR spectrum of PHBV2 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**

(60.4 MHz, CDCl<sub>3</sub>)

PHBV3

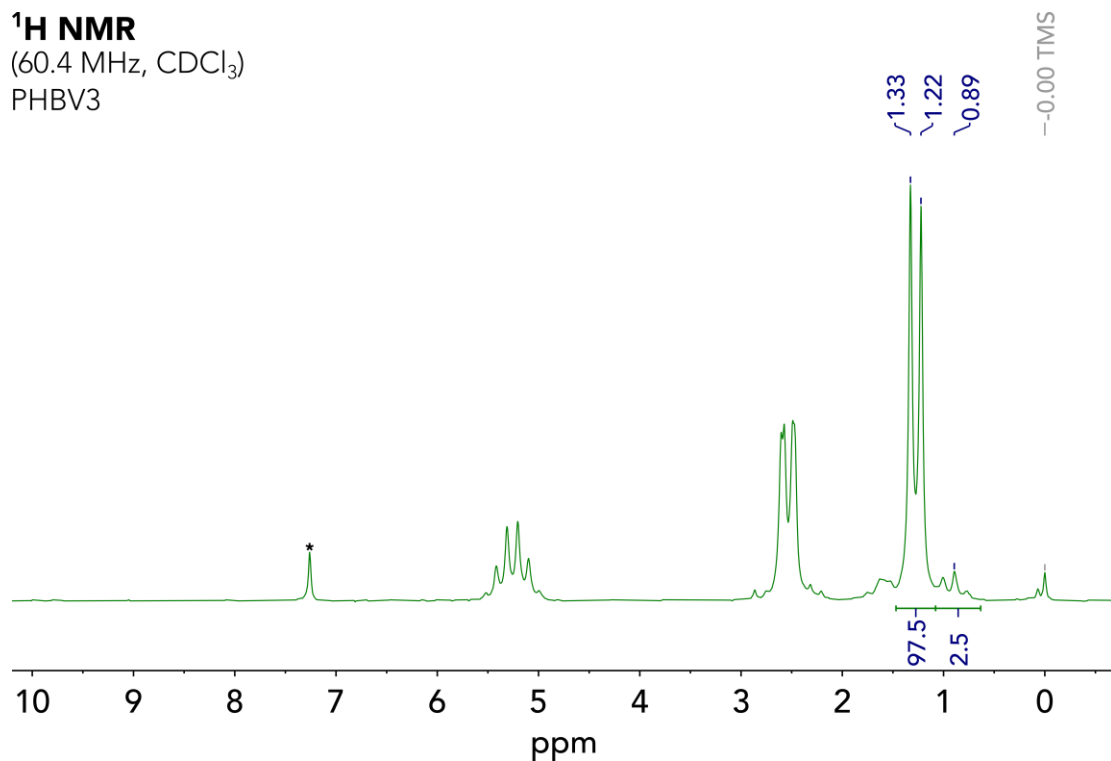


Figure S7. <sup>1</sup>H (60.4 MHz) NMR spectrum of PHBV3 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**

(102.4 MHz, CDCl<sub>3</sub>)

PHBV3

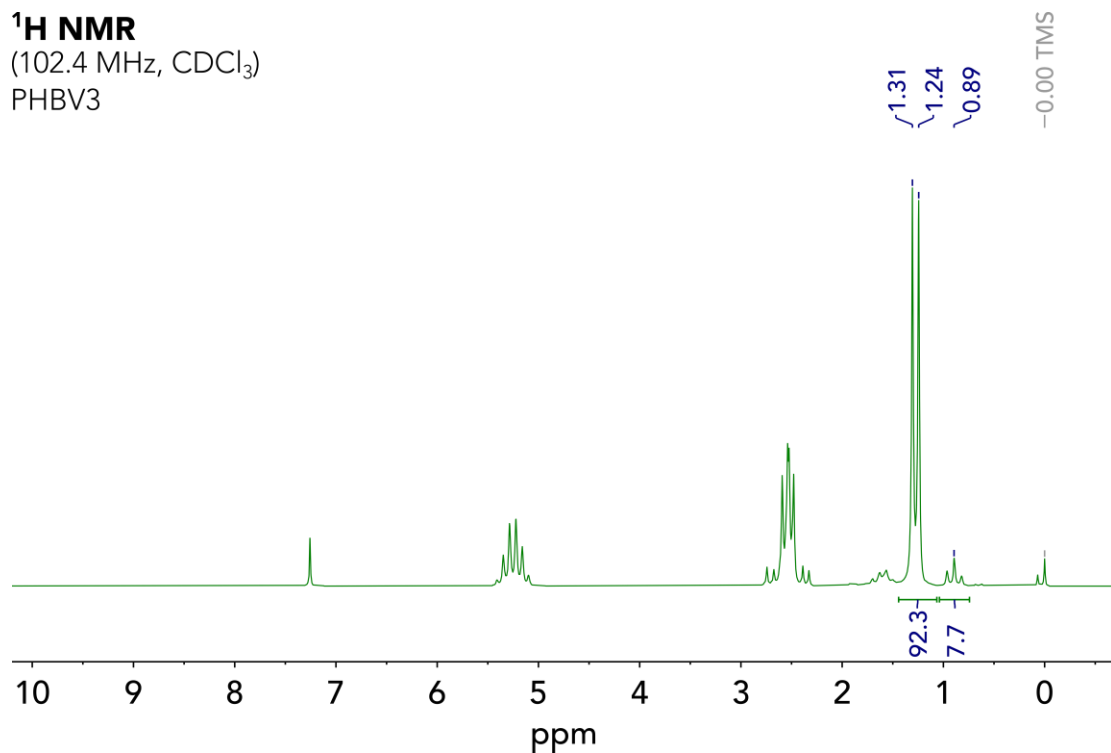


Figure S8. <sup>1</sup>H (102.4 MHz) NMR spectrum of PHBV3 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**

(400.1 MHz, CDCl<sub>3</sub>)

PHBV3

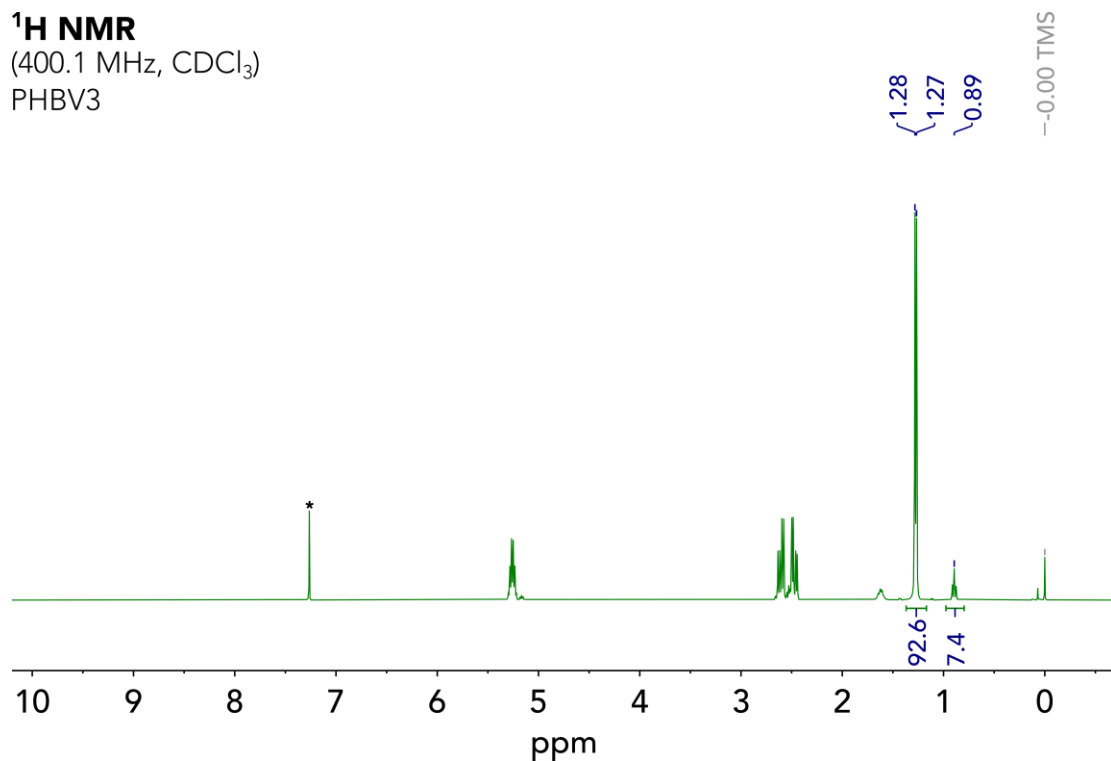


Figure S9. <sup>1</sup>H (400.1 MHz) NMR spectrum of PHBV3 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**

(60.7 MHz, CDCl<sub>3</sub>)

PHBV4

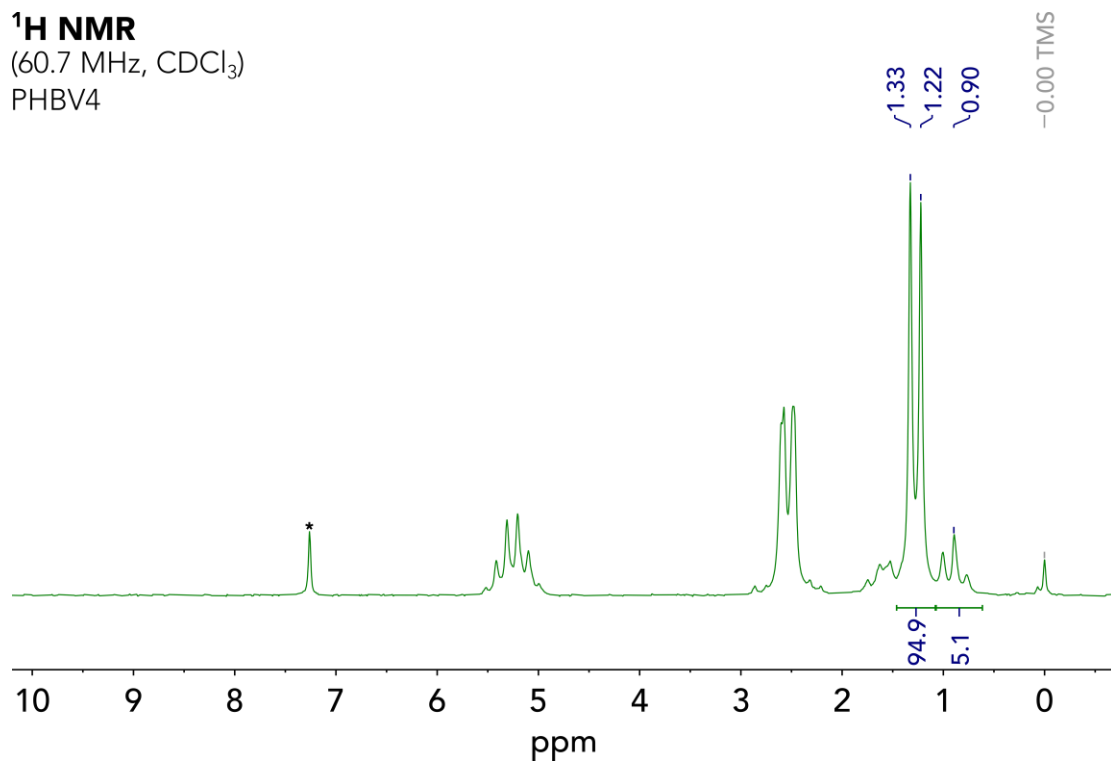


Figure S10. <sup>1</sup>H (60.7 MHz) NMR spectrum of PHBV4 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

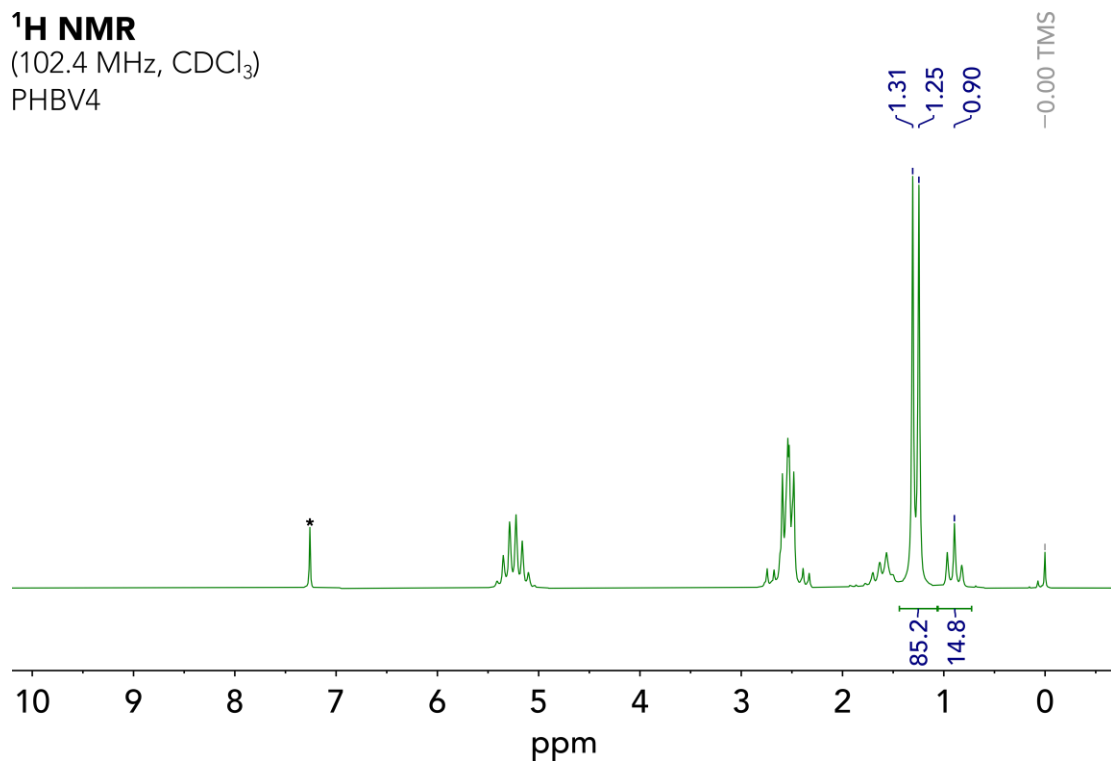


Figure S11. <sup>1</sup>H (102.4 MHz) NMR spectrum of PHBV4 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

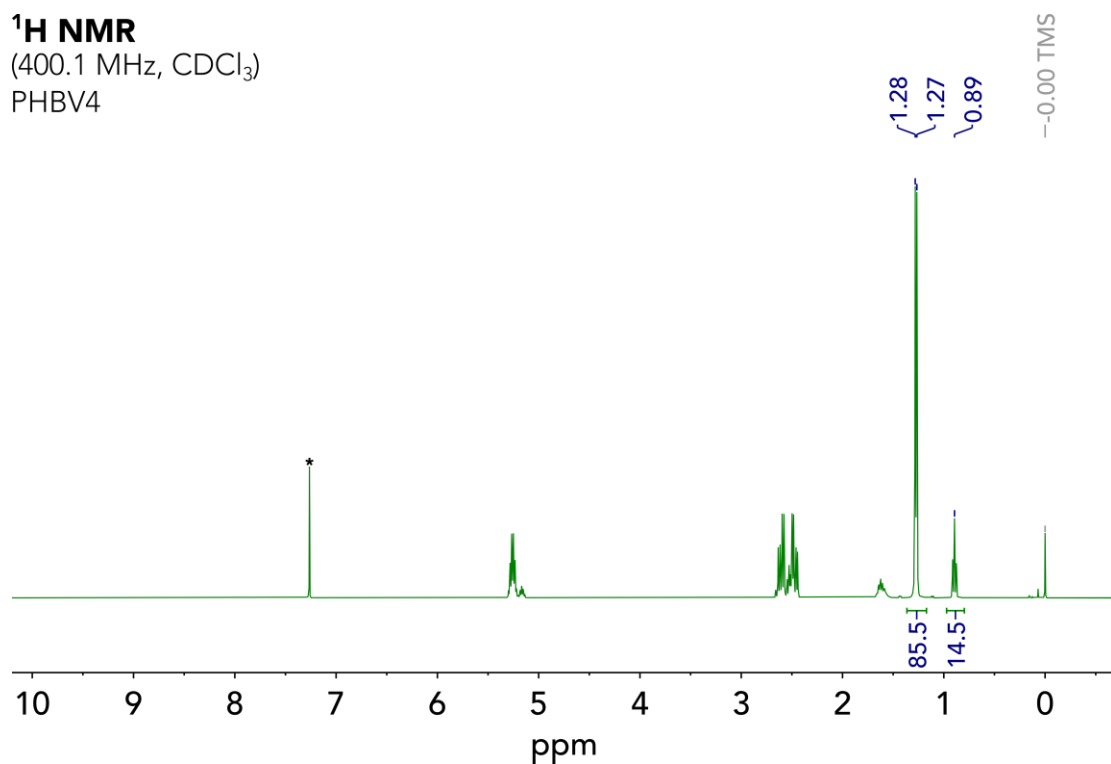


Figure S12. <sup>1</sup>H (400.1 MHz) NMR spectrum of PHBV4 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.



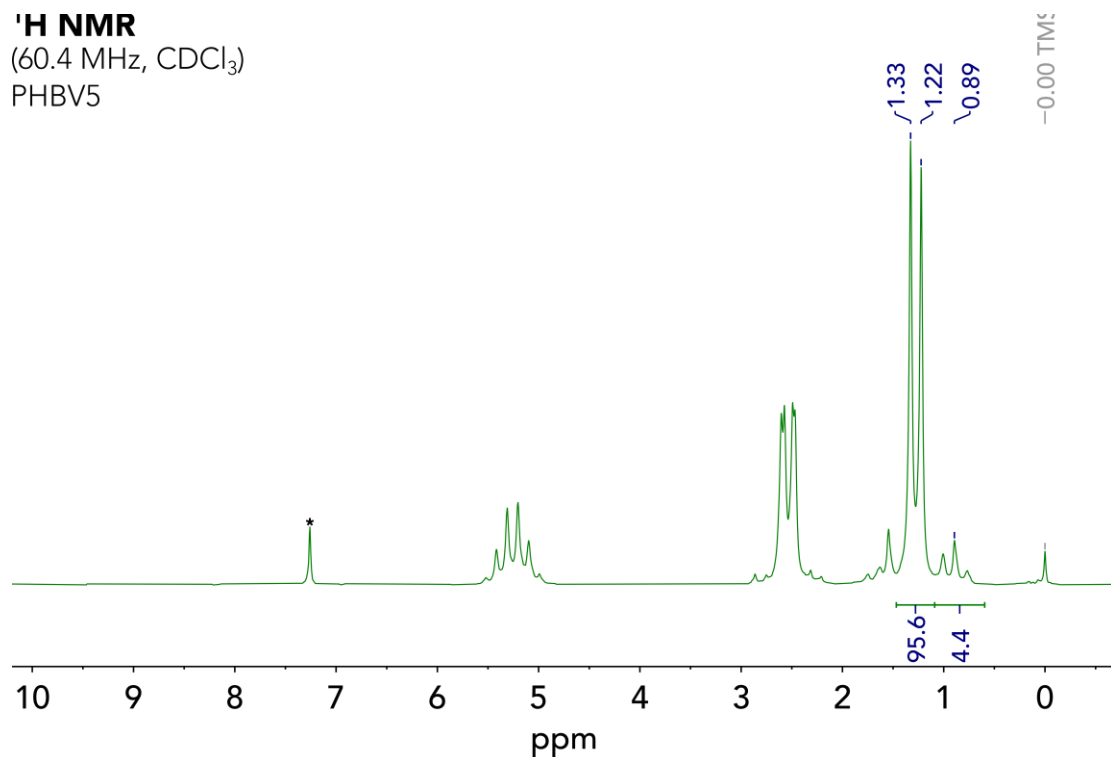


Figure S13. <sup>1</sup>H (60.4 MHz) NMR spectrum of PHBV5 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

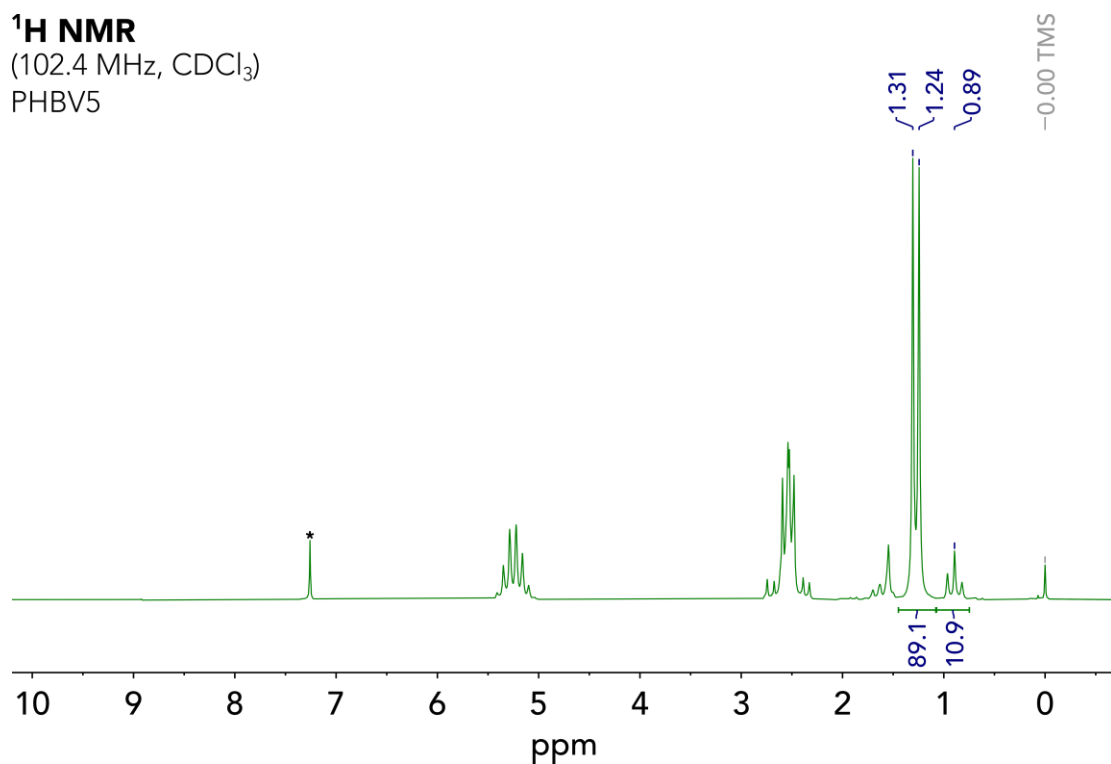


Figure S14. <sup>1</sup>H (102.4 MHz) NMR spectrum of PHBV5 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

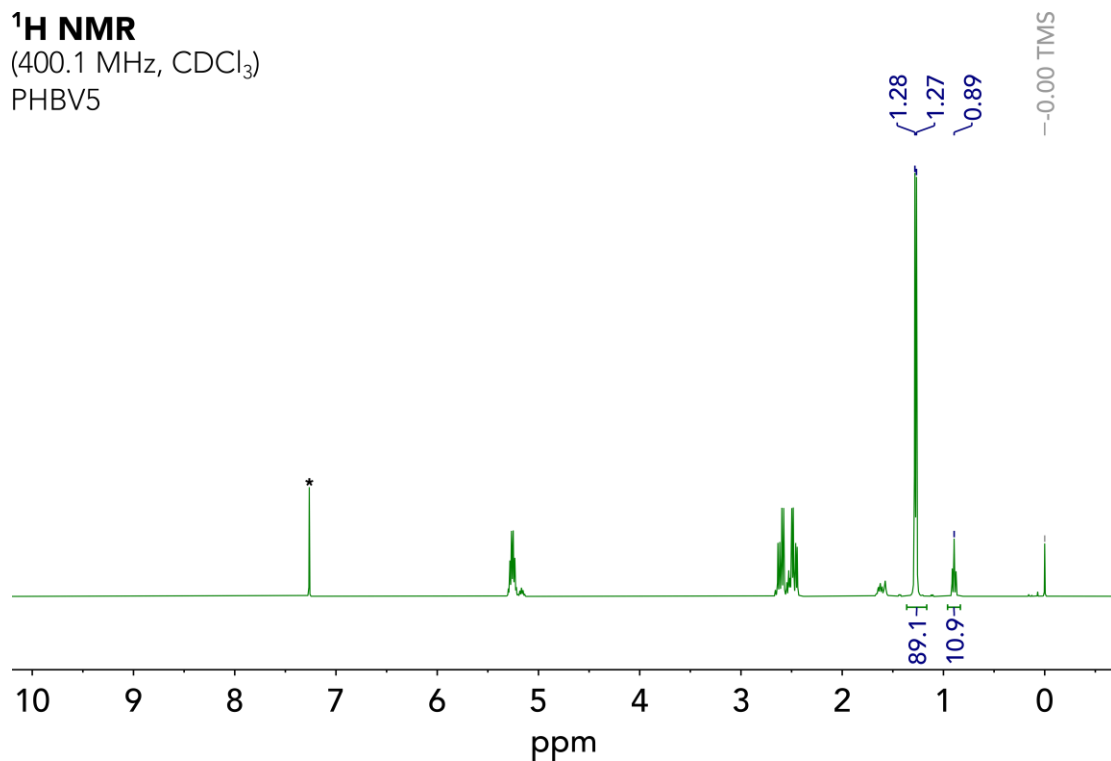


Figure S15. <sup>1</sup>H (400.1 MHz) NMR spectrum of PHBV5 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

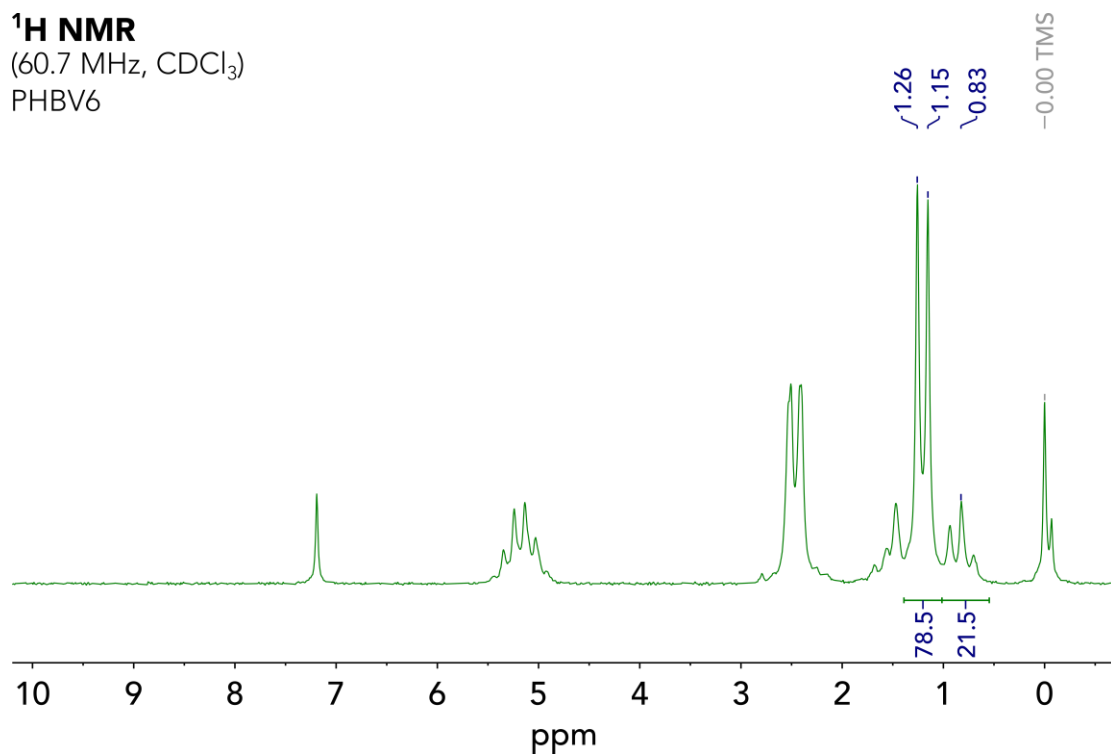


Figure S16. <sup>1</sup>H (60.7 MHz) NMR spectrum of PHBV6 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**

(102.6 MHz, CDCl<sub>3</sub>)

PHBV6

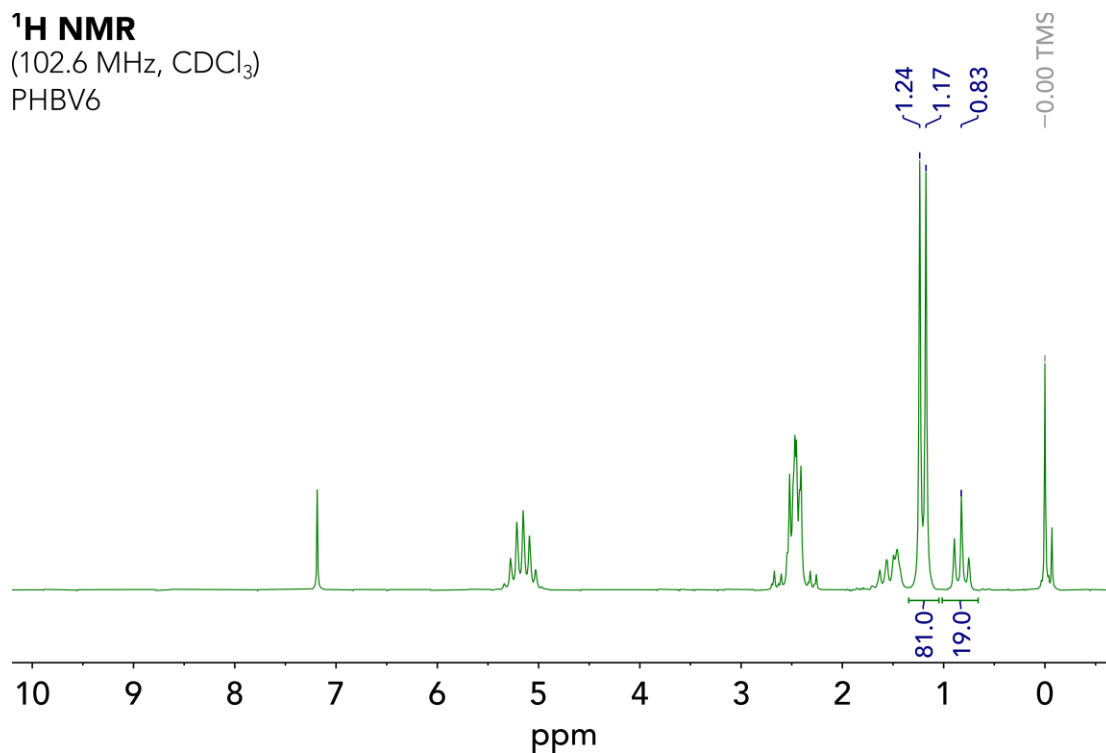


Figure S17. <sup>1</sup>H (102.6 MHz) NMR spectrum of PHBV6 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**

(400.1 MHz, CDCl<sub>3</sub>)

PHBV6

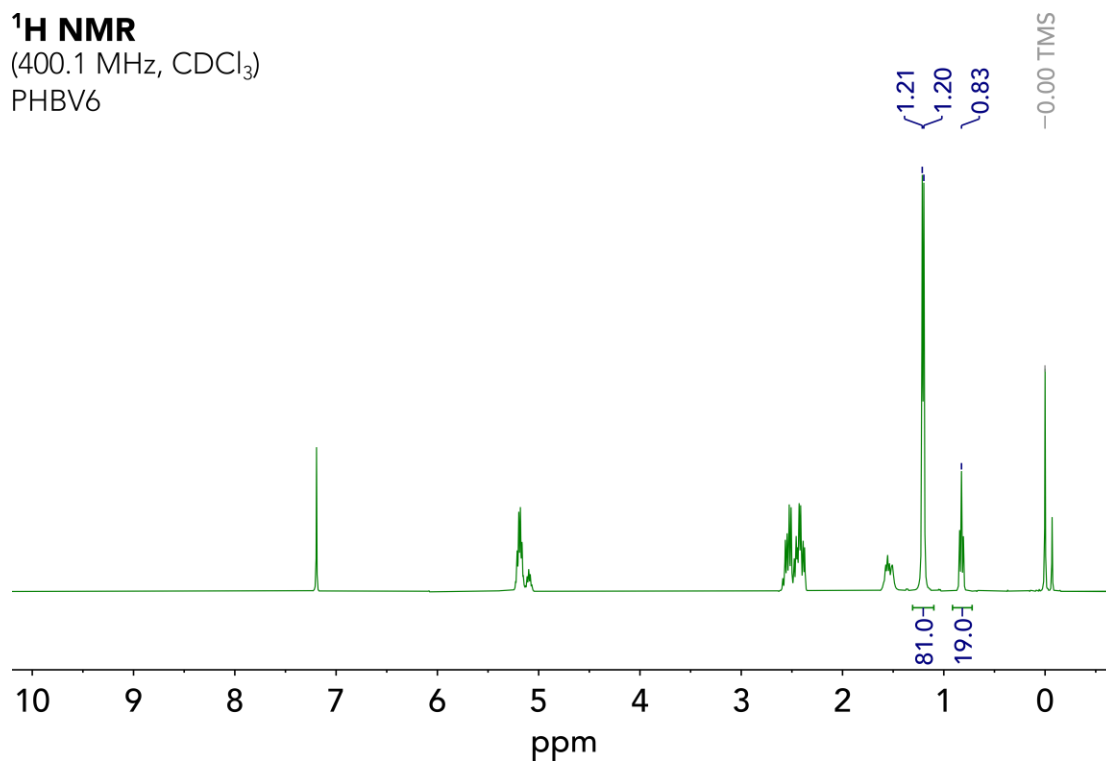


Figure S18. <sup>1</sup>H (400.1 MHz) NMR spectrum of PHBV6 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**

(59.5 MHz, CDCl<sub>3</sub>)

PHBV7

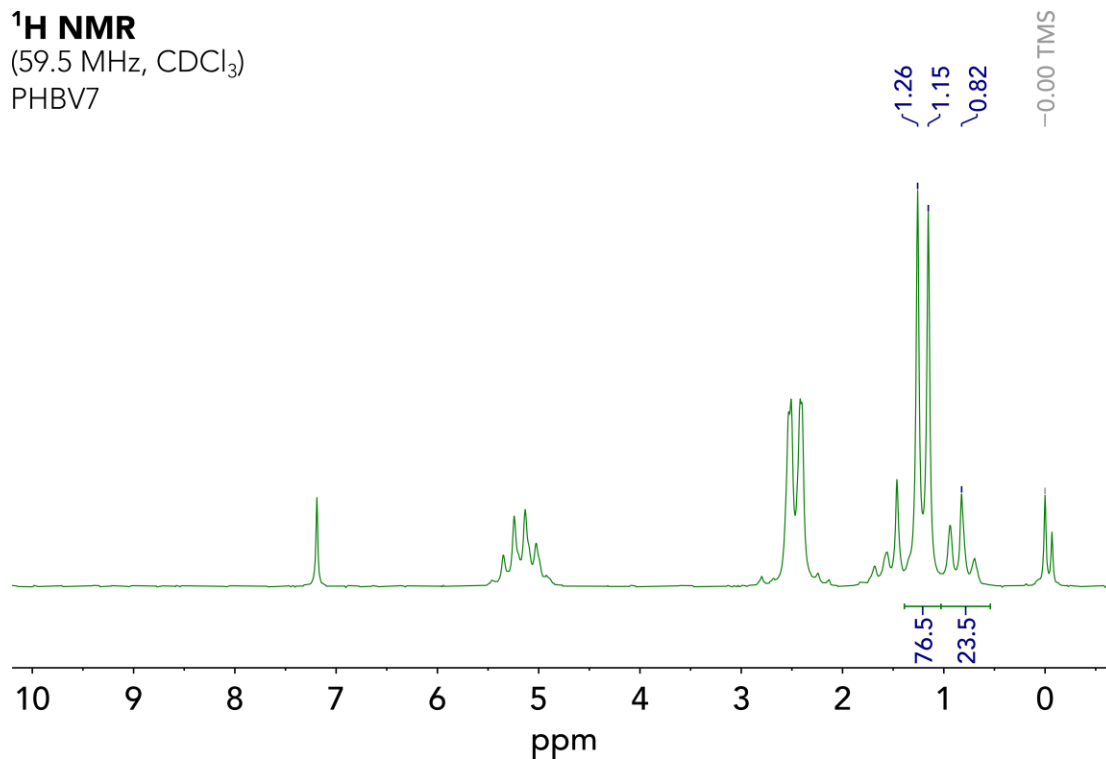


Figure S19. <sup>1</sup>H (59.5 MHz) NMR spectrum of PHBV7 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**

(102.6 MHz, CDCl<sub>3</sub>)

PHBV7

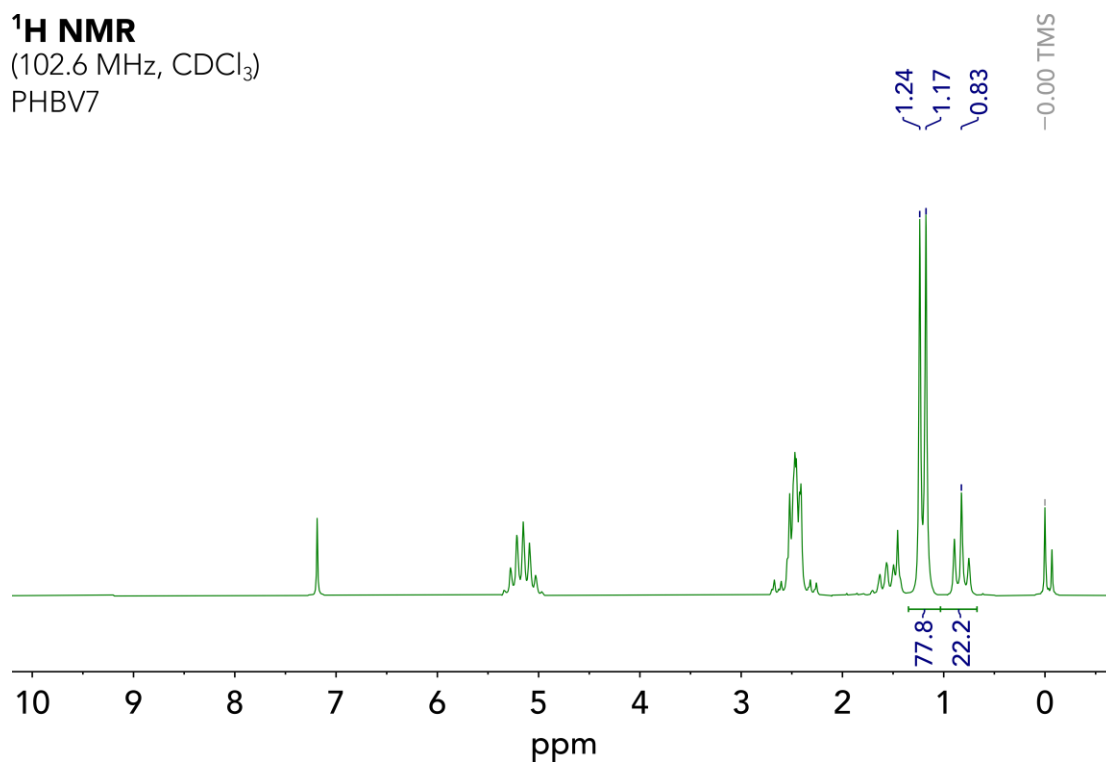


Figure S20. <sup>1</sup>H (102.6 MHz) NMR spectrum of PHBV7 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

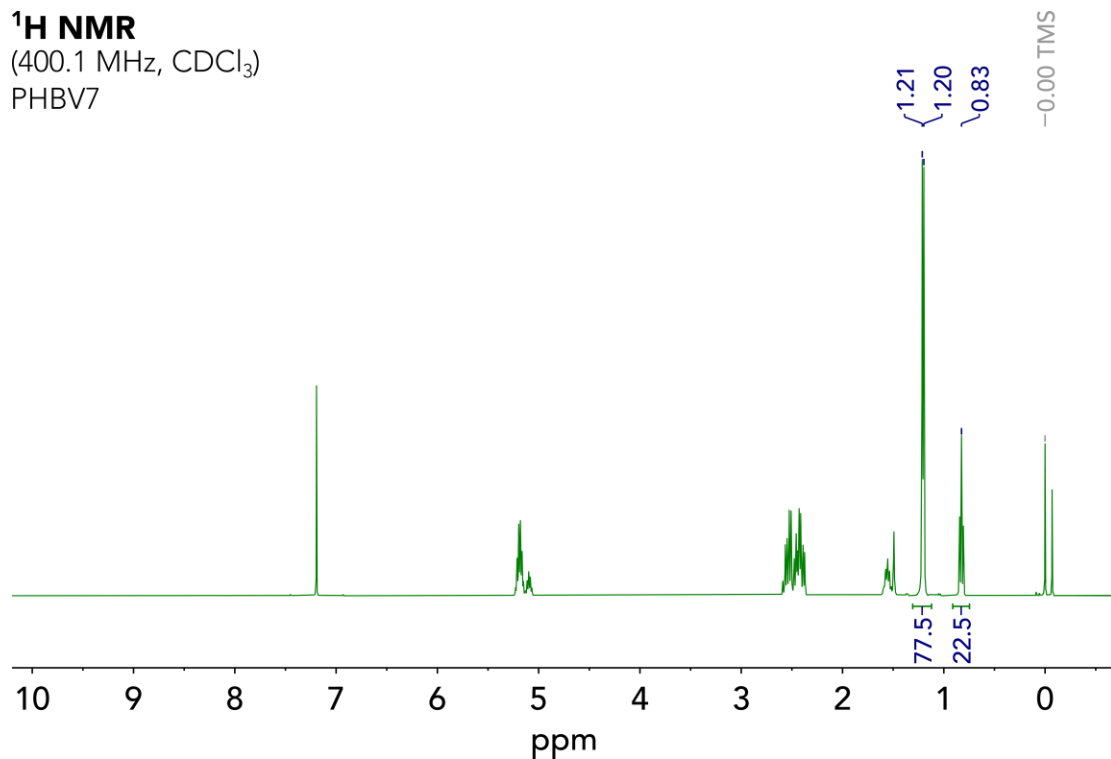


Figure S21. <sup>1</sup>H (400.1 MHz) NMR spectrum of PHBV7 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

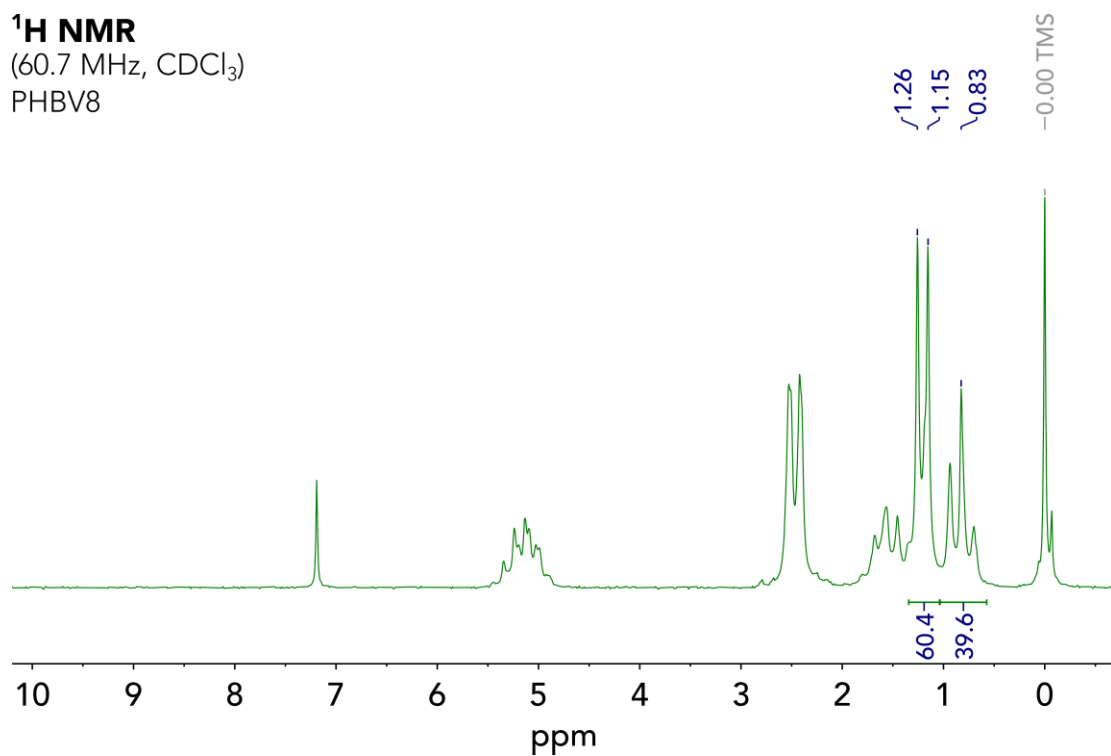


Figure S22. <sup>1</sup>H (60.7 MHz) NMR spectrum of PHBV8 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**  
(102.6 MHz, CDCl<sub>3</sub>)  
PHBV8

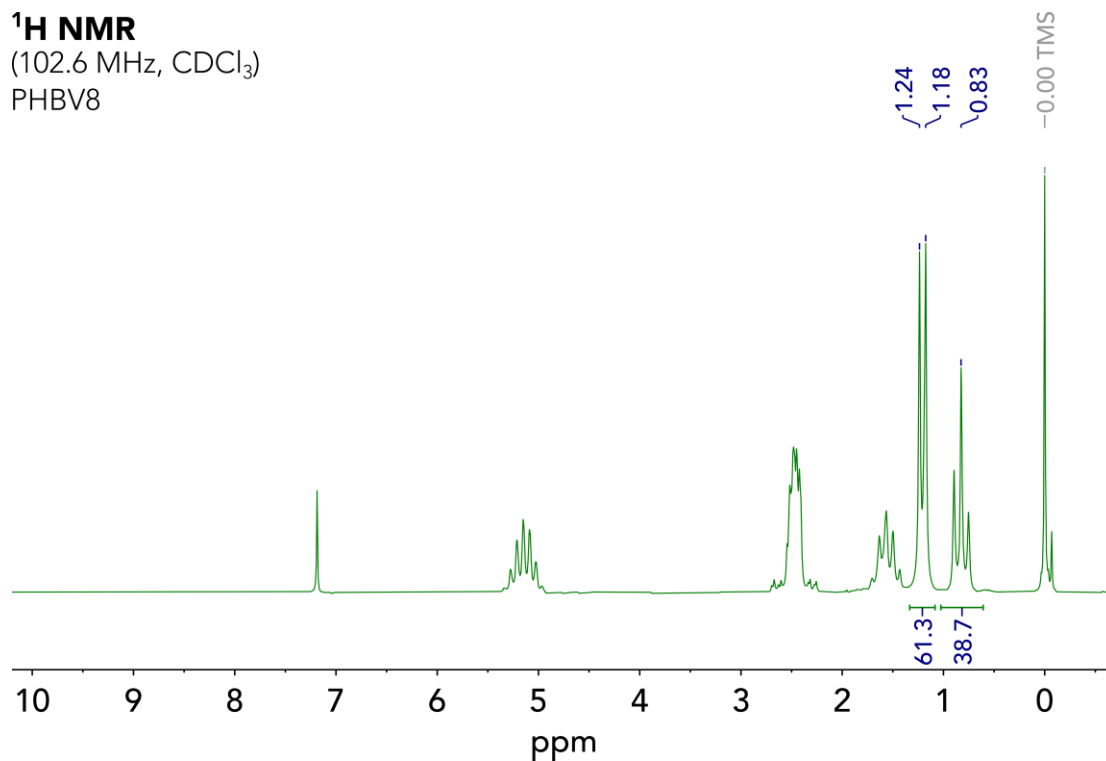


Figure S23. <sup>1</sup>H (102.6 MHz) NMR spectrum of PHBV8 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**  
(400.1 MHz, CDCl<sub>3</sub>)  
PHBV8

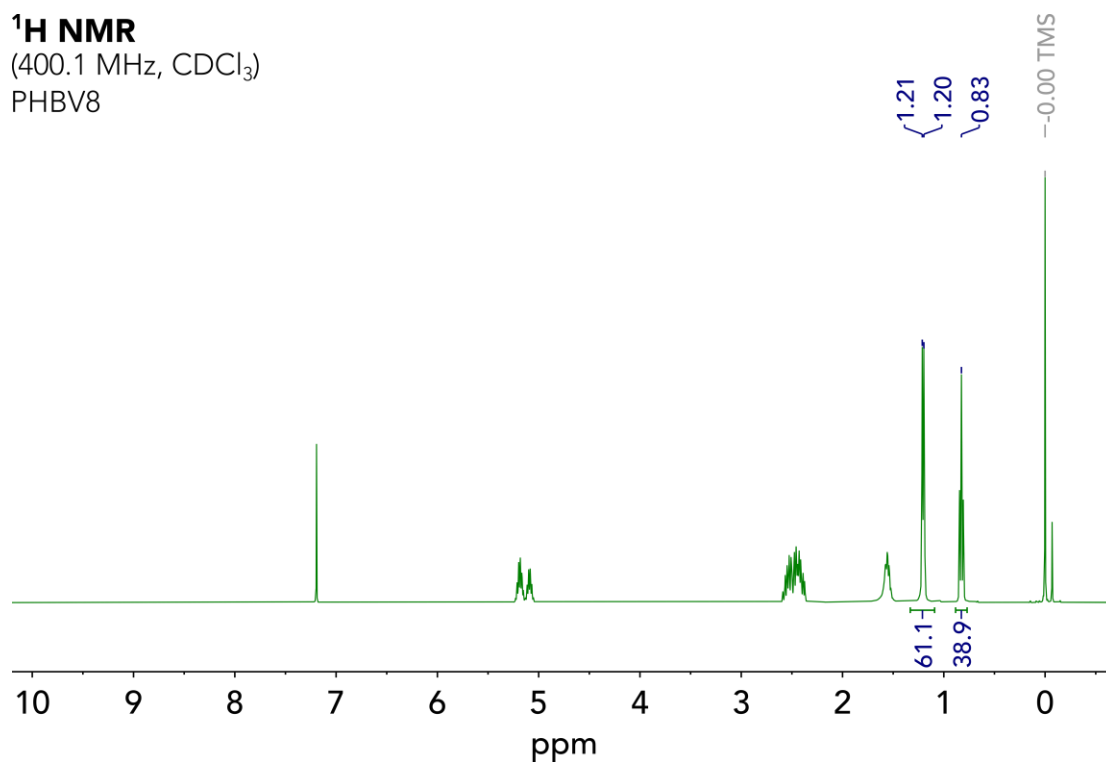


Figure S24. <sup>1</sup>H (400.1 MHz) NMR spectrum of PHBV8 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**  
(60.4 MHz, CDCl<sub>3</sub>)  
PHBV9

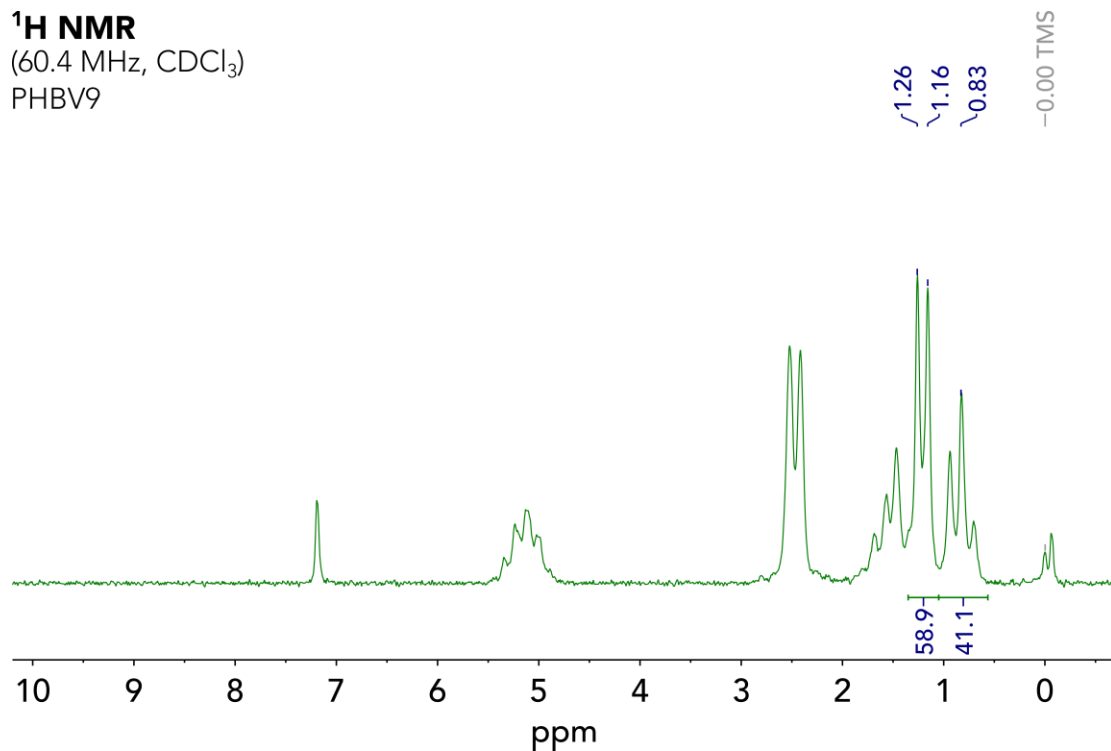


Figure S25. <sup>1</sup>H (60.4 MHz) NMR spectrum of PHBV9 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

**<sup>1</sup>H NMR**  
(102.6 MHz, CDCl<sub>3</sub>)  
PHBV9

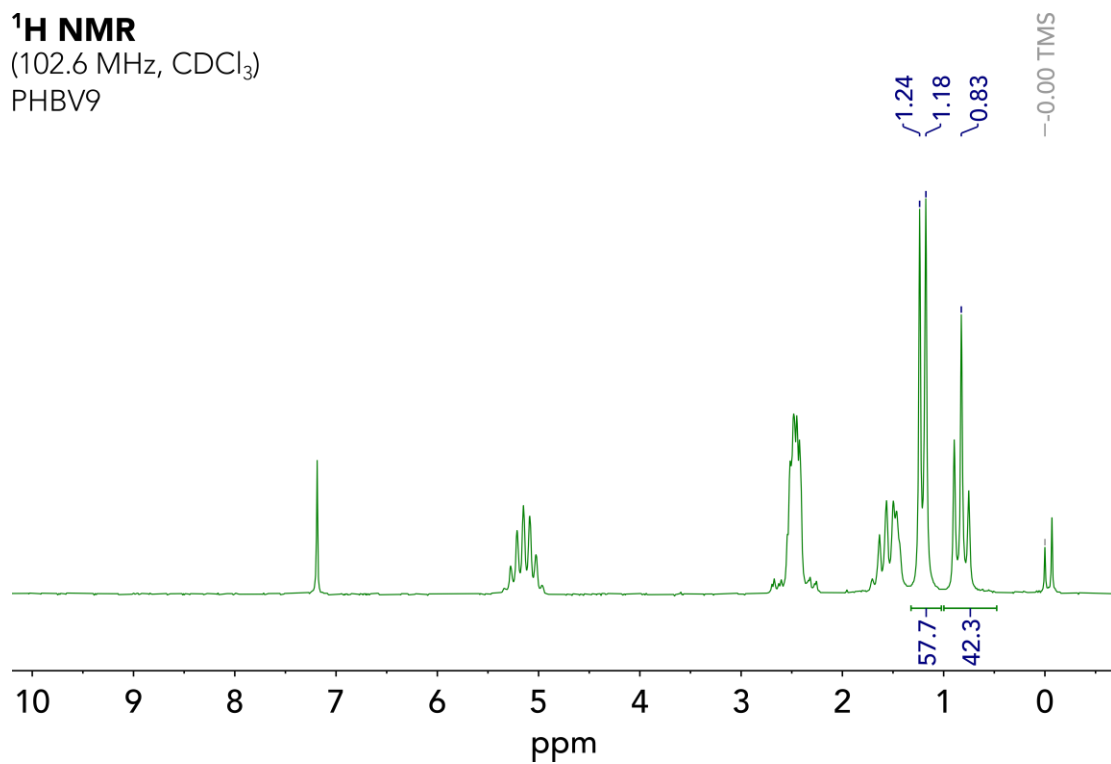


Figure S26. <sup>1</sup>H (102.6 MHz) NMR spectrum of PHBV9 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.

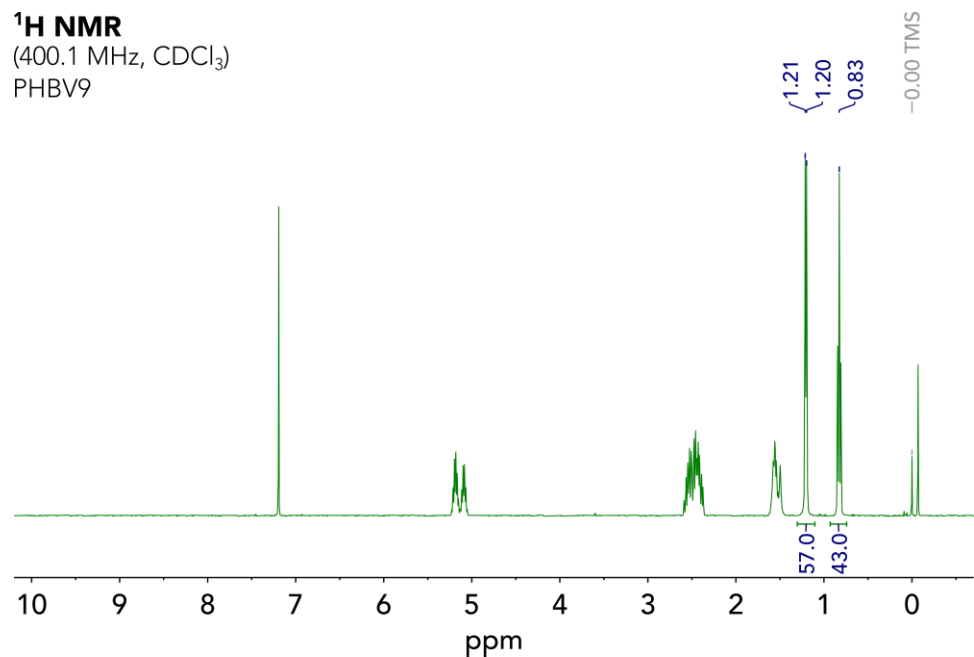


Figure S27. <sup>1</sup>H (400.1 MHz) NMR spectrum of PHBV9 (Assay 1, Run 1) in CDCl<sub>3</sub>. The asterisk represents the residual solvent peak for chloroform. The regions of interest are integrated.