

## 1. Title of Dataset

Characterizing  $^{13}\text{C}$  Spectral Assignments and Substituent Distributions of Hydroxypropylmethylcellulose Acetyl Succinate Using Dynamic Nuclear Polarization Nuclear Magnetic Resonance Spectroscopy

## 2. Authors list and Information

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## 3. Methodology and Hardware used to create the data

DNP MAS NMR experiments were recorded on a Bruker AVANCE III HD NMR spectrometer with a 9.4 T wide-bore magnet and a 263.334 GHz gyrotron source, using a Bruker 3.2 mm low temperature double resonance DNP  $^1\text{H}/^{13}\text{C}$  MAS NMR probe.

Room temperature  $^{13}\text{C}$  CP MAS NMR experiments were recorded on a Bruker AVANCE III HD NMR 9.4 T spectrometer with a 4mm triple-resonance HXY MAS probe.

Scanning electron microscopy (SEM) was performed on a Hitachi S4800.

Standard differential scanning calorimetry (DSC) was performed on a TA Instruments DSC Discovery using a TA-Tzero aluminium pan.

More experimental details can be found in the linked publication.

## 4. Software used to process the data

All NMR data were acquired and processed using Bruker TopSpin 4.0.6 software.

## 5. Data file overview

| File name                    | Description                                                                    |
|------------------------------|--------------------------------------------------------------------------------|
| Figure 2 blue                | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) at 107 K                            |
| Figure 2 red                 | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) at 298 K                            |
| Figure 3b black              | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) with microwave                      |
| Figure 3b gray               | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) without microwave                   |
| Figure 3c                    | $^{13}\text{C}$ - $^{13}\text{C}$ refocused INADEQUATE spectrum of HPMC-AS(LF) |
| Figure 4                     | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) with microwave                      |
| Figure 5a blue               | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) with microwave                      |
| Figure 5a gray               | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) without microwave                   |
| Figure 5b blue               | $^{13}\text{C}$ CP spectrum of HPMC-AS(MF) with microwave                      |
| Figure 5b gray               | $^{13}\text{C}$ CP spectrum of HPMC-AS(MF) without microwave                   |
| Figure 5c blue               | $^{13}\text{C}$ CP spectrum of HPMC-AS(HF) with microwave                      |
| Figure 5c gray               | $^{13}\text{C}$ CP spectrum of HPMC-AS(HF) without microwave                   |
| Figure 6a1 with microwave    | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) with microwave                      |
| Figure 6a1 without microwave | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) without microwave                   |
| Figure 6a2 with microwave    | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) with microwave                      |
| Figure 6a2 without microwave | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) without microwave                   |
| Figure 6a3 with microwave    | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) with microwave                      |
| Figure 6a3 without microwave | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) without microwave                   |
| Figure 6b1 with microwave    | $^{13}\text{C}$ CP spectrum of HPMC-AS(MF) with microwave                      |
| Figure 6b1 without microwave | $^{13}\text{C}$ CP spectrum of HPMC-AS(MF) without microwave                   |
| Figure 6b2 with microwave    | $^{13}\text{C}$ CP spectrum of HPMC-AS(MF) with microwave                      |
| Figure 6b2 without microwave | $^{13}\text{C}$ CP spectrum of HPMC-AS(MF) without microwave                   |
| Figure 6b3 with microwave    | $^{13}\text{C}$ CP spectrum of HPMC-AS(MF) with microwave                      |
| Figure 6b3 without microwave | $^{13}\text{C}$ CP spectrum of HPMC-AS(MF) without microwave                   |
| Figure 6c1 with microwave    | $^{13}\text{C}$ CP spectrum of HPMC-AS(HF) with microwave                      |
| Figure 6c1 without microwave | $^{13}\text{C}$ CP spectrum of HPMC-AS(HF) without microwave                   |
| Figure 6c2 with microwave    | $^{13}\text{C}$ CP spectrum of HPMC-AS(HF) with microwave                      |
| Figure 6c2 without microwave | $^{13}\text{C}$ CP spectrum of HPMC-AS(HF) without microwave                   |
| Figure 6c3 with microwave    | $^{13}\text{C}$ CP spectrum of HPMC-AS(HF) with microwave                      |
| Figure 6c3 without microwave | $^{13}\text{C}$ CP spectrum of HPMC-AS(HF) without microwave                   |
| Figure S2                    | Standard DSC measurement data                                                  |
| Figure S3a                   | $^{13}\text{C}$ CP spectrum of HPMC-AS(LF) with microwave                      |
| Figure S3b                   | $^{13}\text{C}$ - $^{13}\text{C}$ refocused INADEQUATE spectrum of HPMC-AS(LF) |
| Figure S4a blue              | $^{13}\text{C}$ CP echo filter HPMC-AS(LF) with microwave                      |
| Figure S4a gray              | $^{13}\text{C}$ CP echo filter HPMC-AS(LF) without microwave                   |
| Figure S4b blue              | $^{13}\text{C}$ CP echo filter HPMC-AS(MF) with microwave                      |
| Figure S4b gray              | $^{13}\text{C}$ CP echo filter HPMC-AS(MF) without microwave                   |
| Figure S4c blue              | $^{13}\text{C}$ CP echo filter HPMC-AS(HF) with microwave                      |
| Figure S4c gray              | $^{13}\text{C}$ CP echo filter HPMC-AS(HF) without microwave                   |