

Chemical interaction between N,N'-diisopropylcarbodiimide and N-hydroxysuccinimide by infrared spectroscopy

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ABSTRACT An equimolar mixture of N,N'-diisopropylcarbodiimide (DIC) and N-hydroxysuccinimide (NHS) yielded a residue with characteristic infrared spectrum of a bimolecular complex.

Keywords: N,N'-Diisopropylcarbodiimide, N-hydroxysuccinimide, and infrared spectroscopy

A mixture of N,N'-diisopropylcarbodiimide (DIC) and N-hydroxysuccinimide (NHS) each at 145 mM in anhydrous acetonitrile resulted in precipitate formation within minutes. Heating the mixture to 60 °C for 30 min dissolved the precipitate. Cooling to RT resulted in the formation of crystals. After overnight incubation at RT, the solvent in the reaction mixture was removed under reduced pressure. The residue was characterized utilizing attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy on the PerkinElmer Spectrum 100 spectrometer. Spectra (Figure 1) are the average of 4 scans at 4 cm⁻¹ resolution.

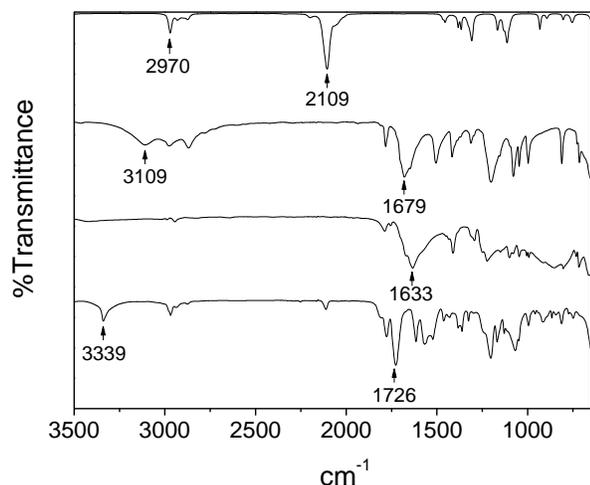


Figure 1 ATR-FTIR spectra. From top to bottom: DIC, NHS, NHS Na⁺ salt, and residue of the DIC+NHS mix.

Assignments of selected ATR-FTIR stretching bands are listed on Table 1. Infrared (IR) spectral changes of

the DIC+NHS mix were compared to pure DIC and NHS. The IR spectrum of the mix had near complete or complete signal loss for the N=C=N stretch of DIC and the O-H stretch of NHS, respectively. In addition, the new IR band at 3339 cm⁻¹ can be assigned to N-H vibration. Another new IR band centered at 1726 cm⁻¹ can be assigned to shifted C=O stretching vibration on NHS. This C=O stretching band shifted to lower wavenumber (1633 cm⁻¹) on the sodium salt of NHS instead. Hence, infrared spectroscopy suggested the formation of a bimolecular complex (Figure 2) resulting from the mixing of DIC with NHS. To prepare the sodium salt of NHS, a solution of 30 mg NHS in purified water was titrated to pH 7.5 with 5 M NaOH and the solution was freeze-dried.

	cm ⁻¹	Assignment
DIC	2970	C-H
	2109	N=C=N
NHS	3109	O-H
	1679	C=O
NHS Na ⁺ salt	1633	C=O
DIC+NHS	3339	N-H
	1726	C=O

Table 1 Assignment of selected ATR-FTIR stretching bands.

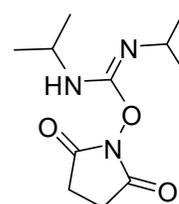


Figure 2 Structure of the proposed product that formed upon mixing DIC with NHS.