

Supporting Information
for
A Procedure for the Preparation and Isolation of
Nucleoside-5'-Diphosphates

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Experimental procedures for the preparation of 5'-tosylates and their ¹H and ¹³C NMR spectra, and ³¹P and ¹H NMR spectra of NDPs

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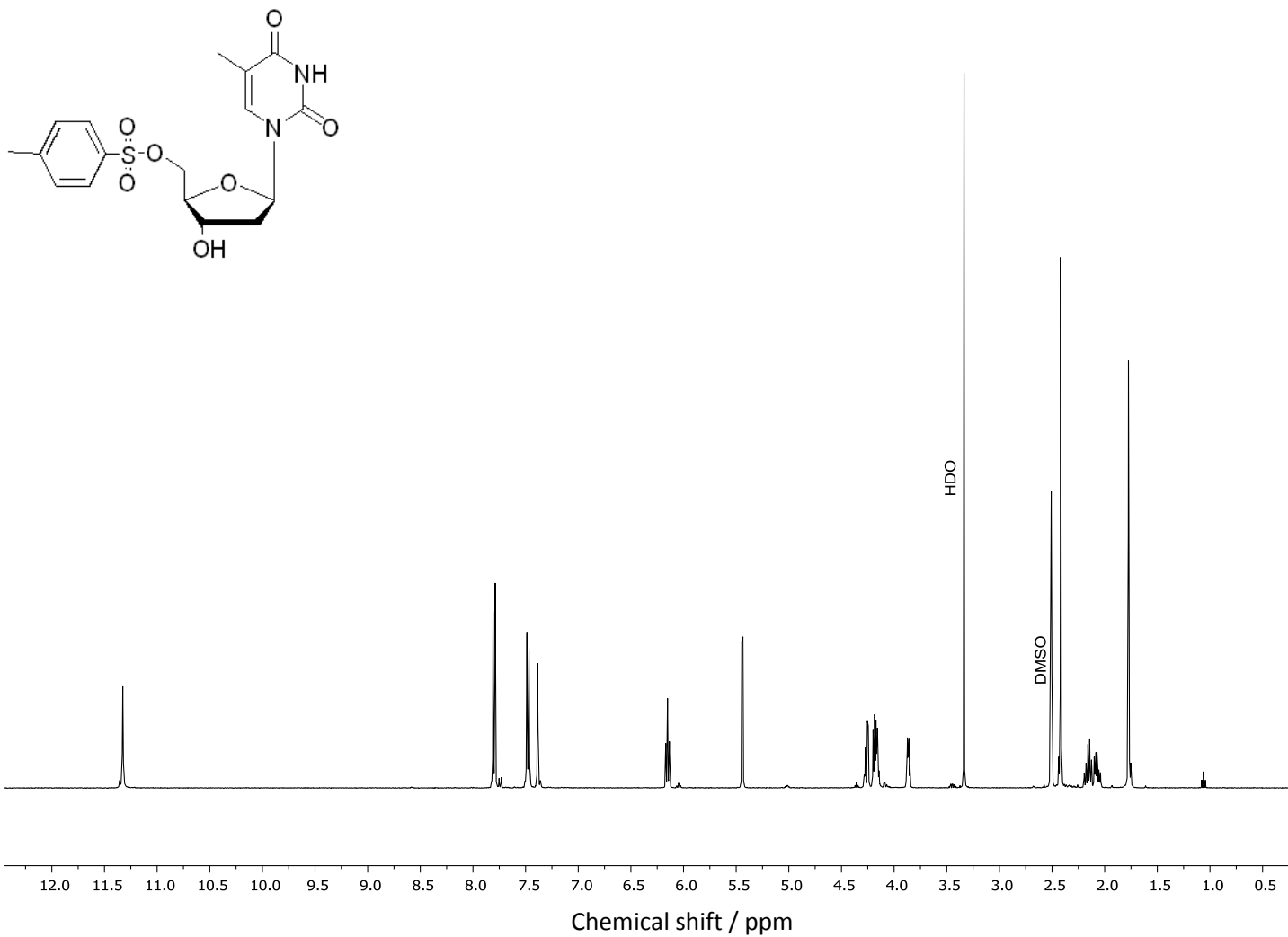
The Preparation of 5'-Tosylates

2',3'-O-Isopropylideneuridine and 5'-tosyladenosine **1d** were purchased from Carbosynth and used as received. 5'-tosylthymidine was synthesized according to a literature procedure [1] to give **1a** as a white powder (1.70 g, 51%). 2'-Deoxy-5'-tosyladenosine **1c** was prepared from 2'-deoxyadenosine hydrate via a published procedure [2] to yield 1.97 g (60%) of **1c**.

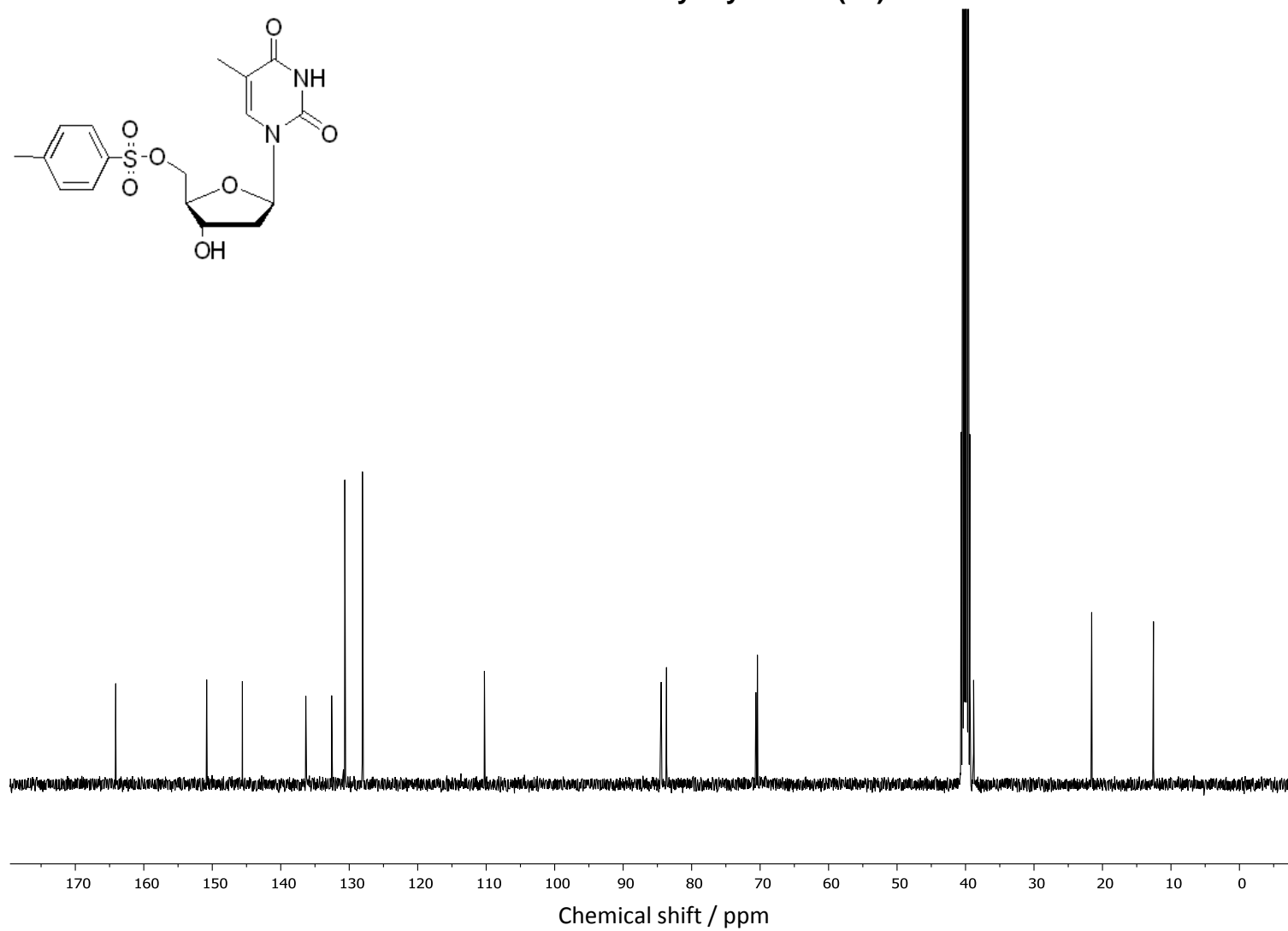
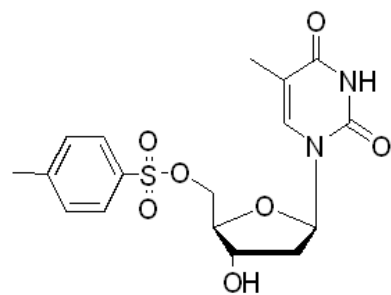
2',3'-O-Isopropylidene-5'-O-tosyluridine **3**: p-toluenesulfonyl chloride (1.34 g, 7.00 mmol) was dissolved in pyridine (5 ml) and added dropwise to a solution of 2',3'-O-isopropylideneuridine (1.60 g, 5.63 mmol) in dry pyridine (10 ml) at 0 °C, and the mixture was stirred at 0 °C for 2 h. The mixture was added to an ice/water mixture (100 ml), stirred for 20 min. and extracted with ethyl acetate (2 × 120 ml). The organic layers were combined and washed with saturated sodium hydrogen carbonate solution (2 × 20 ml) and distilled water (2 × 20 ml), and dried over magnesium sulfate. The solution was filtered, the solvent removed *in vacuo*, and the residue was subjected to silica gel chromatography (CH₂Cl₃:MeOH, 50:1) to yield 1.62 g (65%) of 2',3'-O-isopropylidene-5'-O-toluenesulfonyluridine. ¹H and ¹³C NMR spectra correspond to data in the literature [3,4].

5'-O-Tosyluridine **1b**: 2',3'-O-isopropylidene-5'-O-toluenesulfonyluridine (0.740 g, 1.69 mmol) was stirred in 90% TFA in water (2 ml) for 10 min. The solvent was removed *in vacuo* and the product was isolated by silica gel chromatography (CH₂Cl₂:MeOH, 9:1) to yield 0.483 g (71%) of 5'-O-tosyluridine. ¹H and ¹³C NMR spectra correspond to data in the literature [1].

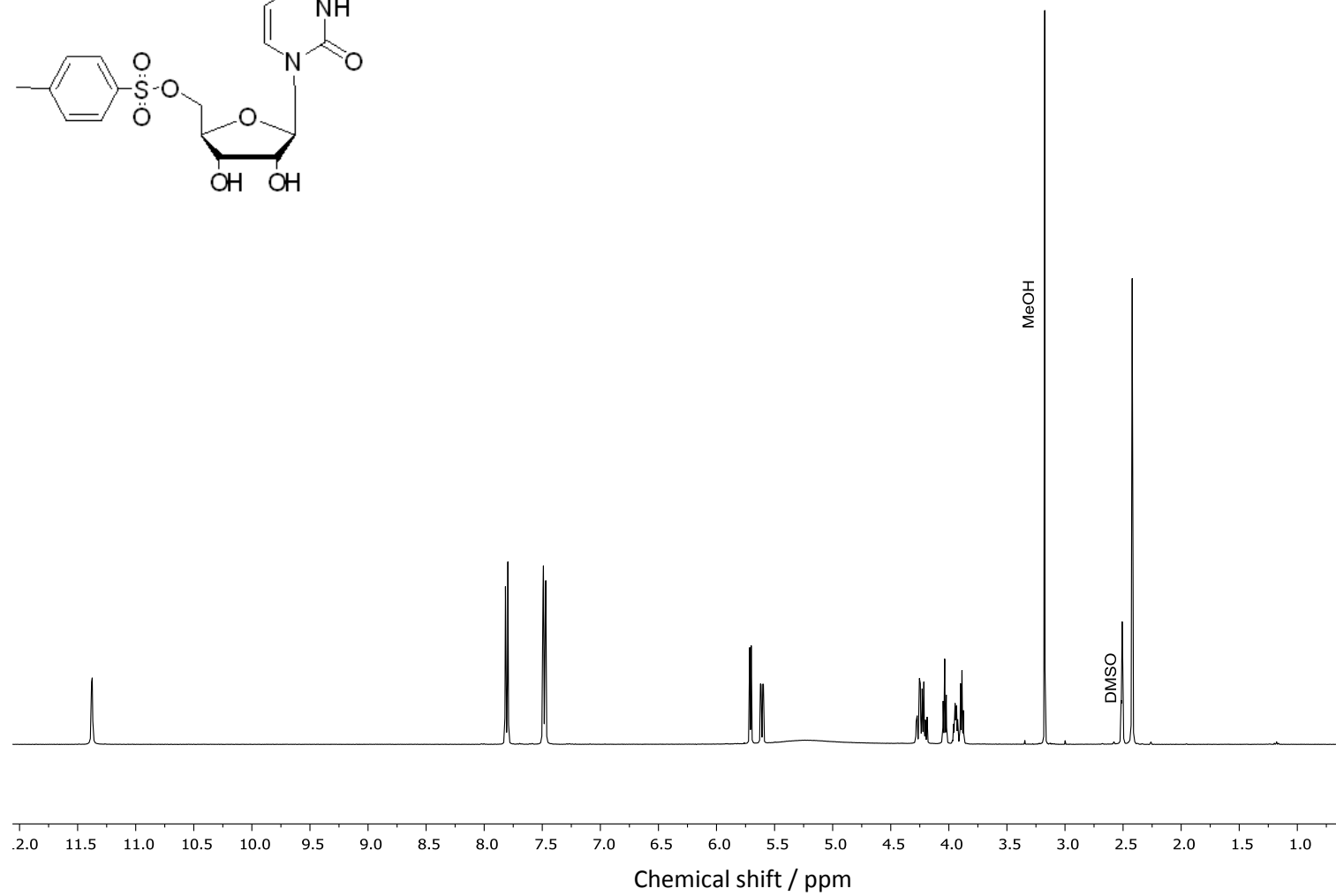
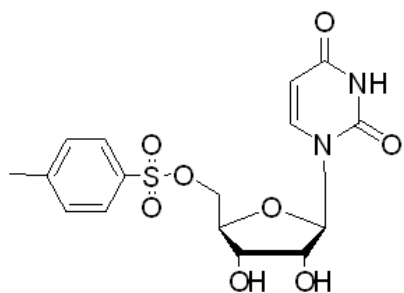
¹H NMR of 5'-O-tosylthymidine (1a)



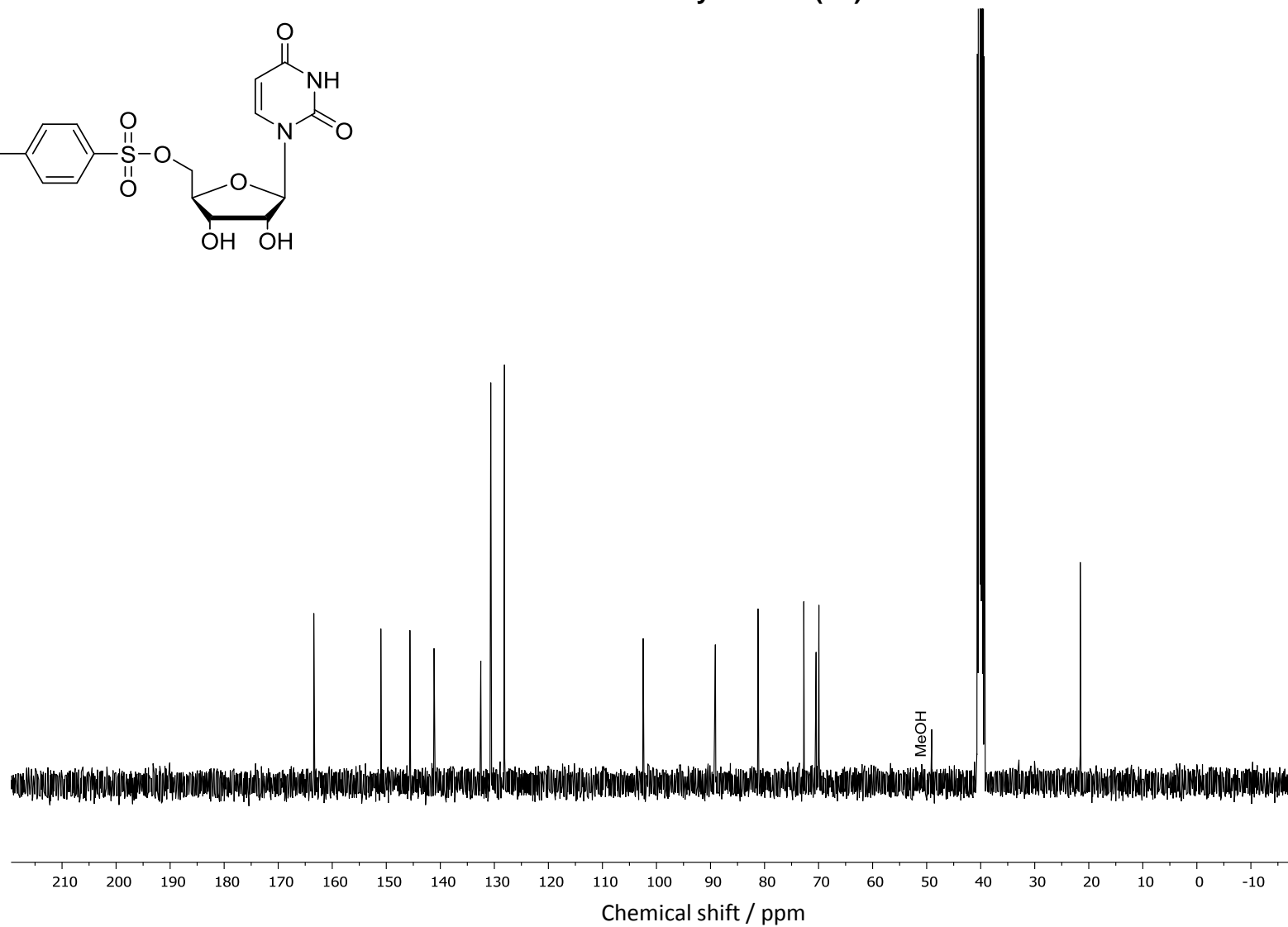
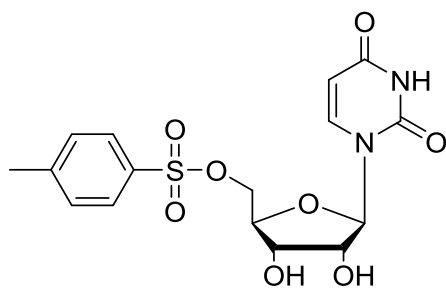
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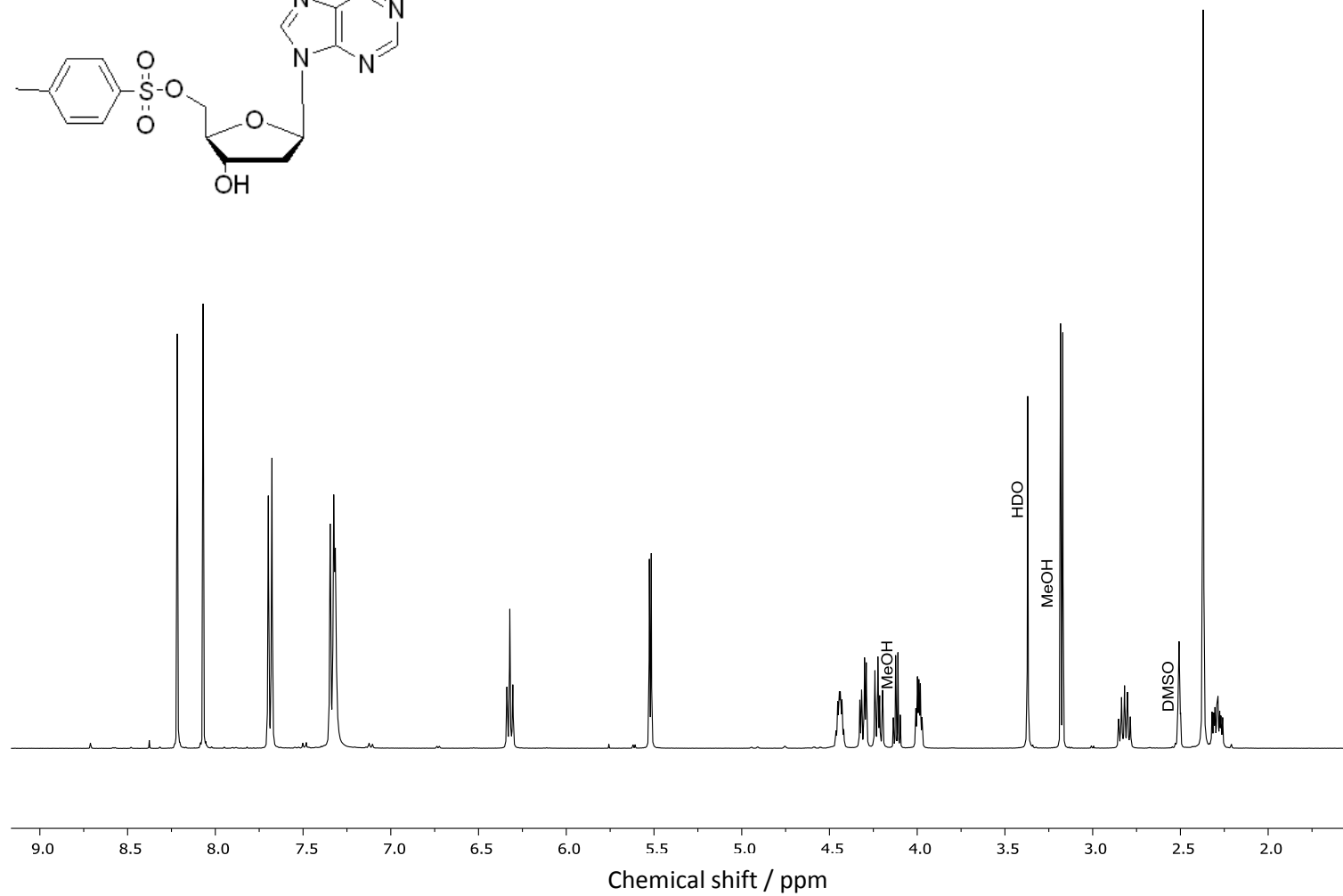
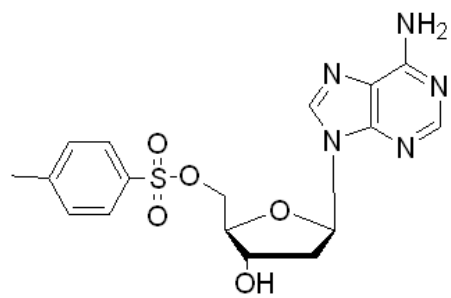
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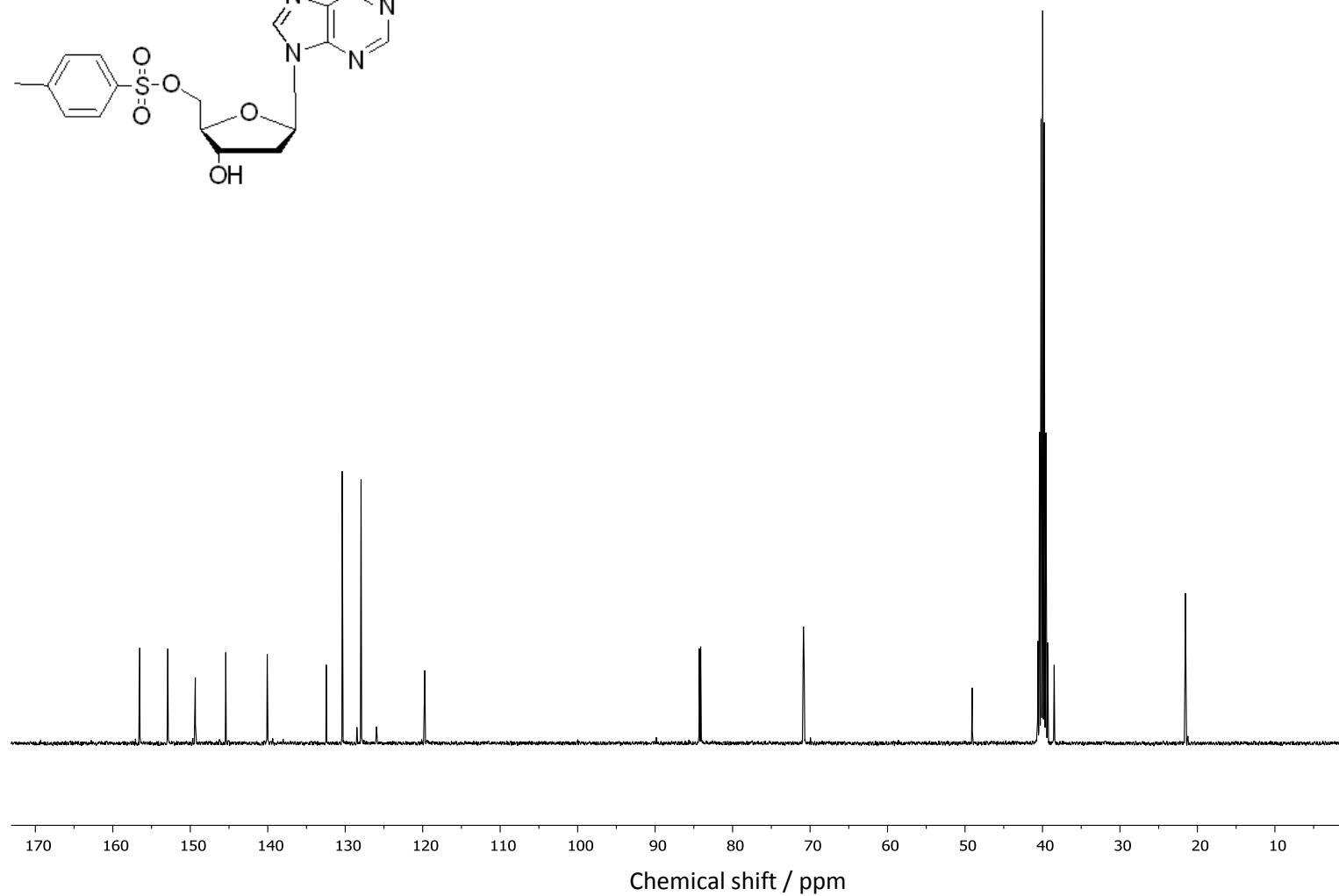
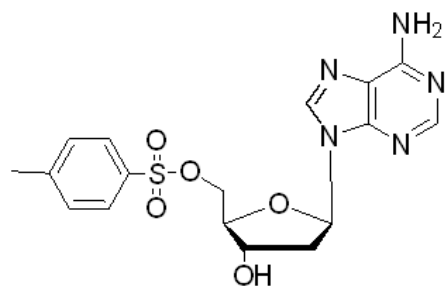
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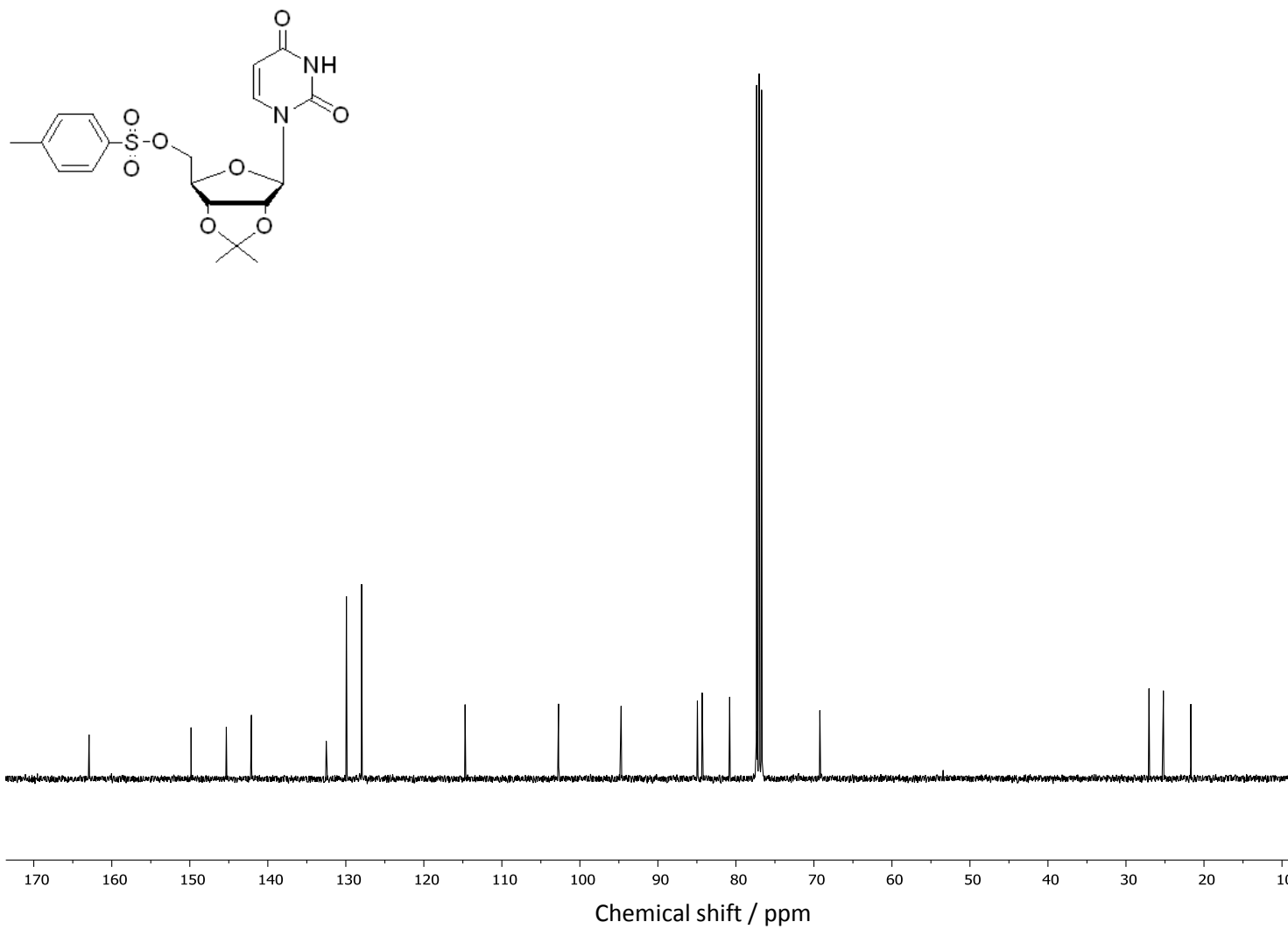
¹H NMR of 2'-deoxy-5'-O-tosyladenosine (1c)



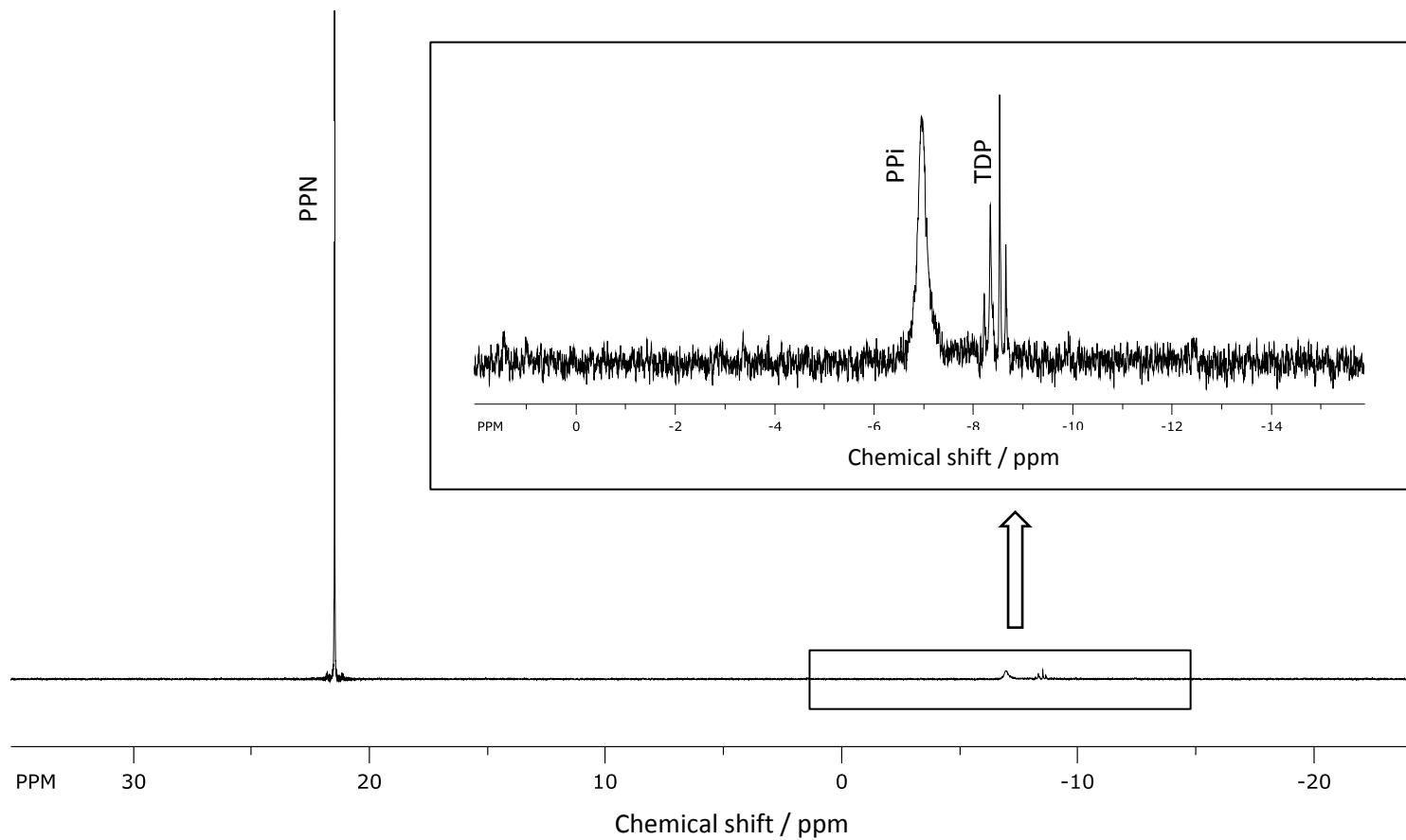
¹³C NMR of 2'-deoxy-5'-O-tosyladenosine (1c)



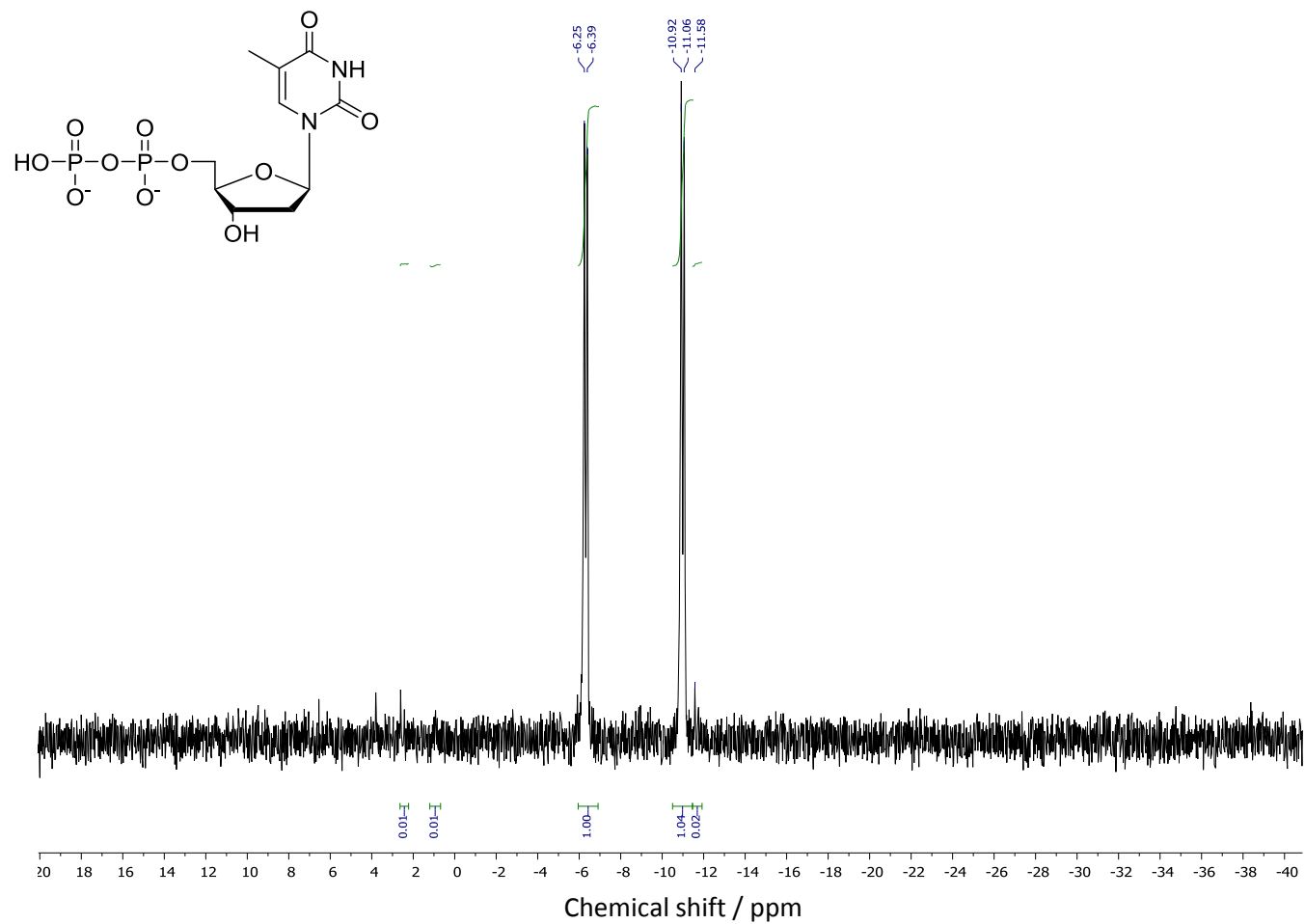
^{13}C NMR of 2',3'-O-isopropylidene-5'-O-tosyluridine (3)



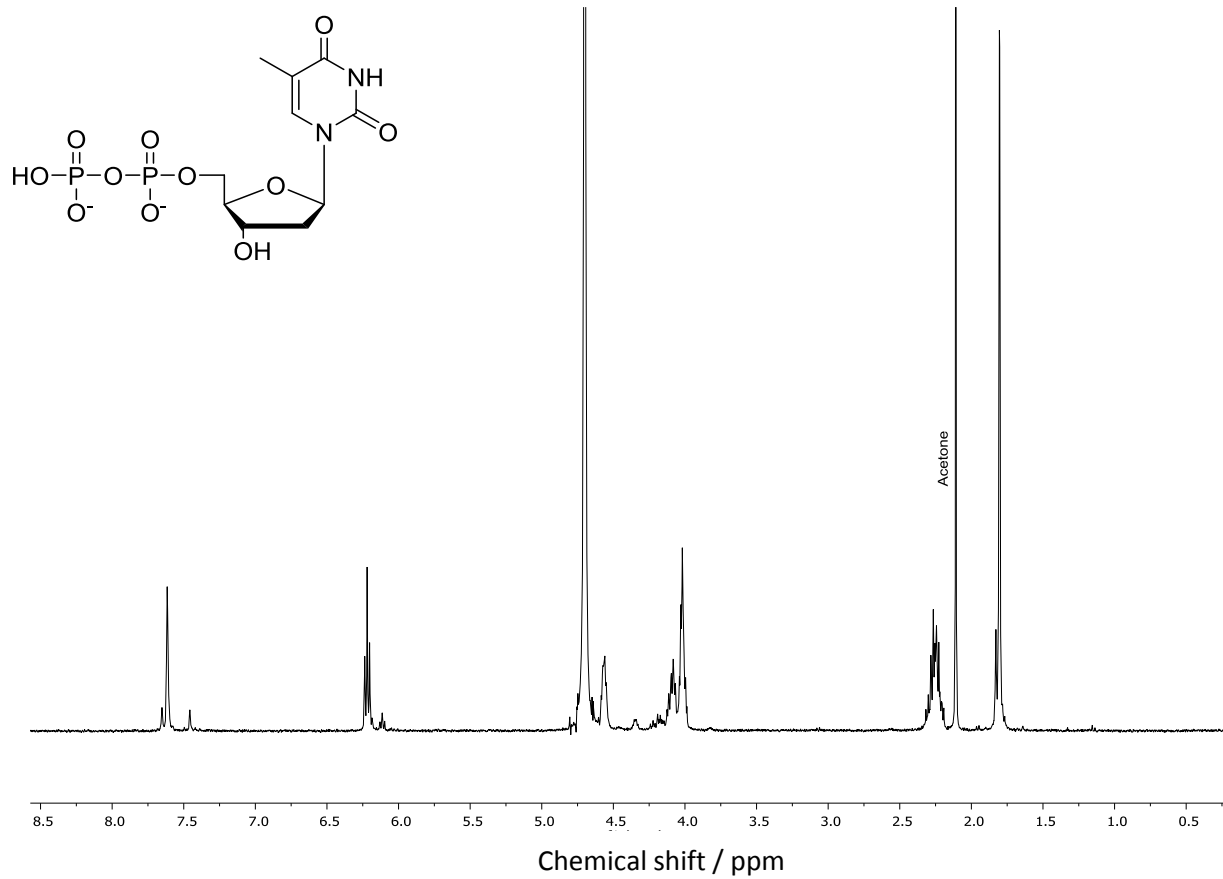
³¹P NMR of crude TDP reaction mixture



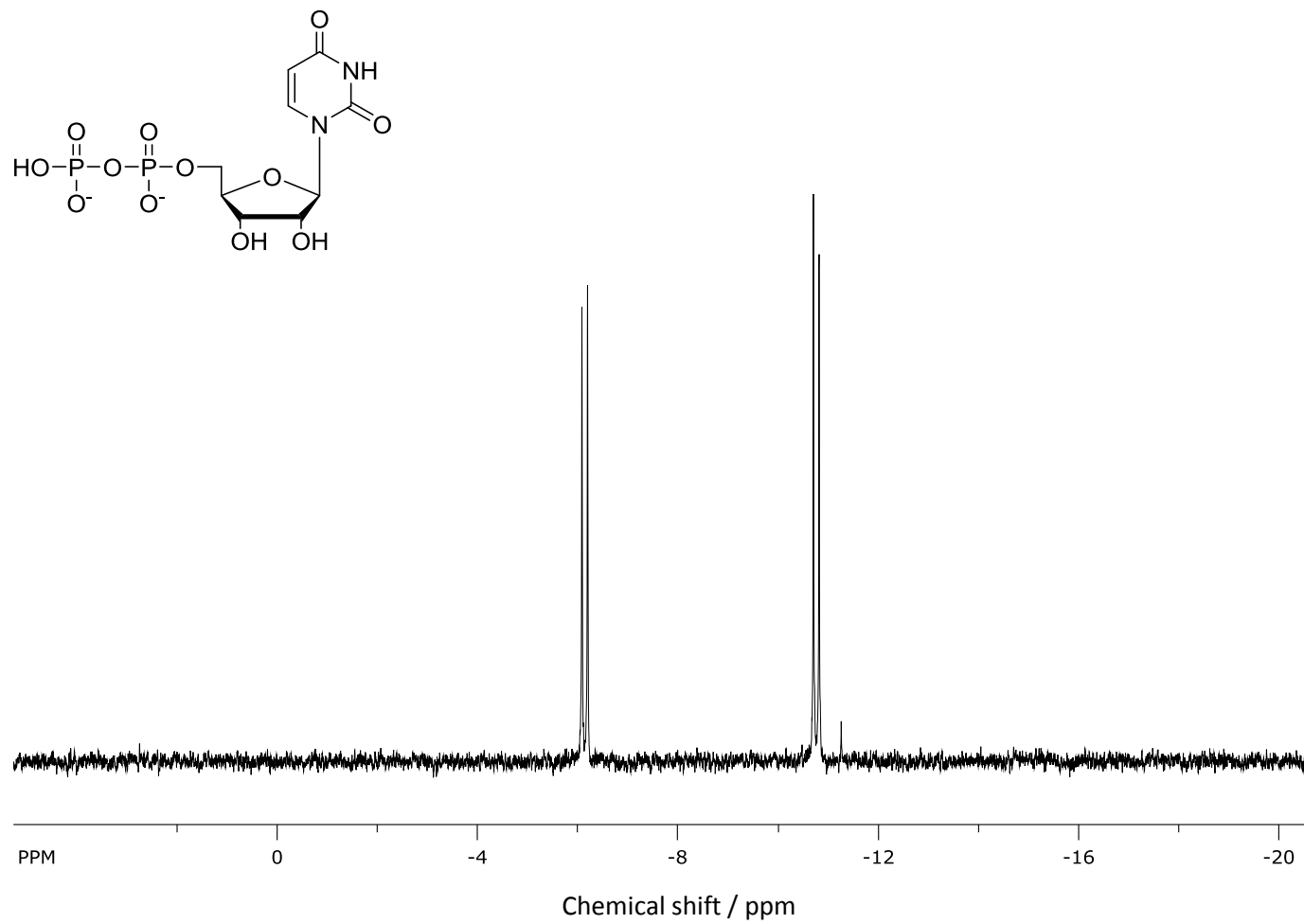
³¹P NMR of thymidine 5'-diphosphate (2a)



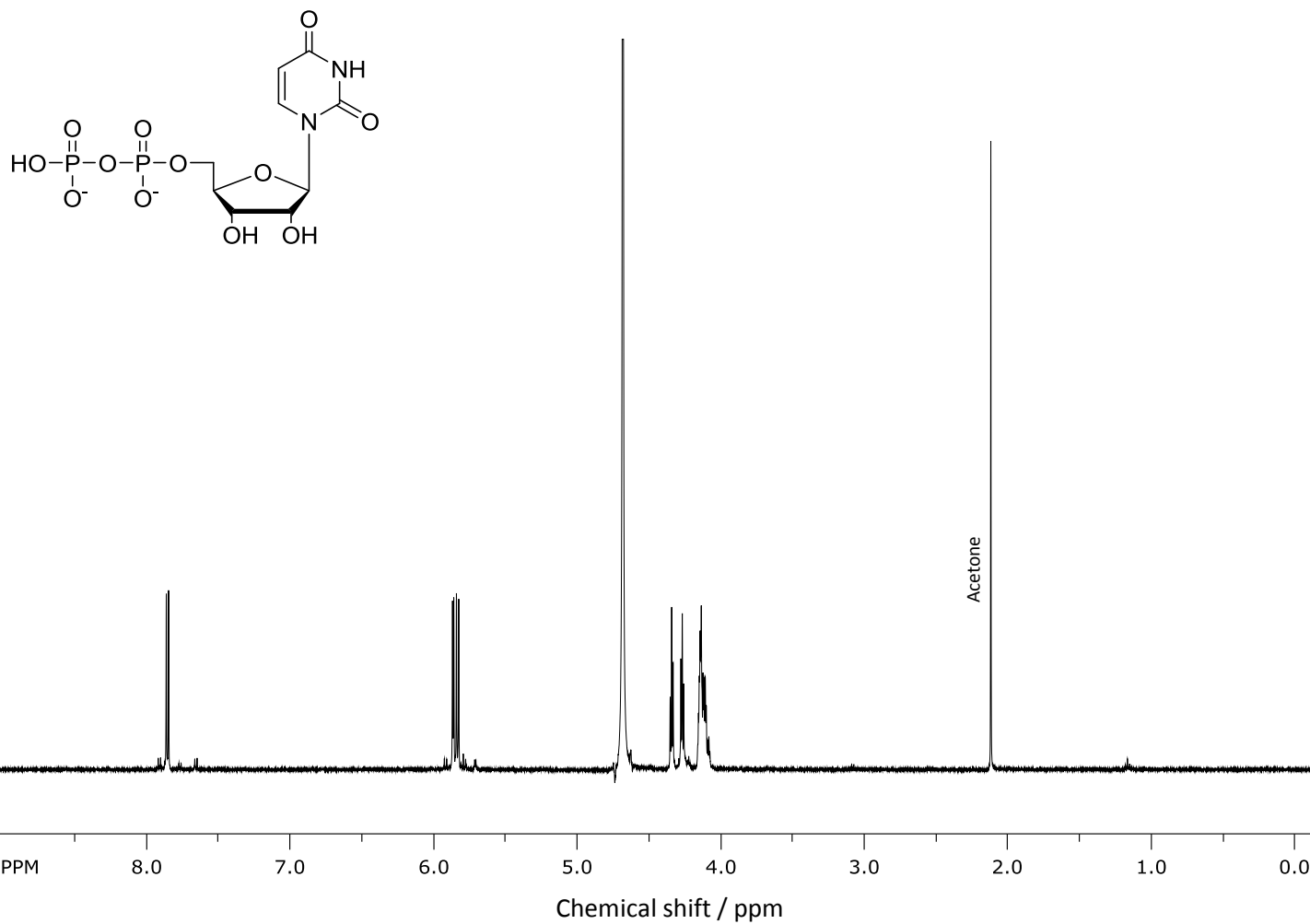
¹H NMR of thymidine 5'-diphosphate (2a)



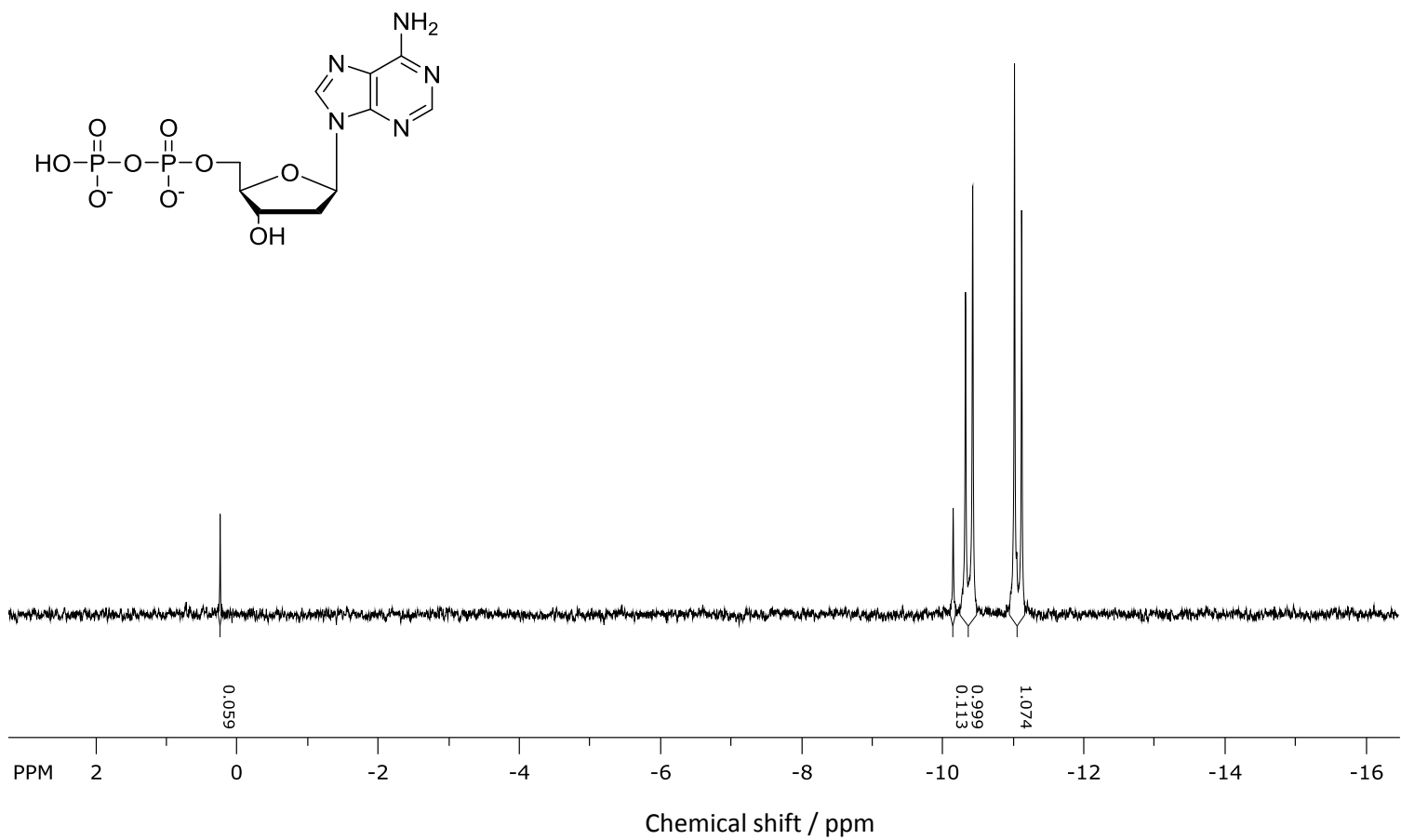
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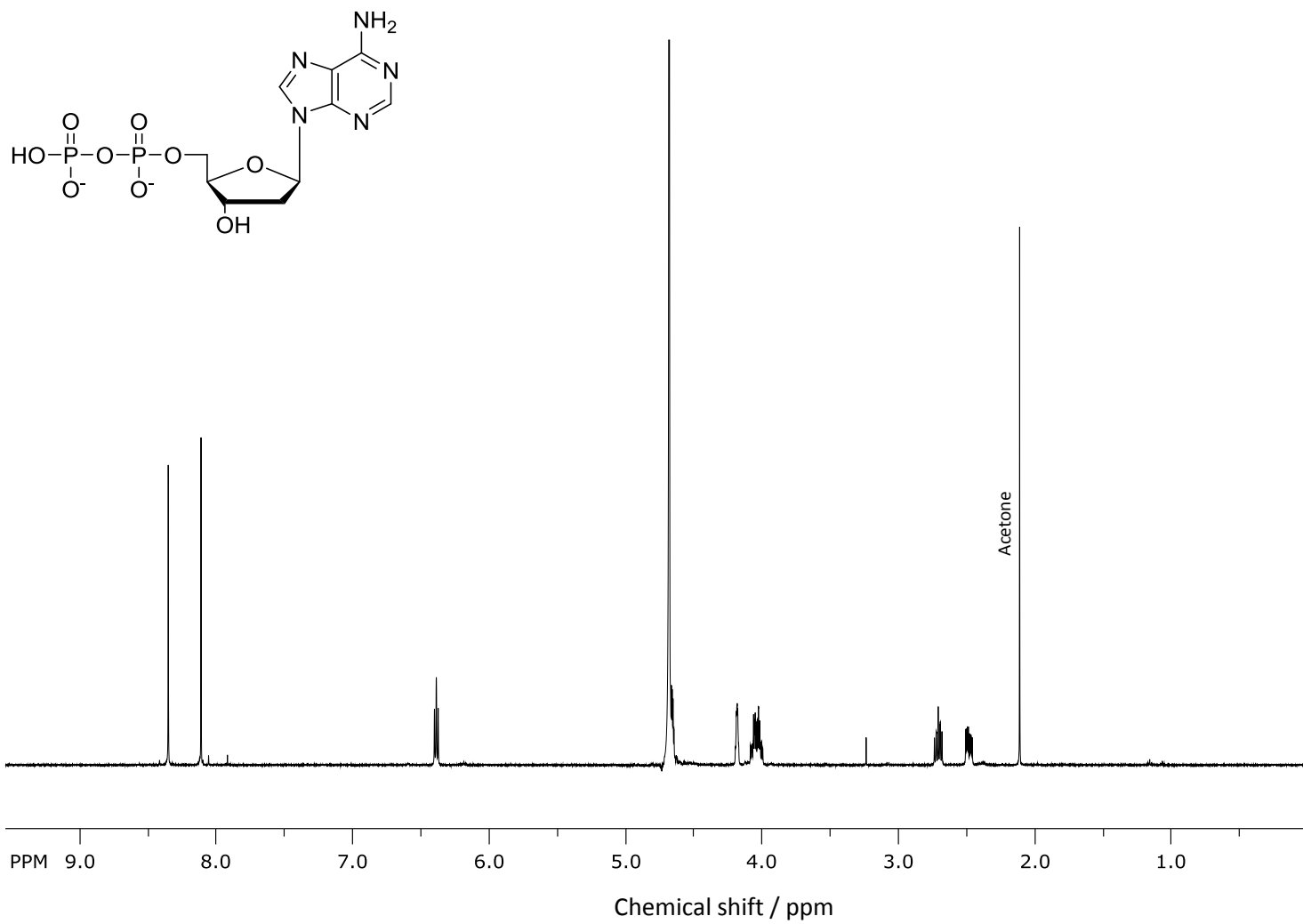
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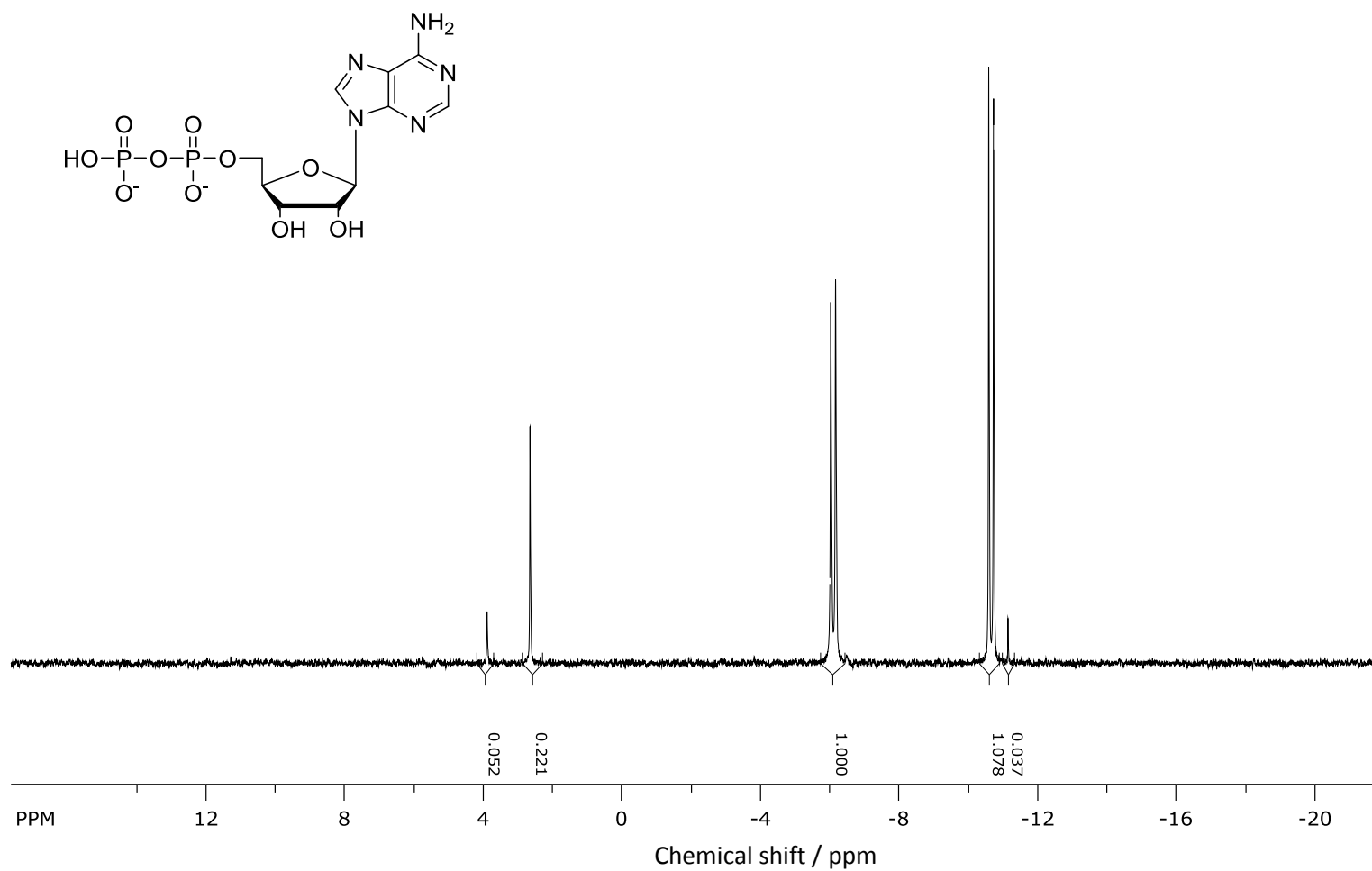
^{31}P NMR of 2'-deoxyadenosine 5'-diphosphate (2c)



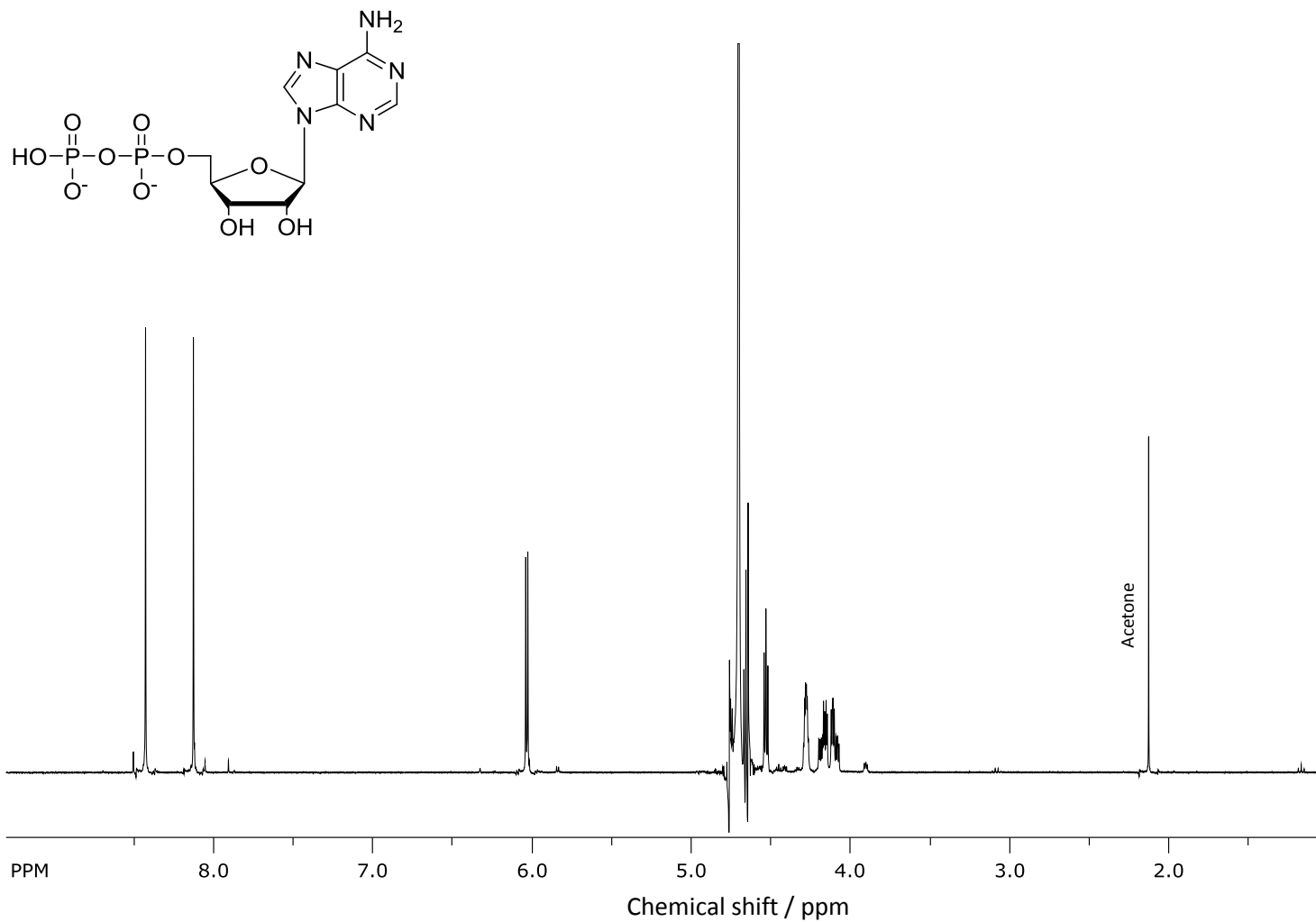
¹H NMR of 2'-deoxyadenosine 5'-diphosphate (2c)



³¹P NMR of adenosine 5'-diphosphate (2d)



¹H NMR of adenosine 5'-diphosphate (2d)



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