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## Nucleosides, Nucleotides and Nucleic Acids

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UNAMBIGUOUS ASSIGNMENT OF ANOMERIC CONFIGURATION OF  
NUCLEOSIDES BY NOE-DIFFERENCE SPECTROSCOPY

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Abstract.- An unambiguous assignment of anomeric configuration of D-ribo-, 2'-deoxy-D-ribo-, D-arabino- and 2',3'-dideoxy-D-ribonucleosides based on nuclear Overhauser enhancement spectroscopy is presented.

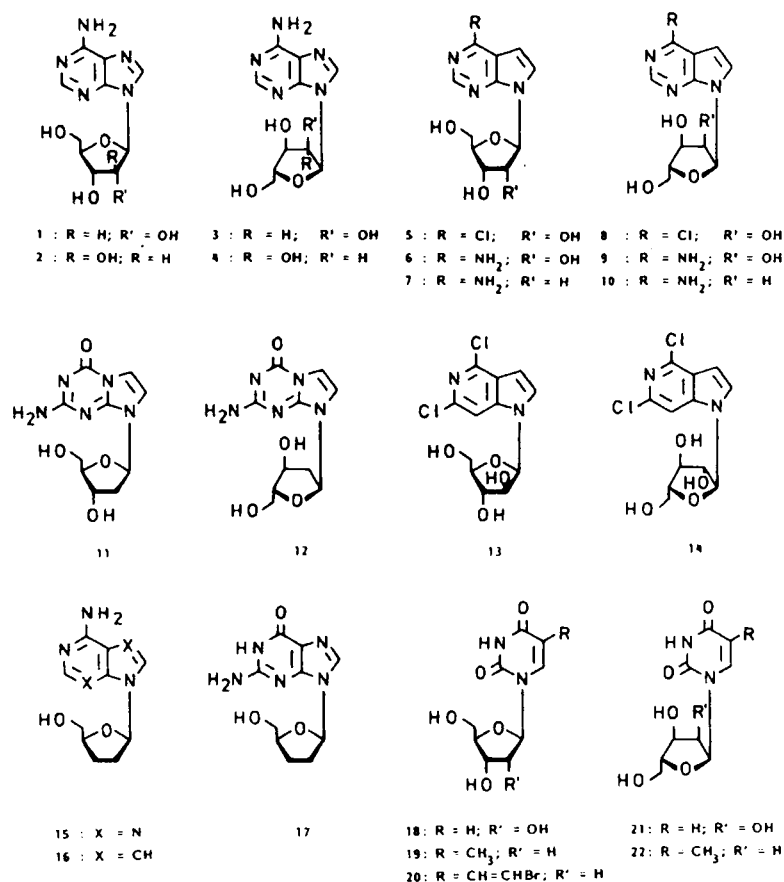
Assignment of anomeric configuration of regular and modified nucleosides is an important task in nucleoside chemistry. Apart from chemical and biochemical methods, CD-,  $^1\text{H}$ -, and  $^{13}\text{C}$ -NMR spectroscopy is utilised to solve this problem [1]. Empirical rules derived from comparative analysis of  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR chemical shifts or coupling constants of anomeric pairs do not always allow the elucidation of the anomeric configuration. This is caused by either electronic and anisotropy effects of the substituents at C-1' and/or the conformation of the glyconic moiety. Here, we present an unambiguous, reliable, and convenient method for the assignment of the configuration at C-1' employing  $^1\text{H}$ - $^1\text{H}$  nuclear Overhauser enhancement spectroscopy [2]. Irradiation of 1'-H and observation

Table. 1-D NOE Difference Data (%) upon Irradiation of 1'-H in DMSO- $d_6$ .

	1 $\beta$	3 $\alpha$	6 $\beta$	9 $\alpha$	5 $\beta$	8 $\alpha$	18 $\beta$	21 $\alpha$	2 $\beta$	4 $\alpha$	13 $\beta$
4'-H	2.1	0	2.4	0	2.1	0	1.7	0	2.0	0	2.5
3'-H	0	1.5	0	3.5	0	1.5	0	3.7	0	3.0	0
2'-H( $\beta$ )	1.9	10.9	2.1	11.3	2.1	10.3	2.2	10.0	-	-	-
2'-H( $\alpha$ )	-	-	-	-	-	-	-	-	9.9	4.1	12.1

	14 $\alpha$	7 $\beta$	10 $\alpha$	11 $\beta$	12 $\alpha$	19 $\beta$	22 $\alpha$	20 $\beta$	15 $\beta$	16 $\beta$	17 $\beta$
4'-H	0	2.0	0	1.5	0	2.3	0	2.3	1.8	2.0	2.6
3'-H	3.5	0	1	0	0	0	1	0	0	0	0
2'-H( $\beta$ )	-	0	5.6	0	5.0	0	6.3	0	0	0	0
2'-H( $\alpha$ )	2.3	5.6	0	5.1	0	6.3	0	5.9	6.6	7.8	6.4



of the n.O.e. of 4'-H yields enhancements only in case of  $\beta$ -nucleosides, whereas values of  $\alpha$ -nucleosides are zero (Table).

These findings are due to the close spatial proximities of the corresponding protons. As a control, in particular for the  $\alpha$ -nucleosides, the n.O.e.'s of 3'-H and 2'-H<sub>B</sub> are used as additional indicators. These are used randomly depending on the conformation of the sugar moiety. Simultaneously, n.O.e. measurements at 8-H give detailed information about the population of syn-anti conformers.

#### REFERENCE

- [1] Mizuno Y. 'The Organic Chemistry of Nucleic Acids' Elsevier, New York, 1986, pp. 70-72.
- [2] Knutsen L.J.S., Judkins B.D., Newton R.F., Scopes D.I.C., and Klinkert G. (1985), *J. Chem. Soc. Perkin Trans. I*, 621-630.

G.T. was on leave from Technical University Budapest.