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Short Communication

NMR STUDIES OF CLATHRATE DEUTER-ATES OF PIPERAZINE AND HEXAMETH-YLENETETRAMINE

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Extending our work on amine clathrates. The piperazine deuterate was prepared from partially deuterated piperazine in an inert atmosphere of nitrogen. HMT-deuterate was prepared by method given in [1].¹H absorption line measurement were done on varian DP-60 NMR spectrometer at 60 MHZ in a 5mm od tube filled, with fine powder of sample. Second moment were calculated as in [2].

Results and Discussions

Piperazine-deuterate spectra were recorded from 77 K to melting point of deuterate 317 K and HMT-deuterate spectra were recorded from 77K to its melting point 286 K. Strong saturation and poor signal-to-noise ratio were characteristic

* Present Address:- National Centre of Excellence in Analytical Chemistry, University of Sindh, Jamshoro. of both specimen deuterates at all temperatures. The experimental second moment of piperazine-deuterate under optimum condition was $16.3 + 2.0 \text{ G}^2$ ($0.163 + 0.020 \text{ mT}^2$, 1 milli Tesla mT = $10^3 \text{ T} = 10 \text{ Gauss G}$) at all temperatures. The theoretical value of rigid lattice second moment based on data of [3] is 15.78 G². This suggests that piperazine molecule is rigid in its water cage from 77 K to its melting point 317K.

The experimental second moment at 77 K for HMTdeuterates was $13.4 + 2.0 \text{ G}^2$ and this value stayed nearly constant upto 281 K. The theoretical rigid lattice second moment based on data of Smith [4] and method given in [2] is 14.78 G². This results also suggests that HMT molecule is rigid in its water cavity in m the temperature range of study. H-bonding between amino group and water cage appears to be strong and cause of rigidity of guest molecules in water cages.

Key words: NMR, Piperazine, Deuterates.

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