[MS45-P06] Crystal structure of 2-ethyl-3-hydroxy-6-methylpyridinium N-acetyl-glutamate. Testing the accuracy of DFT calculations for the refinement of H-atoms

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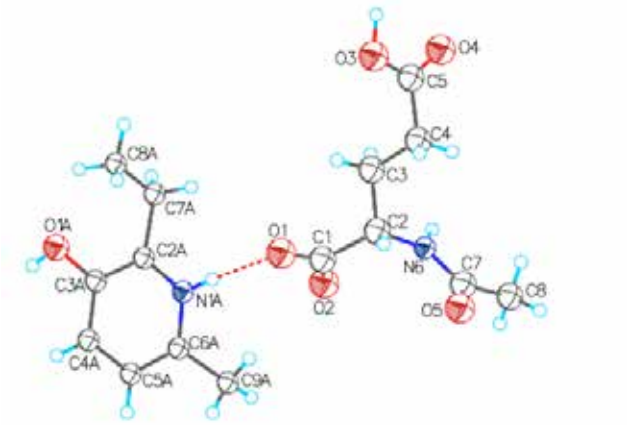
The crystal structure of 2-ethyl-3-hydroxy-6-methylpyridinium N-acetyl-glutamate (Figure) was studied by using a combined approach utilized powder X-ray diffraction (PXRD), periodic DFT calculation and solid solid-state NMR spectroscopy (SS-NMR). The positions of non-hydrogen atoms were revealed and verified by means of simulated annealing technique with subsequent refinement of PXRD data using the previously proposed Morse restraints [1]. At the same time the exact positions of hydrogen atoms remained unknown. The most intriguing problem of refinement was the identification of the position of the H atom between the carboxylic group and the pyridine moiety. This atom can be covalently bounded to N1A or O1A atoms. Accurate positions of the most of H-atoms were revealed from the results of the quantum chemical calculations with plane wave basis set. To reveal the positions of the H-atom of the above mentioned strong N-H…O bond we compared the ^{13}C and ^{15}N chemical shifts calculated by GI-PAW method with information from the experimental SS-NMR study. In fact, ^{13}C chemical shifts are almost not sensitive to the crystal packing while the ^{15}N chemical shifts are highly dependent on N…H and N…O interatomic distances. In the present report we discussed the details of the structural refinement and quantum chemical calculations to derive the most efficient methodology of combined PXRD and SS-NMR studies.

Figure. Cation and anion in crystal structure of 2-ethyl-3-hydroxy-6-methylpyridinium N-acetyl-glutamate.



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[1] I. S. Bushmarinov, A. O. Dmitrienko, A. A. Korlyukov, M. Yu. Antipin, J. Appl. Cryst. 2012, 45, 1187–1197.

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