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The aim of the study of optical resolution processes is to achieve a more efficient and less time consuming way of enantiomer separation. Supercritical fluid extraction as an alternative green technology for optical isomer separation was applied for the resolution of ibuprofen. Ibuprofen is widely used as non-steroidal anti-inflammatory drug. The resolution of ibuprofen (1) with the mixture of (+)-(R)-phenylethylamine (2) and benzylamine (3) was compared to the results obtained without the achiral additive [1]. Single crystal X-ray studies were performed on the salts of (+)-(R)-phenylethylamine crystallized with (-)-(R)-ibuprofen (4) and (+)-(S)-ibuprofen (5) as well as on the co-crystal resulted from the crystallisation of the racemic ibuprofen with the achiral benzylamine (6) in order to study the influence of the structurally similar achiral additive. (+)-(R)-phenylethylamine crystallises with (-)-(R)ibuprofen and (+)-(S)-ibuprofen, respectively with 1:1 stoichiometry. Unexpectedly, the crystal of the benzylamine ibuprofen salt contains an additional neutral ibuprofen molecule thus having 1:2 stoichiometry obtaining both from solution crystallisation and by mass balance calculation of the supercritical fluid CO₂ extraction. The lack of the methyl moiety in the benzylamine in structure (6) compared to the (+)-(R)-phenylethylamine molecule in structures (4) and (5) results a sterically favourable closer packing, higher crystal density. There is enough space in crystal (6) close to the ladder like hydrogen bond column to bond a neutral ibuprofen molecule as end group in the low acidity medium.

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Keywords: co-crystal, crystal packing, chiral separation

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p-Toluidinium nitrate. <u>Houda Boussekine</u>, Nourredine Benali-Cherif, *Laboratoire des Structures, Propriétés et Interactions Inter Atomiques (LASPI²A). Centre Universitaire de Khenchela, Algérie.* E-Mail: <u>houda.bouss@hotmail.fr</u>

P-toluidine is an organic benzene derivative with a methyl substituent and an amino group, the name is derived from toluene and aniline. P-toluidine can cause anoxia due to formation of methemoglobin and hematuria in man [1].

The crystal structure of *p*-methylanilinium nitrate was determined as part of our investigations on the structural characteristics of organic-inorganic layered compounds and an ongoing study on D—H···A hydrogen-bonding in systems of hybrid materials including anilinium derivatives such as, 3-hydroxyanilinium hydrogensulfate [2], *o*-methylanilinium nitrate [3], 2-carboxyanilinium dihydrogenphosphite [4] and 2-carboxyanilinium nitrate [5].

The asymmetric unit of the title compound, *p*-toluidinium nitrate or *p*-methylanilinium nitrate, $C_7H_{10}N^+$. NO₃⁻ contains a monoprotonated p-methylanilinium cation and nitrated anion. The crystal of the title compound, $C_7H_{10}N^+$. NO₃⁻ consists of anionic- cationic layers linked by a complex three-dimensional hydrogen-bond network. The structure is stabilized by anion-cation N—H···O hydrogen-bonding.

The structure of $(C_7H_{10}N^+$. NO₃⁻) is composed of cationic $(C_7H_{10}N^+$.) and anionic (NO_3^-) linked through N-H...O hydrogen bonds and building up a corrugated layers parallel to the (001) plane

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FA4-MS26-P09

Hydrogen bonding in Anilinium hydrogensulfate. Zina Boutobba & Nourredine Benali-Cherif,

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The main purpose of this structural study was a determination of the arrangement of the cations and anions which are held together by two-dimensional hydrogen-bond networks.

Hydrogen bonding is one of the most versatile noncovalent forces in supramolecular chemistry and crystal engineering [1]. Therefore, in the past decades assessment of discrete hydrogen bonding patterns had received great attention [2] because of its widespread occurrence in biological systems.

The aim of this paper is to discuss hydrogen patterns assuring the connection between anilinium and hydrogensulfate entities and to establish their different graph-set motifs [3].

Bis (anilinium hydrogensulfate) is one of the hybrid compounds, rich in H-bonds [4-5], which could have potential importance in constructing sophisticated assemblies from discrete ionic or molecular building blocks due to the strength and the directionality of hydrogen bonds [6].

Recently, similar structures containing anilinium cations have been reported. Among examples, can be named the fo-lowing ones: anilinium nitrate [7], , anilinium hydrogenphosphite and anilinium hydrogenoxalate [8].

The structure of may be described as formed by alternating sheets of cations and anions which are held together with four five centered N-H...O bonds to form $C_4^4(10)$ infinite chains running through the c direction. Moreover, strong O-H...O hydrogen bonds observed between bisulfate anions generate

 $C_2^2(8)$ chains in the *a* axis direction. The infinite chains resulting from anion-anion and anion-cation interactions can be described as zigzag layers parallel (ac) plans. The crossing

of these chains builds up different rings with $R_3^4(10)$ and $R_5^4(16)$ graph set motifs [9].

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