Poster Presentation

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Powder Diffraction study of different forms of Thiocolchicoside

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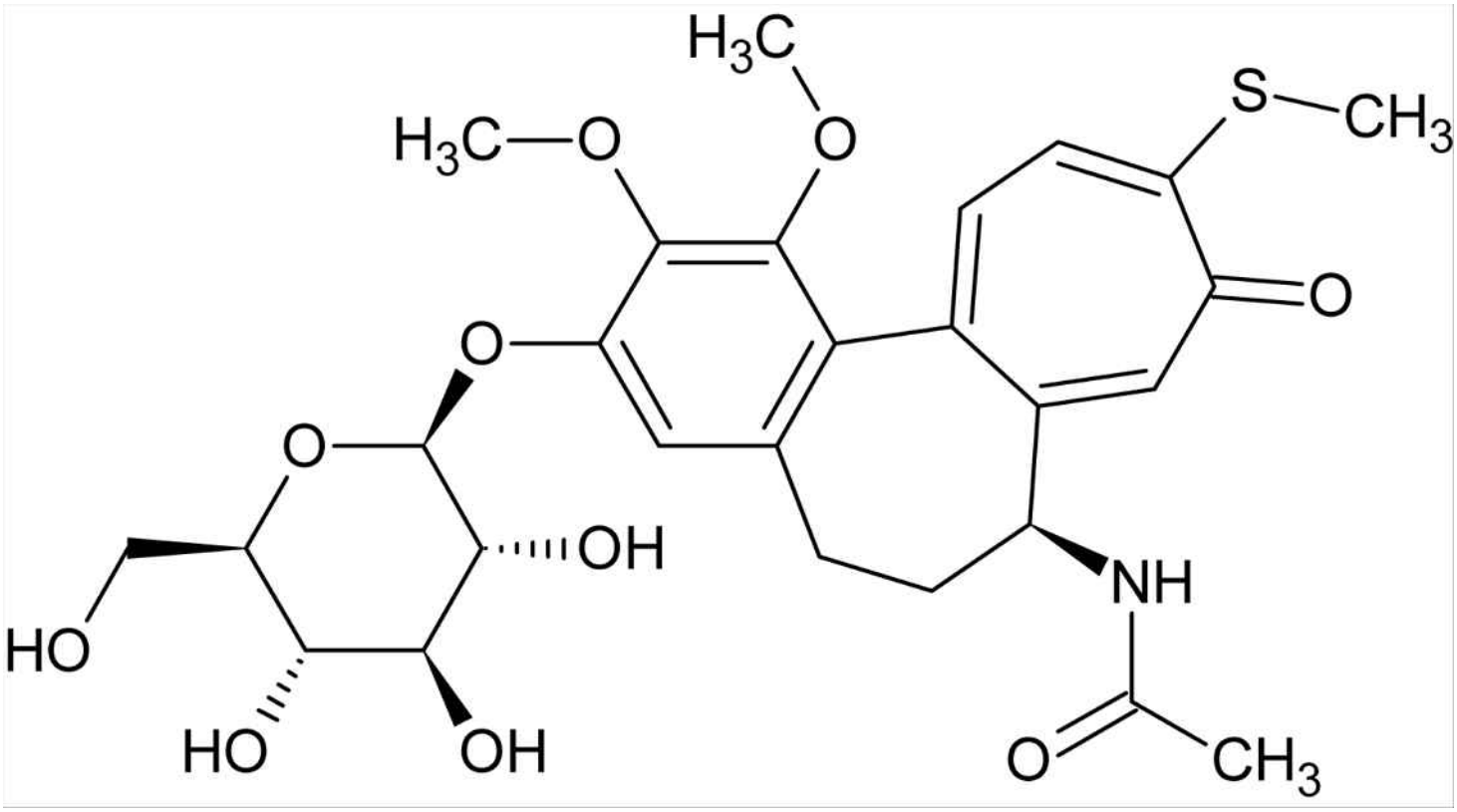
It is well-known that the stability, solubility, bioavailability and effectiveness of a pharmaceutical product are affected by the composition (solvates, hydrates, etc.) and the possibility of polymorphism of the Active Pharmaceutical Ingredient (API). In the present study, three different phases of thiocolchicoside, an API used as muscle relaxant, were obtained. The 47 maxima of the X-ray powder diffraction pattern of the raw material were indexed in an orthorhombic unit cell, with a=28.018(7) Å, b=12.519(2) Å, c=8.519(1) Å, V=2988.01 Å3 (M20=56.5; F20=137.6(0.0035,42)) The yellow crystals, obtained after recrystallization of the raw material in water at 37 °C, produced a pattern with 63 maxima. This pattern was indexed in another orthorhombic unit cell: a=25.264(4) Å, b=13.537(3) Å, c=8.553(1) Å, V=2925.12 Å3 (M20=47.3; F20=131.2 (0.0040, 38)). A third phase was obtained upon heating the yellow crystals. The 51 peaks of its powder pattern are consistent with a monoclinic unit cell with a=17.090(5) Å, b=19.485(5) Å, c=8.526(3) Å, β=100.30(2)°, V=2793.34 Å3 (M20=36.5; F30=56.3 (0.0067, 79)). All the patterns were indexed with DicVOL06 and analyzed with NBS*AIDS83. TGA/DSC analysis showed that the raw material is anhydrous, the yellow crystals are dihydrated, and the third phase is anhydrous. It must be noted that, to our knowledge, these phases have not been reported before. The only entry for a thiocolchicoside-related phase, present in the Powder Diffraction File, corresponds to an ethanol solvate hydrate (PDF 02-073-3591), which is also present in the Cambridge Structural Database (REFCODE: THCLCS). The structure of the hydrated phase was determined, from the powder diffraction data, with TALP[1]. It is worth noting the complexity of the thiocolchicoside molecule, the large number of torsion angles that need to be defined and its potentiality for forming hydrogen bonds (see figure 1).

[1] *O. Vallcorba, J. Rius, C. Frontera, et al., J. Appl. Cryst., 2012, 45, 1270-1277*



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