Preparation, polymorphic study and structural resolution by X-ray diffraction of polycrystalline samples of *(E)-N*-benzylidene-4-haloanilines

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The *(E)-N*-benzylidene-4-haloanilines belong to the Schiff base family, an important class of organic compounds which appears as an intermediate in several biological processes and during synthesis of organic compounds, due to the presence of azomethine group it finds extensive applications in analytical, coordination chemistry and biochemistry [1]. Some patents included versatile applications of this type of compounds, for example, in the protection of the skin against the harmful effects caused by sun exposure (Erythema), as well as the importance of mixtures of *N*-benzylideneanilines with aqueous acid solutions used as anticorrosives [2].The use of organic solvents translates into a significant environmental cost, which is why synthetic methods have been developed that do not involve solvents and involve other principles of green chemistry; the clean and efficient preparation of imines in aqueous suspension is one of them, in which acid catalysis is not necessary and provides relatively high yields when 1: 1 mixtures of aromatic aldehydes are reacted with primary aromatic amines [3].

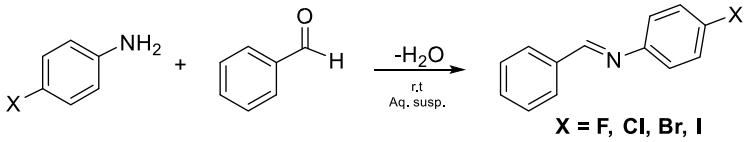

Figure 1. Formation of *N*-benzyliden-4-haloanilines without the use of organic solvents

In the X-ray powder diffraction crystallography literature, there are still no records for the resolution of the structure of the *(E)-N*benzylidene-4-fluor(chloride)aniline, although for the other two haloanilines there are available data obtained in a previous work made by a former undergraduate student. The resolution of the structures was made using a D8 ADVANCE with DaVinci geometry, these data, as well as the other different spectroscopic results will be exposed and discussed in the poster.

Parameter/X	**F**	**Cl**	**Br**	**I**
Crystal system, Space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1$	Orthorhombic, $Pna2_1$
***a* (Å)**	7.3758(3)	7.384(1)	8.8607(2)	7.989(4)
***b* (Å)**	26.8366(10)	26.859(4)	5.8639(2)	10.569(4)
***c* (Å)**	5.7146(4)	5.716(1)	21.939(2)	27.926(3)
α (°)	90	90	90	90
β (°)	90.638(5)	90.565	91.203(2)	90
γ (°)	90	90	90	90
volume (Å³)	1131.08	1133.6(3)	1139.71(5)	2358.2(6)
Z	4	4	4	8

Table 1. Crystallographic powder data for *(E)-N*-benzylidene-4-haloanilines

[1] P. Suna, P. K. Misra, y S. Panigrahi, «Substituted benzylideneanilines: A family of solvatochromic probes», *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, vol. 213, pp. 398-409, abr. 2019.

[2] G. Kaur, P. Panini, D. Chopra, y A. Roy Choudhury, «Structural Investigation of Weak Intermolecular Interactions in Fluorine Substituted Isomeric N -Benzylideneanilines», *Cryst. Growth Des.*, vol. 12, n.º 10, pp. 5096-5110, oct. 2012.

[3] K. Tanaka y R. Shiraishi, «Clean and efficient condensation reactions of aldehydes and amines in a water suspension medium», *Green Chem.*, vol. 2, n.º 6, pp. 272-273, 2000.

Keywords: bencylideneanilines, polymorphic study, crystal structure, green chemistry.

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