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GREEN SYNTHESIS OF A NEW CHIRAL IMINE WITH P-TOLUALDEHYDE AND ITS PALLADIUM (II) COMPLEX.

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Abstract: In this investigation, the synthesis of a new chiral imine and its palladium (II) complex was carried out. The reaction was carried out with p-tolualdehyde and (S)-(-)-1-phenylethylamine, in the absence of solvents, which is one of the principles proposed by Green Chemistry called Solvent-Free, to reduce the amount of toxic compounds that are generated in traditional reactions.
Imines have a great application in chemistry as they have a pair of valence electrons without sharing, they are also known as Schiff bases and they have the facility to coordinate with metals in this case Palladium. Compounds with biological activity have been generated. (Khan and Yusuf, 2009). The products were characterized by 1H and 13C nuclear magnetic resonance spectroscopy, Fourier transform Infrared spectroscopy. mass spectrometry, polarimetry. In both compounds the structures were obtained by X-ray diffraction.
Keywords: Green Chemistry, chiral, complex.

INTRODUCTION

Chemistry has an important role in the investigation of new compounds useful to society and must have the necessary conditions to achieve sustainable development, achieve new approaches to solve problems caused to the environment and avoid causing new problems (Eilks and Rauch, 2012). In the future, these issues will be a condition for future generations, within sectors such as the industrial, governmental and social sectors, becoming increasingly aware. (Anastas and Lankey, 2000). The simple fact of considering the impact that a new substance that is obtained through Green Chemistry implies, either at an environmental or human level; it is a key difference from conventional chemistry. (Warner et al., 2004).

Organic synthesis must carry out its transformations anticipating the consequences that this may cause to the environment. have the objective of minimizing pollution from its origin avoiding the formation of polluting waste, this is called Green Chemistry or chemistry that deals with the design of chemical products and processes that reduce or eliminate the use and generation of harmful substances for people and the environment. One of the most active areas has been to use benign solvents, these are used in large quantities in the chemical industry, many of them are volatile, dangerous, polluting, flammable, toxic or carcinogenic. (Anastas and Kirchhoff, 2002).

The means used by Green Chemistry focus on the reduction or elimination of the use of toxic chemical products and the recycling of waste produced by technological progress, in a creative way in such a way that a minimum impact on human beings is achieved. and the environment, without sacrificing scientific and technological progress. (Clarke et al., 2018)

The "traditional" solvents used in the reactions are very toxic and to continue carrying out synthesis in chemistry, solvents that are harmless to living beings must be used. (Poliakoff et al., 2002)

To provide a solution, the development of a compound synthesis technique known as "Solvent-Free" (All, 1995). This technique drastically reduces the production of effluent waste and air pollution; In addition to this methodology, it also has other advantages such as greater reactivity, a maximum concentration of reagents, and greater productivity due to the smaller amount of material in the same volume of the reactor. Also, washing and extraction processes are simplified, or possibly avoided. (Tanaka and Toda, 2000).

Imines, also known as Schiff bases, are biologically active compounds and may exhibit propertiesbactericides, fungicides, herbicides,analgesic, anti-inflammatory,anti-

tuberculosis anticancer. Its applications range fromsupramolecular chemistry, catalysis in encapsulation processes until the formation of compounds with unusual properties. (Vanco et al., 2008).

DEVELOPMENT

A new chiral imine was synthesized, starting from p-tolualdehyde and (S)-(-)-1-phenylethylamine, in a 1:1 molar ratio in the absence of solvent using the dry medium technique (Figure 1).

IR spectra were recorded on the Perkin Elmer Spectrum One FT-IR spectometer Universal ATR. The 1H NMR and 13C NMR spectra were performed on the Bruker-500 equipment (500 MHz); chemical shifts are expressed in ppm towards low fields taking tetramethylsilane (TMS) (δ=00) as reference. The mass spectra were performed using the electronic impact (IE) technique, they were recorded with a JEOL JMS-SX 102a spectrometer operated in the positive ion mode at 70 eV, the data are expressed in mass/charge (m/z) units. Optical rotation was measured on a Perkin-Elmer 241 polarimeter. Melting points were determined on the Electrothermal MEL-TEMP 3.0 apparatus.

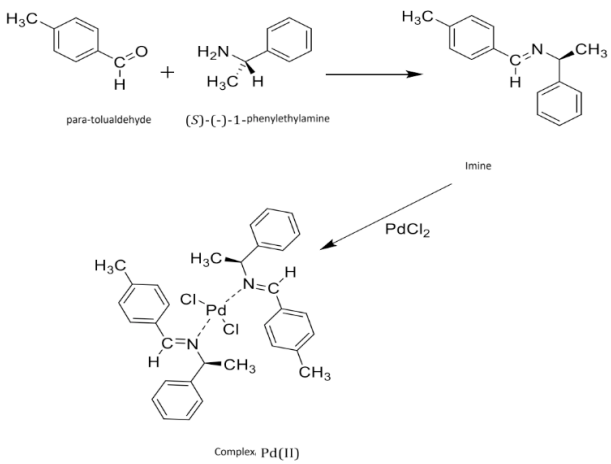

Figure 1.- Synthesis of the imine and its Palladium(II) complex.

RESULTS AND DISCUSSIONS

The reaction of p-tolualdehyde and (S)-(-)-1-phenylethylamine. in equimolar proportions it gave rise to the formation of the corresponding dextrorotatory imine.

IMINE SYNTHESIS (S)-(+)-1-[(PHENYL)-N-(4-METHYLPHENYL) METHYLIDENE] ETHYLAMINE.

The synthesis of the imine was carried out by reacting p-tolualdehyde (188.0 mg, 1.571 mmol) and (S)-(-)-1-phenylethylamine (190.4 mg, 1.571 mmol) using the technique in a dry medium, obtaining a solid white with a melting point of 92-94°C, with a yield of 92%, a = + 99.7 (c = 1, CHCl3).$[\alpha]_D^{25^\circ}$

FT-IR υmax: 1634 cm-1 (C=N), 1H NMR (CDCl3/TMS): δ = 1.585 (d. 3H; CH3), 2.37 (s. 3H; OCH3), 4.52 (c, 1H, CHCH3), 7.24-7.19 (m, 3H; H-Ar;), 7.35-7.32 (m, 2H; H-Ar;), 7.43-7.42 (m, 2H; H-Ar;) 7.68-7.66 (m, 2H ;H-Ar;) 8.33 (s, 1H; HC=N).

^{13}C NMR (CDCl3/TMS): δ = 21.54 (CH3), 24.86 (OCH3), 69.70 (CHCH3), 126.66, 126.80, 128.26, 128.42, 129.28, 133.81, 140.85, 145.33 (C-Ar), 159. 47 (HC= N).

The mass spectrum allows us to observe the molecular ion of the IE compound (m/z): 253 M•+ and confirms the proposed molecular formula: C21H18FN. The structure of this new compound was confirmed by single crystal X-ray diffraction (Figure 2).

PALLADIUM COMPLEX SYNTHESIS.

The synthesis of the palladium complex was carried out with a 1:2 molar ratio with the PdCl2 salt (63.0 mg, 0.166 mmol) and twice the equivalents of the imine obtained (101.0 mg, 0.30 mmol) (Figure 3).

CONCLUSIONS

Using Green Chemistry, the new chiral imine has been synthesized, obtaining

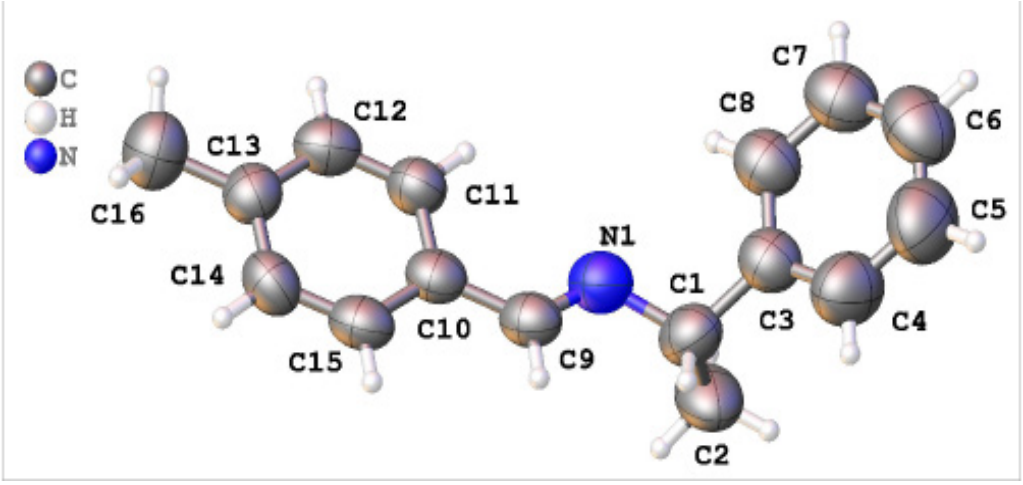

Figure 2.- X-ray structure of the imine.

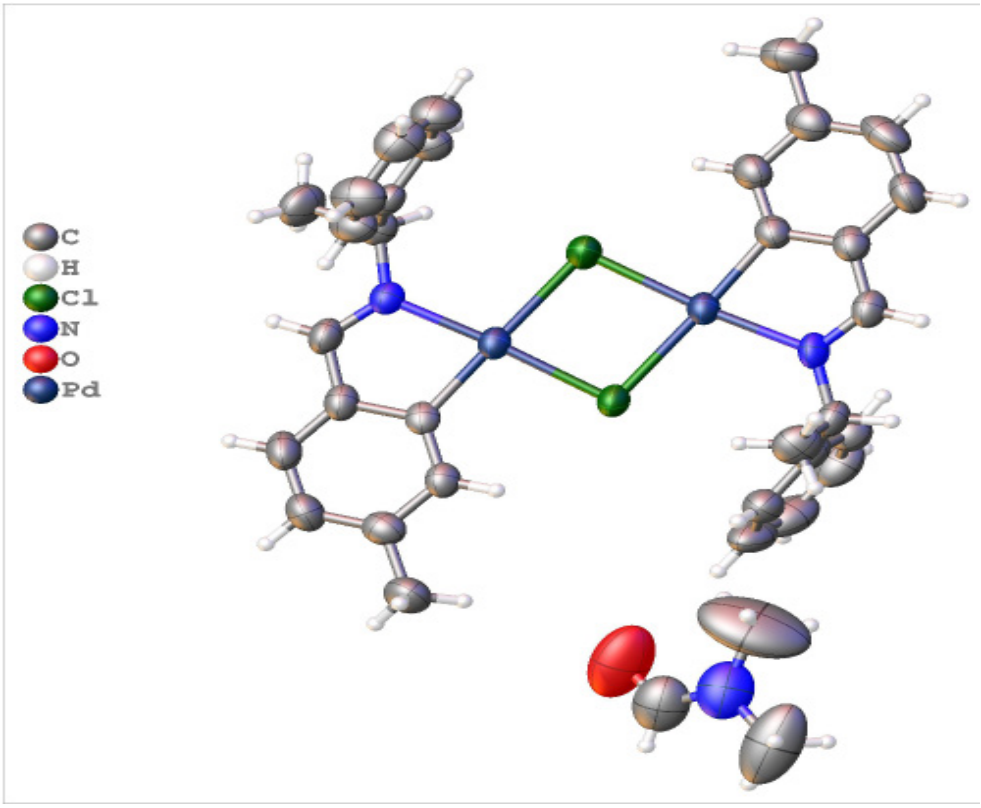

Figure 3. X-ray structure of the palladium(II) complex.

excellent performance, improving the image of traditional Chemistry, obtaining products that are friendly to the environment and human health. From the imine, a monocrystal with palladium chloride was obtained, to later study its behavior in catalytic and pharmacological studies.

The products were characterized using spectroscopic techniques: IR, 1H and 13C Nuclear Magnetic Resonance and the structures of the synthesized imines were confirmed by X-ray diffraction.

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