Synthesis and Characterization of a N, N'- Bissalicylidene- 1, 2-Phenylenediamine (I)( Salophene) Galodinium

Shahriar Ghammamy and Sajjad Sedaghat

Department of Chemistry, Faculty of Science, Imam Khomeini International University, Qazvin, Iran
Department of Chemistry, Faculty of Science, Islamic Azad University, Malard Branch, Malard, Iran

Abstract: N, N'- bissalicylidene- 1, 2-phenylenediamine (I) (salophene) abbreviated as SPDAS was synthesized and characterized. N, N'- bissalicylidene- 1, 2-phenylenediamine (I) ( salophene) galodinium prepared by reaction of nitrate salt of Gd(NO$_3$)$_3$·6H$_2$O with SPDAS. In this research, some of the inorganic complexes of galodinium with N- donor ligands were synthesized. These compounds were characterized by FT-IR and UV/Visible techniques. The electronic and vibrational spectra of SPDAS and N, N'- bissalicylidene- 1, 2-phenylenediamine (I) (salophene) galodinium have been measured and studied. Analytical methods have been applied to the investigation of the structure of the compounds SPDAS and N, N'- bissalicylidene- 1, 2-phenylenediamine (I) (salophene) galodinium.

Key words: Synthesis • Characterization, Spdas • N, N'- BisSalicylidene- 1 • 2-Phenylenediamine (I) (Salophene) Galodinium • Ft-Ir • Uv/Visible Techniques

INTRODUCTION

Gadolinium is a chemical element with the symbol Gd and atomic number 64. It is a silvery-white, malleable and ductile rare-earth metal. It is named for gadolinite, one of the minerals in which it was found, in turn named for chemist Johan Gadolin. Gadolinium as a metal or salt has exceptionally high absorption of neutrons and therefore is used for shielding in neutron radiography and in nuclear reactors. The Gd(III) ion occurring in water-soluble salts is quite toxic to mammals. However, chelated Gd (III) compounds are far less toxic because they carry Gd (III) through the kidneys and out of the body before the free ion can be released into tissue. Because of its paramagnetic properties, solutions of chelated organic gadolinium complexes are used as intravenously administered gadolinium-based MRI contrast agents in medical magnetic resonance imaging. However, in a small minority of patients with renal failure, at least four such agents have been associated with development of the rare nodular inflammatory disease nephrogenic systemic fibrosis. This is thought to be due to gadolinium ion itself, since Gd(III) carrier molecules associated with the disease differ. It crystallizes in hexagonal, close-packed $\alpha$- form at room temperature, but, when heated to temperatures above 1235°C, it transforms into its $\beta$- form, which has a body-centered cubic structure. Gadolinium has no known native biological role, but its compounds are used as research tools in biomedicine. It is used in various ion channel electrophysiology experiments to block sodium leak channels and stretch activated ion channels. A Schiff base, named after Hugo Schiff, is a compound with a functional group that contains a carbon-nitrogendouble bond with the nitrogen atom connected to an arylalkyl group, not hydrogen. Schiff bases in a broad sense have the general formula $R'R'\text{C}==\text{NR}'$, where $R$ is an organic side chain. In this definition, Schiff base is synonymous with azomethine. Some restrict the term to the secondaryaldimines (azomethines where the carbon is connected to a hydrogen atom), thus with the general formula $\text{RCH}=\text{NR}'$. The chain on the nitrogen makes the Schiff base a stableimine [1-8]. A Schiff base derived from aniline, where $R'$ is a phenyl or a substituted phenyl, can be called an anil. Schiff bases can be synthesized from

Corresponding Author: Shahriar Ghammamy, Department of Chemistry, Faculty of Science, Imam Khomeini International University, Qazvin, Iran. Fax: +98-281-3780040.
Experimental

Material and Instruments: Acetonitrile (Fluka, P.A.) was distilled several times from phosphorus pentoxide before use, thereby reducing its water content to <4 ppm. N, N'-bissalicylidene-1, 2-phenylenediamine (I) (salophene) was bought from Merck. Gd(NO$_3$)$_3$.6H$_2$O (Merck, p.a.) was used without further purification. Solvents were purified by standard methods. Infrared spectra were recorded as KBr disks on a Shimadzu model 420 spectrophotometer. Galodinium was estimated iodometrically. The percent compositions of elements were obtained from the Microanalytical Laboratories, Department of Chemistry, OIRC, Tehran.

Synthesis Of N, N'-Bissalicylidene-1,2-phenylenediamine (I) (Salophene), SPDAS: For synthesis of the SPDAS to a magnetically stirred of 1, 2-phenylenediamine (1.26g, 1mmol) in ethanol(25ml) was added to salicyladdehyde (2.318g, 2mmol) at room temperature. The compound was refluxed for 3 hours to ensure the completion and precipitation of the formed complex. The precipitated solid complex was filtered and washed several times with hexane and ether to remove any traces of the unreacted starting materials. Anal. Caled of (C$_{32}$H$_{20}$N$_2$O$_3$)Gd; C; 51.09, H; 2.76, N; 5.95; found: C; 51.16, H; 2.84, N; 5.99. Mp: 197°C. FT-IR (KBr, cm$^{-1}$): 3432.22 (v OH of phenol), 1623.88 (v C-O of phenol), 115.03 (v C=C of Ar ring), 1009.14 (v C=N of immine), 1479.50 (v C=C of Ar ring), 1614.36 (v C=N of imine), 1479.50 (v C=C of Ar ring), 1275.70 (v C-O of phenol), UV/Vis:266(1416.67) [ε, M$^{-1}$ cm$^{-1}$], 316.68(1291.67) [ε, M$^{-1}$ cm$^{-1}$], 320(1417) [ε, M$^{-1}$ cm$^{-1}$], 228(1367) [ε, M$^{-1}$ cm$^{-1}$], 117.79 (v C=C of Ar ring), 1111 cm$^{-1}$; 3444.70 (v C-O of phenol), 1111 cm$^{-1}$.

RESULTS AND DISCUSSION

The chemistry of Schiff bases is a field that is being noticed. Schiff bases are potentially capable of forming stable complexes with metal ions. Schiff bases derived from the reaction of aromatic aldehydes and aliphatic or aromatic amines represent an important series of widely-studied organic ligands. Schiff bases form a significant class of compounds in medicinal and pharmaceutical chemistry with several biological applications that include antibacterial, antifungal and antitumor activity. The ligand and complex are stable at room temperature.

The Advantages of the New method Are:

- There are no sides products,
- The reaction are quite fast, mild conditions and
- The accompanied color change that providing visual means for ascertaining the progress of the reaction.

Preparation of Ligand and Complex: In this paper, we report a new method of the synthesis of N, N'-bissalicylidene-1, 2-phenylenediamine (I) (salophene) and N, N'-bissalicylidene-1, 2-phenylenediamine (I) (salophene) galodinium. The reaction between 1,
Fig. 1: FTIR spectrum of SPDAS (KBr Disk)

Fig. 2: FTIR spectrum of Gd (C$_{6}$H$_{11}$N$_{2}$O$_{2}$) (KBr Disk)

Fig. 3: UV/Vis spectrum of SPDAS
Fig. 4: Chemical structure of SPDAS

2-phenylenediamine, salicylaldehyd, SPDAS and Gd(NO₃)₃.6H₂O produced two new galodinium compounds was synthesized through a one-step reaction. Our procedure for producing compound has some advantages. For example, there is no side product in preparing SPDAS and Gadolinium diethylenetriaminepentaacetic acid hyaluran conjugates: Preparation, properties and applications. Macromolecular Symposia, 186: 105-110.


Gadolinium diethylenetriaminepentaacetic acid hyaluran conjugates: Preparation, properties and applications. Macromolecular Symposia, 186: 105-110.

Galodinium compound and was synthesized through a one-step reaction. Our procedure for producing compound has some advantages. For example, there is no side product in preparing SPDAS and Gadolinium diethylenetriaminepentaacetic acid hyaluran conjugates: Preparation, properties and applications. Macromolecular Symposia, 186: 105-110.

These compounds were characterized by FT-IR and UV/Visible techniques. The SPDAS and Gadolinium diethylenetriaminepentaacetic acid hyaluran conjugates: Preparation, properties and applications. Macromolecular Symposia, 186: 105-110.

The spectral data of the complexes have good relationship with the literature data. In the case of SPDAS we observed the following changes. The bands appeared around 3444.70, 1614.36, 1479.50 and 1275.70 cm⁻¹ due to OH of phenol, C=N of imine, C=C of Ar ring, C-O of phenol. In the case of Gadolinium diethylenetriaminepentaacetic acid hyaluran conjugates: Preparation, properties and applications. Macromolecular Symposia, 186: 105-110.


REFERENCES


