

Sakarya University Journal of Science SAUJS

ISSN 1301-4048 e-ISSN 2147-835X Period Bimonthly Founded 1997 Publisher Sakarya University http://www.saujs.sakarya.edu.tr/

Title: 2,4,6-Tris(p-aminoanilino)-1,3,5-triazine: Synthesis and Electron Paramagnetic Resonance (EPR) Analysis

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Recieved: 2022-06-24 00:00:00

Accepted: 2022-10-10 00:00:00

Article Type: Research Article

Volume: 26 Issue: 6 Month: December Year: 2022 Pages: 1170-1179

How to cite Özgül KARATAŞ, Yusuf CEYLAN, Ziya Erdem KOÇ ; (2022), 2,4,6-Tris(p-aminoanilino)-1,3,5-triazine: Synthesis and Electron Paramagnetic Resonance (EPR) Analysis. Sakarya University Journal of Science, 26(6), 1170-1179, DOI: 10.16984/saufenbilder.1135112 Access link https://dergipark.org.tr/en/pub/saufenbilder/issue/74051/1135112



Sakarya University Journal of Science 26(6), 1170-1179, 2022



2,4,6-Tris(p-aminoanilino)-1,3,5-triazine: Synthesis and Electron Paramagnetic Resonance (EPR) Analysis

Özgül KARATAŞ *1^(D), Yusuf CEYLAN²^(D), Ziya Erdem KOÇ ²^(D)

Abstract

A significant group of compounds arise from substituted s-triazine derivatives that have tripodal heterocyclic compound. Compounds classified as heterocyclic possible created the largest and most diverse family of organic compounds. In this study, we reported that a new template has been syntheses from a cyanuric chloride and its diamine derivative. The desired triamine a tripodal 2, 4, 6-tris (p-aminoaanilino)-1, 3, 5-triazine ($C_{21}H_{21}N_9$), called to be TRIPOD, has been obtained from cyanuric chloride with 3 eq of p-phenylenediamine reaction in acetone. After synthesis, TRIPOD sample which was polycrystal form was irradiated by cobalt-gamma source. The electron paramagnetic resonance spectra of TRIPOD were recorded using X-band EPR spectrometer at room temperature in three axes (x, y, z) which are perpendicular at 10° intervals. The EPR spectrum was simulated using computer program and by using the spectrum. The radical structure was determined in the sample.

Keywords: EPR, S-triazine, radical, synthesis, irradiation effects

1. INTRODUCTION

It is known that 1,3,5-Triazine (or striazines) derivatives attract mainly great attention because of their applications in different fields. These compounds have commonly used such explosives, as pesticides, dyestuffs, polymer photostabiliser, optical bleaches and surfaceactive agents [1-11]. Looking at the past studies, it is seen that this compounds group has been studied quite a lot [12-19]. The 1,3,5-triazine ring has been shown to be an appropriate structural element to be assembled into thermotropic liquid crystals.

The s-triazine derivatives can be simply prepared with cyanuric chloride (C₃N₃Cl₃), 2,4,6-(trichloro)-1,3,5-triazine (1) which is inexpensive and easily existing. [20-21]. Cyanuric chloride is a fantastic starting compound for preparation of a very well multitopic molecule. Any nucleophile reactant can substitute every 2,4,6-trichloro-1,3,5-triazine chloride atom (Figure 1.) [22]. When looking on the tripodal-s-triazine derivatives preparation, the first substitution step was exothermic, so, the mixture of reaction temperature was kept at 0°C. Then, at room temperature the second one step is

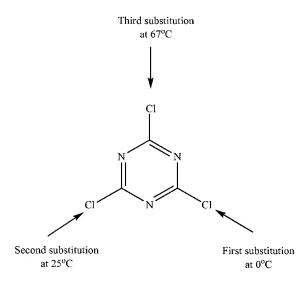
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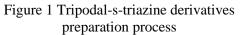
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chloride and the third step was operationalized under with refluxing solvent. the synthesis of 2,4,6-Consequently, trisubstituted-triazines was obtained with the temperature control during the substitution reactions and sequential addition of grignard reagents, amines, thiols, or alcohols. The performance of every substitution step generally exceeded 95%. Also, the symmetric trisubstituted derivatives could even be obtained in a one pot synthesis [23].

Irradiation has an important role to change of chemical and physical characteristics of materials. It is in preference for chemistry fields for different usage purposes such as sterilization or pathogen killing etc. [24-29]. The electron paramagnetic resonance (EPR) the most effective method is for determination of free radicals formed in various crystalline materials by gamma irradiated [30-34]. Furthermore, this method is widely used for providing information about the paramagnetic defect centers and detail description for structures [35-40].

To understand chemical reaction, it is necessary to investigate the electronic

structure of molecules. In this study, we aimed to synthesize of 2,4,6-tris(p-aminoaanilino)-1,3,5-triazine (C $_{21}H_{21}N_{9}$), TRIPOD, understanding its chemical reaction after gamma irradiation by using EPR method.

2. MATERIALS AND METHODS

The chemicals were used in the study. The 2,4,6-trichloro-1,3,5-triazine was used like a connection agent and provided commercially from Aldrich. The pure cyanuric chloride by recrystallizations was used for providing the pure petroleum ether ($60-90^{\circ}$ C) [41].

Measurements as follow: Carlo Erba 1106 elemental analyzer was used for elemental analyses; Perkin Elmer 1600 model FT-IR spectrophotometer was used for recording the IR spectra; Varian MAT 711 spectrometer was used for examination of metal contents; Bruker 200 MHz spectrometer was used for obtained the ¹H -NMR spectra in $\delta 6$ -DMSO; MMM-Medcenter and Einrichtungen GmbH Vacucell 22 were used as Vacuum Cabinets; Buchi SMP-20 melting point apparatus was used for measuring the melting points; Arex Velp Sci. as Heating Magnetic stirrer equipped with a contact Vetex thermostat connection was used for direct control of the temperature of the stirred liquid; Jeol JES-FA 300 X-Band ESR spectrometer was used for EPR measurements.

2.1 The 2,4,6-tris(*p*-aminoaanilino)-1,3,5triazine Synthesis

Firstly, acetone (75 mL) was used for disolving the cyanuric chloride (1) (1.84 g, 10 mmol). Then, NaHCO₃ (6.30 g, 75 mmol) in water (100 mL) which was saturated with N₂ was added and three necked round bottomed flask was cooled to 0°C. p-Phenylenediamine (3.24 g, 30 mmol) was

added gradually. When the addition was complete, the suspension mix was warmed to room temperature and then heated under reflux for 48 h. The residual was suspended in water (100 mL) and the acetone was removed by vacuum. The water phase was extracted three times with dichloromethane and the precipitate was removed by filtration. Powder solid product was washed with cold water (3x100 mL) to remove the sodium bicarbonate and collected by filtration. The CHN analyse results of the compound were similar the molecular formula C₂₁H₂₁N₉. (Elemental analysis was obtained as: C, 63.94; H, 5.34; N, 31.68 %). Calc.: C, 63.14; H, 5.30; N, 31.56. FT-IR (cm⁻¹) 3335–3290 (NH), 2835 (CH), 1589-1484 (triazine C=N). ¹H NMR (δ_6 -DMSO) 6.73 (δ,6H), 7.32 (δ, 6H).

2.2 The EPR Measurement

Polycrystalline form of sample was obtained as the TRIPOD was synthesized. This polycrystalline sample was irradiated for 72h at room temperature. For irradiation, cobaltgamma irradiator which has 0.985 kGy/h dose speed was used. After that, all EPR spectra of this sample were recorded in the magnetic fields for three perpendicular planes temperature. room The at spectrometer conditions were set as following: microwave power of 5mW, modulation amplitude of 0.4mT, center field of 325mT, modulation frequency of 100kHz.

3. RESULTS AND DISCUSSION

The results showed that, a new synthetic route was enhanced for versatile ligands prepared using 2,4,6-trichloro-1,3,5-triazine as core. The reaction contained the selective substitutions of p-phenylenediamine onto three chlorides of the triazine ring via a stepwise manner at 1:1, 1:2, or 1:3 eq. and 0,

25, 130°C, respectively. An effective synthesis of a new class of versatile molecules was enhanced with high efficiency chloride substitution steps of 2,4,6-trichloro-1,3,5-triazine by amines. The versatile molecules which was mono-new 1,3,5-triazine derived were synthesized and characterized by means of elemental analysis, ¹H NMR, FT-IR spectroscopy (Figure.2 and Figure.3).

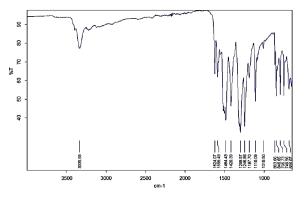


Figure2 FT-IR spectrum of 2,4,6-tris(paminoaanilino)-1,3,5-triazine

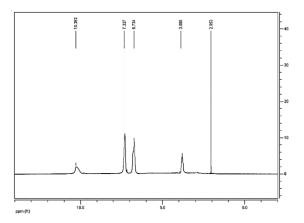


Figure 3 ¹H NMR spectrum of 2,4,6-tris(paminoaanilino)-1,3,5-triazine

These analyzes showed that replacement of the chloro by the amine group induced lowering of the energy of the NH₂ stretch in the FT-IR spectrum and a shift to higher field of the NH proton signal in the ¹H-NMR spectrum. The particularity of amine substitution over three chlorides of cyanuric chloride and the optimization of reaction temperatures were studied using *p*-phenylenediamine as the intial amine. The *p*-phenylenediamine has only a single $-NH_2$ functional group for substitution on the triazine ring. During the reaction, the different reaction temperatures and molar ratios of triazine to amine at 1:3 eq. were used for confirmition the particularity mode for the substitutions of three chlorides (Figure 4.).

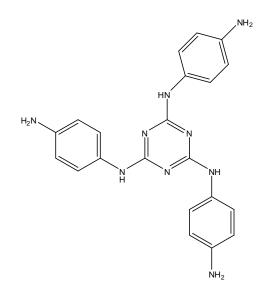


Figure 4 Reactions of cyanuric chloride with *p*-phenylenediamine

The ¹H-NMR spectra were obtained in DMSO- δ_6 . ¹H-NMR spectra were also correlated with the synthesized compounds structures. ¹H-NMR, FT-IR, of the product *p*-phenylenediamine and cyanuric chloride established that each a NH group of pphenylenediamine had reacted with triazine ring. This was confirmed by Fujiwara test [23,41]. This s-triazine compound was easily converted to the amine derivative and the yield of the conversion reaction was very high 82%. In the ¹H NMR spectra of compounds 2.4.6-tris(p-aminoaanilino)-1,3,5-triazine, the signals were detected at about 10.28 and 3.88 ppm, respectively. All signals appeared as broad singlets and were attributed to the N–H in the tripodal-s-triazine. The chemical shifts of the aromatic protons are in a range of 6.73 and 7.32 ppm respectively [42-46]. The presence of N–H was also identified by FT-IR spectroscopy as a sharp band at about $3325-3290 \text{ cm}^{-1}$. Also, the vibrations of the triazine C=N of compounds s-triazine ring were observed at $1589-1484 \text{ cm}^{-1}$ range, respectively [47].

According to the EPR measurements, there were not detected any EPR signal on nonirradiated TRIPOD polycrystalline sample. But, after the sample was irradiated using gamma irradiator with about 70 kGy dose, by using EPR spectrometre the obtained spectra results showed that free radical occured over the sample (Figure 3.).

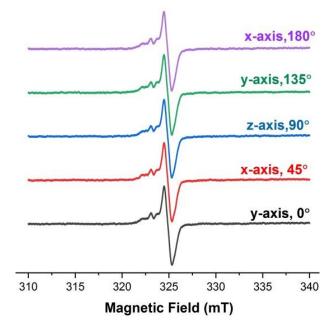


Figure 5 The EPR spectra of gamma-irradiated TRIPOD at different angles towards the x, y, z axes in magnetic field at room temperature

Figure 5 showed that one strong peak and one weak peak were obtained from the EPR measurements. Also, the shapes, intensities and distances between lines were not changed in the spectra along the three (x, y, z) axes which are perpendicular to each other. So, the results mean that those isotropic behaviors were seen. Also, it was found that obtained spectra were not dependent on the magnetic field.

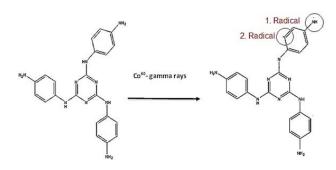


Figure 6 Radicalic molecular structures

When the recorded spectra were examined, it was defined unpaired electrons, which were produced by breaking of bonding of nitrogen and hydrogen atoms (NH₂, NH). As a result of deep analyzed, it was determined that there were two radicals formed under the gamma-rays (its representation is marked as 1. radical and 2. radical in Figure.6). Taking account to recorded EPR spectra and molecular structure, the first radical was defined as NH, so the spectrum was split into 1:1 intensity ratio, firstly and then split into 1:1:1 intensity ratio, due to hydrogen and nitrogen atoms in the molecular structure, respectively. The second radical was localized on the carbon atom and interacted with hydrogen atom. But, it was found that, its hyperfine constant was small. So, it was observed that second radical has small effects on the spectrum. The hyperfine constants and spectroscopic splitting factors (g-values) were calculated for each radical as follow: $a_H=1.475$ mT, $a_N=0.985$ mT and g=1.9995 for labeled 1. radical; $a_{\rm H}$ = 0.38mT and g=1.9957 for labeled 2. radical. Also, these arguments were supported by a simulation, as shown in Figure 7.

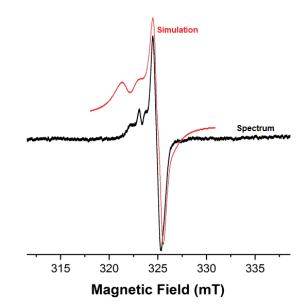


Figure 7 The EPR spectrum of gamma irradiated TRIPOD and its simulation

4. CONCLUSION

In present work, multidirectional cyanuric chloride based was synthesized. The versatile molecule including 1, 3, 5-triazine derivative was prepared by the reaction of cyanuric chloride with p-phenylenediamine. The one step was consisted of preparing 2,4,6-tris(*p*-aminoaanilino)-1,3,5-triazine by substitution of only chloride atoms of cyanuric chloride. It was characterized by the elemental analysis, ¹H - NMR, FT-IR measurements where exchange of the chloro by the amine group was seen. According to measurement, the EPR unirradiated polycrystalline sample did not give any signal. After irradiation, the stable free radical structure was determined. Hyperfine couplings and spectroscopic splitting factor of radical structure were formed under the gamma-rays and these values were calculated and verified by computer simulation.

Acknowledgments

The authors would like to acknowledge the reviewers and editors of Sakarya University Journal of Science.

Funding

This study was partially supported by the Scientific Research Projects coordination center of Selçuk University, Konya, Turkey (Project No: 20401116).

Authors' Contribution

The authors contributed equally to the study.

The Declaration of Conflict of Interest/ Common Interest

No conflict of interest or common interest has been declared by the authors.

The Declaration of Ethics Committee Approval

This study does not require ethics committee permission or any special permission.

The Declaration of Research and Publication Ethics

The authors of the paper declare that they comply with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that they do not make any falsification on the data collected. In declare addition. they that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic publication environment other than Sakarya University Journal of Science.

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