Testing the Possibility of Photochemical Synthesis of Chlorinated Phenols, Benzenes and Biphenyl: Pre-study Guide for Standards Synthesis

Nabil Shahin1*, Nawaf Abu-Khalaf2, Mazen Salman1,2, Harun Parlar3

1Technical and Applied Research Center (TARC), Palestine Technical University-Kadoorie (PTUK), Tulkarm, Palestine
2College of Agricultural Sciences and Technology, Palestine Technical University-Kadoorie (PTUK), Tulkarm, Palestine
3Former Department for Chemical Technical Analysis and Chemical Food Technology, Research Center Weihenstephan for Brewing and Food Quality, Freising-Weihenstephan, Germany.

* Corresponding author E-mail address: nabilshahin@hotmail.de

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Abstract: Since deuterium 2H (D) is an isotope of hydrogen 1H, the testing of the possibility of photochemical synthesis of marked chlorinated phenol, biphenyl and benzene using normal solvents was studied. The irradiation of full chlorinated compounds dissolved in normal solvents such as MeOH or n-hexane has led to a reaction substitution in which a chlorine atom was substituted by hydrogen atom forming less grade chlorinated chlorophenols, biphenyls and benzenes. The quantum yields of pentachlorophenol, decachlorobiphenyl and hexachlorobenzene under irradiation using polychromatic light were calculated and found to be 5.7 x 10⁻³, 1.6 x 10⁻² and 1.2 x 10⁻² Mol·Einstein⁻¹, respectively. Depending on this study the production of marked chlorinated or non-chlorinated compounds using deuterated appropriate solvents such as MeOH d₄ or n-hexane d₁₄ is possible. However, more efforts should be made towards chromatographically separation of synthesized standards and byproducts in order to make the use of these marked compounds as standards in residue analysis feasible.

Keywords: marked standards, polychromatic quantum yield, synthesis, photochemical, pentachlorophenol, decachlorobiphenyl, hexachlorobenzene.

Introduction

The development of the chemical industry at the end of previous century has left its fingerprint in almost all areas of life. Without this development the mass production of food, textile, equipment, dyestuffs, pharmaceuticals etc. would be quite impossible. Nevertheless, these great achievements of the chemical industry were in many cases at the costs of the environment. By application of chemicals on a large-scale such as herbicides, pesticides and fungicide in the agriculture (Jeliazkov et al. 2016) or flame retardants in the construction or equipment industry (de Jong et al. 2016) they could be released into water, soil and air (Careghini et al., 2015; Syed et al., 2013). By accumulation of these chemicals in the food chain they could reach the human body and accumulate in the fatty tissues, since many of these chemicals are lipophilic (Parlar, 1991). Since many of these compounds are carcinogenic and mutagenic the necessity of their analyses in blood, mother milk and human tissues and in different environmental matrices like air, water and soil has gained more importance (Martellini et al. 2016; Wilson et al. 2016).

Despite that enormous advances have been made to the residue analysis in the last decades (Mol et al., 2015), this domain is still considered as error-prone (Griepink, 1982). In general the quality assurance of the environmental analysis (Hoff et al., 2015) depends on the additive of the suitable standards at different points of sample preparation process (Oehme, 1998). These standards are usually C₁₃ labeled or deuterated compounds with the same chemical and physical properties as that of the analyst and these are inexistent in nature. The quality of the analytical method could be evaluated by determination of the standards recovery rate after sample preparation (Oehme, 1998). Most of the contaminants are fully or partially halogenated compounds and the synthesis of their...
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correspondent standards may represent an economical drawback. Since these substances can undergo a substitution chemical reaction, in presence of suitable light energy and another hydrocarbons compound, this process could present a cheap way to synthesize deuterium labeled standards (Mansour, 1980; Parlar, 1978). As a first step a radical halogen atom could be abstracted from excited halogenated molecules possessing a high electron density such as benzene rings and double bonds after irradiation (chemical equations 1 and 2). The evolved molecule radical could attack R-H bond of an organic solution to form a hydrogen radical and another molecule radical. The abstracted hydrogen radical would substitute the lost halogen atom in the halogenated compound (chemical equation 3). A dimerization between two radical molecules could take place as a side reaction (chemical equation 4). The substituted halogen radical would react with the radical molecule of the organic solution to form another halogenated compound (chemical equation 5) (Dwivedi & Pande, 2003; Manzano et al., 2004; Ngim et al., 2000; Nyman et al., 2002; Wei, 1999)

Within the scope of this work suitable hydrogen labeled standards such as chlorinated phenols, benzenes and biphenyls for purpose of residue analysis use should be photochemically synthesized. Since we are considering photochemical reactions the quantum yield of the process will be calculated as well as the half lifetime and the reductive degradation constant of the chemical precursor.

Materials and methods

Fe (II) ions determination

In this work Fe (II) was determined photometrically by complexing Fe (II) ions with 1, 10-phenanthroline. The absorption of the formed brown red complex was measured by means of UV/VIS-photospectrometer (UV-2401 PC, Shimadzu, Japan) at a wavelength of 510 nm.

Poly chlorinated phenols analysis

Pentachlorophenol was analysed by HPLC with a pump of Gynkotek model 480, UV detector of Gynkotek type SP-6, autosampler of Spark Holland model SPW 125 Fix and Chromelone software version 6.70 of DIONEX. A 100-5 c18 ec and 125 mm length column with internal diameter of 4 mm and particle size of 5 µm was applied as a stationary phase. The mobile phase was a mixture of methanol, water and phosphoric acid (800: 200: 3.2 ml) with flow rate of 0.6 ml/minute⁻¹. The detection wavelength was 254 nm. The products and byproducts were studied using GC (HP 5890 Series II) coupled with MS (Finnigan Mat, Model 8200). DB 5 column (30 m length, 0.25 mm inner diameter and 0.25 µm film thicknesses) was applied. Oven temperature was programmed from 80 to 300°C. The injector temperature was maintained at 300°C and transfer line temperature was 250°C. Helium served as a carrier gas with a flow rate of 1.23 ml/minute⁻¹ and the injection volume was 0.4 µl. The MS source temperature was 210°C with ionization energy of 70 e.V. The acceleration and multiplier voltage were 3 and 1.5 kV, respectively.

PCBs analysis

PCBs quantitative analysis were performed using GC/ECD (Dani, model 86.10) and a ZB-5 capillary column with length of 60 m, inner diameter of 0.25 mm and a film thickness of 0.25 µm. The column initial temperature was 90°C at which remained 2 minutes constant and gradually was increased to 290°C at a rate of 8°C/minute. The carrier gas helium had a flow rate of 0.95 ml/minute. The temperature of

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the ions source and quadrupole of MS were 200 and 100°C, respectively.

Chlorinated benzene analysis

The analysis of mono-, di-, tri, tetra-, penta- and hexachlorobenzene was done also by means of GC. HP 5890 (series II) was used as a GC and the chlorinated benzenes were separated on a 30 m long DB-5 column with an inner diameter of 0.25 mm and a film thickness of 0.25 µm. The separation temperature programme was as following: starting with a temperature of 60°C for 5 minutes then increasing to 196°C at a rate of 3°C/minute and remaining for 1 minute and then with a rate of 10°C/minute increasing the temperature to 250°C with a hold time of 3.5 minutes. The injection temperature was 270°C, injection volume was 1 µl and flow rate of helium was 1.2 ml/minute. The temperature source of the MS (Finnigan Mat, model 8200) was 210°C. The acceleration and multiplier voltage were 1.2 and 3 kV respectively. The ionization energy was 70 e.V.

Experimental setup

An optical bench was used to synthesize standards photochemically, which consists of following parts:

1- The irradiation light was provided by a middle pressure mercury lamp (HPK 125 W), which its emission spectrum is given in Figure 1. The lamp was fixed in a box made of metal with a concave mirror behind the lamp and a convex lens in front of it to focus the light.

2- A 3 ml quartz cuvette used as reactor fixed on a metallic bracket with water inlet and outlet in order to enable the reactor temperature to be controlled. The reaction solution in the cuvette was stirred by means of magnetic stirrer with a 2 mm magnetic stirring staff.

3- The lamp metallic box and the cuvette bracket were fixed on a three-cornered steel base plate. The whole gadget was insulated from the day light by a wooden box.

The experimental setup is shown in Figure 2.

Actinometry calculations

In this study the quantum yield of dechlorination reactions of some chlorinated compounds in presence of n-hexane or methanol as a solvent and under poly chromatic irradiation was determined. For this purpose the photons number of the incidental poly chromatic light on the cuvette \( P_{0,real} \) was determined by calculating the theoretical photons number \( P_{0,tho} \), if the whole photons number of the irradiation source would fall on the projection area of cuvette and also by calculating the theoretical flow rate of the reacted molecules number of the actinometer (ferri oxalate) \( \frac{dn_{Ac}}{dt}_{tho} \), if the same irradiation source would be used. By performing an actinometric measurement the real flow rate of the reacted molecules number of the actinometer \( \frac{dn_{Ac}}{dt}_{exp} \) using the above mentioned source of light was experimentally determined. Thus the real incidental photons number of the lamp could
be calculated using a simple proportion formula (equation 1) (Shahin, 2002).

$$\left( P_0 \right)_{real} = \frac{\left( \frac{dn_{Ac}}{dt} \right)_{exp.}}{\left( \frac{dn_{Ac}}{dt} \right)_{theo}} \quad \text{Equation 1}$$

The theoretical incidental photons number of the lamp could be calculated by integrating the differential photons number equation 2, which could be calculated from a power emission density spectrum of the HPK lamp fig.1 as it shown in equation 3.

$$\left( P_0 \right)_{theo} = \int_{\lambda_{ma}}^{\lambda_{mi}} P_{0,\lambda} d\lambda \quad \text{Equation 2}$$

Where as

- $\lambda_{ma}$: the maximal wavelength
- $\lambda_{mi}$: the minimal wavelength
- $P_{0,\lambda}$: differential photon number at a certain wavelength

$$P_{0,\lambda} = \frac{A \cdot L_{\lambda}}{E_{p,\lambda}} \quad \text{Equation 3}$$

where as

- $A$: projection area of the cuvette
- $L_{\lambda}$: differential light power density at a certain wavelength
- $E_{p,\lambda}$: differential power of a photon at a certain wavelength

$E_{p,\lambda}$ could be calculated by implementing Planck’s law:

$$E_{p,\lambda} = h \cdot \nu = \frac{h \cdot c}{\lambda} \quad \text{Equation 4}$$

where as:

- $h$: Planck’s constant
- $\nu$: wave frequency

Based on the definition of the quantum yield of an actinometer for a monochromatic light ($\Phi_{Ac,\lambda}$) (equation 5) and after solving the equation to the flow rate of the reacted actinometer molecule number at a certain wavelength $\left( \frac{dn_{Ac,\lambda}}{dt} \right)$ and substituting the flow rate of the absorbed photons number at a certain wavelength $P_{abs,Ac,\lambda}$ in equation 5 by that in equation 6 we would have equation 7.

$$\Phi_{Ac,\lambda} = \frac{\frac{dn_{Ac,\lambda}}{dt}}{P_{abs,Ac,\lambda}} \quad \text{Equation 5}$$

$$P_{abs,Ac,\lambda} = P_{0,\lambda} \cdot \left( 1 - 10^{-\varepsilon_{Ac,\lambda} c_{Ac,\lambda}} \right) \quad \text{Equation 6}$$

where as:

- $P_{0,\lambda}$: flow rate of the incidental photons number from the lamp on the projection area of the cuvette
- $\varepsilon_{Ac,\lambda}$: extinction coefficient of the actinometer
- $c_{Ac}$: actinometer concentration
- $l$: optical length

$$\left( \frac{dn_{Ac,\lambda}}{dt} \right)_{theo} = \Phi_{Ac,\lambda} \cdot P_{0,\lambda} \cdot \left( 1 - 10^{-\varepsilon_{Ac,\lambda} c_{Ac,\lambda}} \right) \quad \text{Equation 7}$$

The theoretical flow rate of the reacted actinometer molecule number for whole wavelengths could be calculated by integrating equation 7 and after making a simplification by considering the term equal to 1 by a total absorption as shown in equation 8:

$$\int_{\lambda_{ma}}^{\lambda_{mi}} \left( \frac{dn_{Ac,\lambda}}{dt} \right)_{theo} d\lambda = \int_{\lambda_{ma}}^{\lambda_{mi}} \Phi_{Ac,\lambda} \cdot P_{0,\lambda} \cdot \lambda_{max} \cdot \lambda_{mi} \cdot \frac{L_{\lambda}}{E_{p,\lambda}} \cdot d\lambda \quad \text{Equation 8}$$

Again substituting eq. 3 and 4 in 8 will well have this final form (equation 9):

$$\int_{\lambda_{ma}}^{\lambda_{mi}} \left( \frac{dn_{Ac,\lambda}}{dt} \right)_{theo} d\lambda = A \cdot \int_{\lambda_{ma}}^{\lambda_{mi}} \Phi_{Ac,\lambda} \cdot L_{\lambda} \cdot E_{p,\lambda} \cdot d\lambda \quad \text{Equation 9}$$
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Equations 2 and 9 could be solved numerically by for instance implementing Trapez-rule (equations 10 and 11):

\[
(P_{n})_{\text{theo}} = A \cdot \sum_{i=1}^{n} \left( I_{n-i} - I_{i} \right) \cdot \frac{(P_{n,i})_{\text{exp}} - (P_{n,i})_{\text{theo}}}{2} \quad \text{Equation 10}
\]

\[
\left( \frac{dn}{dp} \right)_{\text{theo}} = A \sum_{i=1}^{n} \left( I_{n-i} - I_{i} \right) \left( \frac{(P_{n,i})_{\text{exp}} - (P_{n,i})_{\text{theo}}}{2} \right) \quad \text{Equation 11}
\]

where as:

i: wavelength index

Applying the quantum yield of the used actinometer in this case ferri oxalate (Braun, 1991) the theoretical flow rate of photons and reacted actinometer molecules number could be calculated.

Potassium ferri oxalate was used as an actinometer with a concentration of 0.15 M in order to ensure a total light absorption. By irradiating ferri oxalate complex Fe (III) ions were reduced into Fe (II). After complexing Fe (II) with 1,10-Phenanthroline the complex absorption was measured and thus Fe(II) concentration was photometrically determined. From the slope of Fe (II) concentration curve as function of time, \((dn/Ap)/(dt)_{\text{exp}}\) was calculated.

Results and discussion

Before starting deuteration preliminary tests should be performed using n-C\(_6\)H\(_{14}\) or CH\(_3\)OH solvents in order to make sure that the standards synthesis by reductive dechlorination of chlorinated chemical precursors are going to be working successfully in respect of efficiency and chromatographical separation of the synthesized products, since direct starting with n-hexane D14 and Methanol D4 would be uneconomical. In this case the chemistry of deuterated and normal solvent should be the same.

Pentachlorophenol

Deuterated chlorophenols, such like 2-Deuterotetra-, 2,6-Dideuterotri-, 2,3,6-Trideutero- and 2,3,5,6-Tetradieuterochlorophenol, could serve as internal standards for the analysis of different environmental matrices. Therefore a pentachlorophenol solution in methanol with a concentration of 0.01 M was prepared and in a 3 ml cuvette irradiated in the optical bench after it was saturated with nitrogen in order to prevent any oxidation process during irradiation. This high concentration was chosen to insure a total light absorption.

The photochemical degradation of Pentachlorophenol is a reaction of pseudo first order with a degradation rate constant \((k_{PCP})\) of 0.0488 h\(^{-1}\) and a half-life of about 14 hours. In wave range between 200 nm (< Quarz cut off) and 360 nm, where Pentachlorophenol absorbs light the quantum yield by poly chromatic light absorption \((\Phi_{PCP})\) was calculated to be 5.7 X 10\(^{-3}\) Mol/Einstein\(^{1}\).

After irradiation in methanol the formed dechlorinated chlorophenols and the byproducts were analyzed using GC/MS. A full scan chromatograph of a sample containing pentachlorophenol in methanol after 7 hours irradiation time is shown in Figure 3. After pentachlorophenol was exited by irradiation mainly at a wavelength of 210 nm (Puplampu, 2000) a radical chlorine atom would split off and a tetrachlorophenol radical would be formed. The last one would take a hydrogen atom from H-donor such as methanol to form tetrachlorophenol. By continuing irradiation tetrachlorophenol would take the same course to form trichlorophenols. According to the sample analysis tetra- and trichlorophenols has been proven to be existent in the sample after 7 hours of irradiation. Since photochemical reactions form radical atoms and molecules, which are unselective reactions (Czaplicka, 2006), many trichlorophenols would be formed beside many unrequested intermediates and by products such as pentachlorobenzenes, methoxy-chlorophenols and chlorinated hydroquinone.
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Figure 3. GC/MS chromatogram of a pentachlorophenol solution with an initial concentration of 0.01 M in methanol after 7 hours of irradiation time.

Many preparative separation experiments have been undertaken using thin layer technique and different eluents with different polarity and mixtures and column chromatography to separate the synthesized standards from by-products. With available techniques we were unable to achieve any separation. In this case a preparative HPLC should be tested.

Decachlorobiphenyl

Due to the wide use of poly chlorinated biphenyls in different industries such as automobile, textile, electrical and construction industry and due to their ability to accumulate in blood, and human tissues and milk reaching human body (Edogru, 2005) through the food chain is quite important to develop analytical methods hence PCBs standards.

For this purpose we tested the photochemical dechlorination of decachlorobiphenyl in n-hexane solvent to synthesize chlorinated biphenyls with different chlorination grades. A decachlorobiphenyl solution with a concentration of 0.0035 M in n-hexane was irradiated at optical bench. Since high chlorinated poly deutero biphenyls are considered to be limited standards and because they are interproducts for formation of the suitable low chlorinated polyeutero biphenyls standards the dechlorination of decachlorobiphenyl was timewise persecuted in order to determine the optimal irradiation time to develop the desired standards. Figure 4 shows the percentage distribution of dechlorinated biphenyls produced at various irradiation times. After 5 hours of irradiation time heptachlorobiphenyls have the highest proportion with a value of 42%. Hexachlorobiphenyls would have the highest proportion with a value of 50% after 8 hours irradiation time. If we continue to irradiate, we would have the following distribution: 2.3% the total sum of trichloro biphenyls isomers (Σ TriCB), 3.4 % Σ TetraCB, 20.4% Σ PentaCB, 21.3% Σ HexaCB and the rest is the sum of octa, nona isomers and deca chloro biphenyls.

Under the experimental conditions the photochemical degradation was found to be as pseudo first order reaction with a reaction rate constant of 0.62 h⁻¹ and a half-life of 1.11 hours. The calculated value of the quantum yield of DeCB (Φ_deCB) at an initial concentration of 0.0035 M and in the total absorption range of DeCB between 200 and 308 nm was 1.6 × 10⁻² Mol-Einstein⁻¹.

Hexachlorobenzene

The extensive use of chlorinated benzenes in many applications in the agriculture and industry led to its persistent presence as pollutant in nature (Bailey, 2001). It can accumulate in tissues reaching the human body via food chain and many other ways (Koshioka, 1987). Therefore an appropriate deutero chlorinated benzene is needed to insure a good residue analysis quality of chlorinated benzenes in many contaminated matrices in the nature such as air, soil, water, human tissues, etc.

The photochemical exchange of chlorine atom in chlorinated benzene by a deuterium atom, which could be denoted by deuterated solvents like n-Hexane d14, could be a possible synthesis way for deuterated chlorobenzenes standards. The standards synthesis was tested by irradiating of a HBC solution with a concentration of 0.0133 M in n-Hexane as a solvent. The irradiation took place in a cuvette on an optical bench using HPK (125 W) lamp. The incident light in wavelength range between 200 and 315 nm (the total absorption range of HCB) in the cuvette was calculated to be 5.8 × 10¹⁰ photon/s. The reaction rate constant of HCB dechlorination was estimated to be 0.19 h⁻¹ and a half life time of 3.7 hours. With an initial degradation
rate of 0.00197 Mol⋅l⁻¹⋅h⁻¹ the poly chromatic quantum yield in the same absorption wavelength range of HCB (Φ_{HCB}) was 1.7 × 10⁻² Mol-Einstein⁻¹.

In this study we applied normal hexane instead of hexane d14 to investigate the efficiency and the mechanism of this process. Figure 5 shows the formed fractions of mono, di, tri, tetra, penta and hexachlorobenzene during irradiation time by irradiation of HCB with an initial concentration of 0.0133 M in a n-hexane as a solvent.

Figure 5: formed chlorinated benzenes during irradiation time by irradiation of HCB solution in n-hexane as a solvent.

At the beginning under irradiation HCB would be reductively single dechlorinated forming pentachlorobenzene. The oxidative degradation of HCB was ruled out by saturating the solution with nitrogen gas before irradiation. The HCB concentration decreases with increasing irradiation time. On the other hand pentachlorobenzene concentration increases and reaches the maximum after 5.5 hours. Then it starts to decrease forming tetrachlorobenzenes of three isomers. The formed isomers fractions during irradiation time can be seen in Figure 6.

Figure 6. Fractions of formed tetrachlorobenzene isomers during irradiation time.

After about 8.5 hours of irradiation time tetrachlorobenzenes concentrations reach their maximum and their isomers start to dechlorinate forming three trichloro isomers as it is shown in Figure 7.

Figure 7. Fractions of formed trichlorobenzene isomers during irradiation time.

With increasing irradiation time and trichlorobenzene isomers reaching their highest concentration the concentrations of dichlorobenzene isomers began to increase till 13.5 hours of irradiation time to start to decrease forming monochlorobenzene. Figure 8 shows the trichlorobenzenes fractions, that were formed during irradiation time.
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Figure 8. Fractions of formed Dichlorobenzene isomers during irradiation time.

The photochemical dechlorination way of HCB in our experiments is consistent with the reaction way which is summarized by Koshioka et al. (1987) in Figure 9.

In the same way the deuterated chlorobenzenes standards could be photochemically synthesized by applying deuterated hexane d14 instead of n-hexane as a solvent. From Figure 5 and depending on the irradiation time the mixture of marked chlorobenzene standard could be determined. For instance after 9 hours irradiation time and under the experimental conditions we would have a standard mixture which consist of 1.3% mono-; 5.2% di-; 24.8% tri-; 56.3% tetra-, 10.8% penta- and 1.7% hexachlorobenzene. With increasing irradiation time the concentration of low chlorinated benzenes would increase at the cost of that of high chlorinated. However, after synthesis the standard should be purified and separated from byproducts that were formed during synthesis.

Conclusion

The idea of photochemical synthesis of marked poly chlorinated phenols, biphenyls and benzenes standards was studied applying normal hydrogenated solvents instead of deuterated solvents. The synthesis was performed in an optical bench using a HPK 125 W as an irradiation source. By means of actinometry the polychromatic incident light into the photo reactor, which was a cuvette, was determined as well as quantum yields of hexachlorobenzene, pentachlorophenol and decachlorobiphenyl. The photochemical reductive degradation of these three compounds was pseudo first order reaction. They were photochemically dechlorinated, where the abstracted chlorine atoms were substituted with hydrogen atoms originated from the solvent compound.

The chromatography of a 7 hours irradiated pentachlorophenol samples dissolved in methanol showed that tetra-, trichlorophenols and many byproducts were formed.

The concentration distribution of synthesized chlorinated biphenyls depends on irradiation time. After 5 hours of irradiation time the highest concentration was that of formed heptachlorobiphenyls, while the highest one was hexachlorobiphenyl after 8 hours.

Like pentachlorophenol and decachlorobiphenyl the distribution of formed chlorinated benzene is dependent upon irradiation time. Nine hours of irradiation time gave the following distribution: 1.3% mono-; 5.2% di-; 24.8% tri-; 56.3% tetra-, 10.8% penta- und 1.7% hexachlorobenzene.

Thus photochemical dechlorination is a promising way of a labeled chlorinated standards synthesis and perchlorinated phenol, biphenyl and benzene could serve as good chemical precursors for the synthesis of these standards. However, more efforts are still needed towards standards and by products purification and separation from each other in order to make use of the whole process for effective standards production.
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