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An Extractive Spectrophotometric Method for the Determination of Propargite in Various Environmental and Biological Samples.

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Abstract: ***An extractive, sensitive, selective cheaper spectrophotometric method has been developed for the detection and determination of propargite in various environmental and biological is based on bromination followed by blue Comlpex formation with starch in presence of potassium iodide. The dyes formed are measured at 590nm for propargite. Beer's law is obeyed over concentration ranges from 0.5µg to 8µg. The molar absorptivity and Sandell's sensitivity were found to be 3.13 x 10^{11} L $mol^{-1}cm^{-1}$ and 0.0003µg cm^{-2} respectively. The standard deviation and relative standard deviation are observed as ±0.007 and relative standard deviation is 1.994% respectively. Various important analytical parameters were evaluated. The method was applied successfully to the determination of propargite in grain, fruits and soil samples.***

Keywords: - Propargite; Spectrophotometer; Bromination; Environmental sample; Biological samples.

I. INTRODUCTION

The world is now stepping towards the advancement in technology and also widely exposed to many harmful chemicals used in these technologies, which is later returned back as a revenge of environment to mankind. The needs of human population have now forced to think for the remedies against pest and stop the loss in productivity due to pest. In this context many new and old harmful chemicals are introduced in the market under new brands and names. And now is well accepted fact that many food products that we eat today have the possibility of being contaminated by various chemicals used from planting to processing. These chemicals have been shown to cause illnesses for which some concerned government agencies have instituted regulatory mechanisms to minimize the risks and the effects on human. EPA's human health risk assessment for pesticides indicates that food risk, both acute and chronic, also are carcinogenic based on the appearance of intestinal tumors in the test animals.[1] Despite widespread use of protective devices in this area significant inhibition occurred as a result of protective clothing soaked with pesticides, thereby increasing dermal exposure or because of other factors. It is for such concerns that reliable and alarming determination techniques are needed to effect proper regulatory standards for the protection of people's nutritional health.[2]

Propargite is an organosulphur miticide / acaricide used on variety of bearing agricultural food crops, as well as non- food agricultural sites. It was first registered 1969. Approximately 2 million pounds of propargite active ingredient are applied annually.[3-4,5]. Propargite is a specific miticide which may be used to control European red mite and two spotted

mite on horticultural crops[6]. Propargite is classified as a B2 chemical carcinogen based on the appearance of intestinal tumors in test animals. EPA has identified a chronic reproductive risk of concern to birds and mammals, and some risk to aquatic species. EPA's dietary risk analysis consists of three parts: acute dietary risk, and chronic cancer risk.[7]
Based on toxicity studies submitted by the registrant, propargite poses a potential for adverse effects on reproduction in birds and mammals. Risk to aquatic organisms and plants are generally lower than the risk for birds and mammals. Propargite is also expected to be highly toxic to amphibians. [8].
Based on monitoring data, the time weighted average propargite concentration in ambient surface water samples from the USGS/NAWQA (Oristimba Creek Watershed) for the years 1992 and1993 were 0.30 and 1.24 ppb, respectively. Therefore, there is a potential cancer risk of concern when ambient monitoring data are used to estimate drinking water exposure to propargite from surface water sources.[9-10] There are some methods reported using some advance and sophisticated apparatus such as gas Chromatography method[11], Flame Photometric Method, Gas chromatography-Mass spectrometric method [8- 9] etc.
In this paper, a method for the determination of propargite is described. The method is less toxic and is highly sensitive and rapid. It is based on the bromination of propargite later reaction is proceeded with potassium iodide in presence of starch to form a blue complex, which is extracted in iso amyl achohol. The complex shows maximum absorbance at 590nm.The method was applied to the determination of propargite in various environmental and some biological samples.

II. EXPERIMENTAL

A. *Apparatus*

A Systronic UV-VIS Spectrophotometer model 104 and Systronic digital pH meter model 335.

B. *Reagent*

All reagents used are Anala R grade and distilled water is used throughout the study.
Standard solution of propargite. A stock solution of 1mg/l solution of propargite is prepared in water. Working standard solution is prepared by appropriate dilution of the stock solution.

Bromine water. A saturated solution of bromine in water was prepared. This solution was prepared daily.

Formic acid. 90% solution.

Potassium iodide, 1% solution.

Starch solution. A 100 mg amount of soluble starch was made into a paste with a few drops of hot water and diluted to 100 ml using nearly boiling water.

Iso Amyl Alcohol-LR 98% pure, for extraction.

III. PROCEDURE

A. *Preparation of calibration graph*

To an aliquot (1-8) of a standard solution containing 0.5-8.0 µg of total propargite placed in a 25 ml calibrated tube, was added 0.5 ml of bromine water and the mixture shaken gently for 2 min. The excess of bromine was removed by drop wise addition of formic acid after which 0.5 ml of potassium iodide and 0.2 ml of starch solution were added and the mixture set aside for a further 2 min. The content was diluted to mark with water and the absorbance measured at 590 nm against a reagent blank as reference.

IV. RESULT AND DISCUSSION

A. *Absorption Spectrum*

The absorption spectrum of the colour system showed maximum absorbance at 590 nm. The reagent blank had negligible absorbance at this wavelength.

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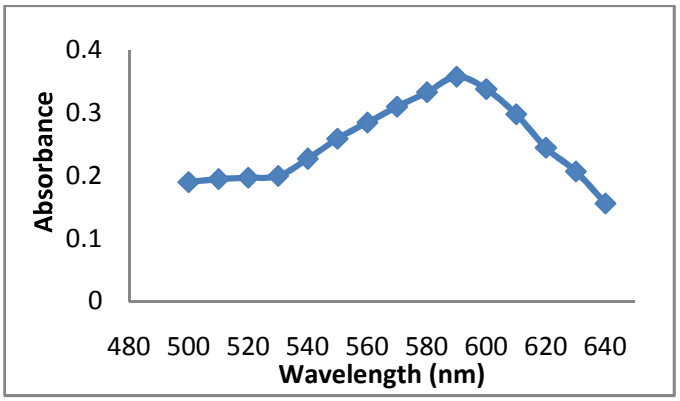

Fig:1.Absorption Curve of the Complex.
Concentration of Propargite in µgm/10ml.

B. Beer's Law Range and Sensitivity

The colour system obeyed Beer's law in the range 0.5-8.0 µg in a final solution volume of 10 ml. The molar absorptivity and Sandell's sensitivity were found to be 3.13×10^{11} l mol^{-1} cm^{-1} and 0.0003µg cm^{-2}, respectively.

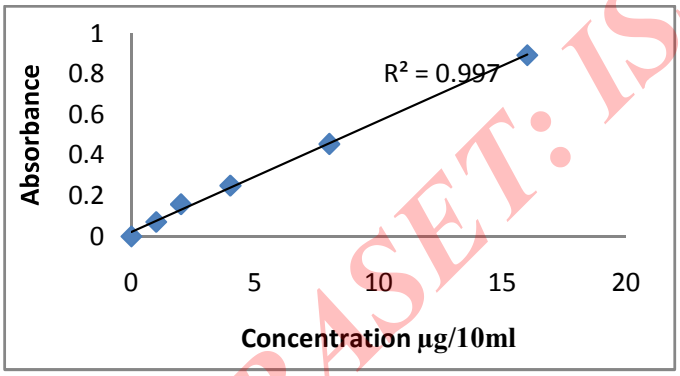

Fig:-2. Calibration Curve for the Determination of Propargite Concentration of Propargite in µgm/10ml. Absorbance λmax=590nm.

C. Reproducibility

The reproducibility of the method was assessed by carrying out seven replicate analysis of a solution containing 4.0 µg of propargite in a final solution volume of 10 ml. The standard deviation and relative standard deviation were found to be ±0.007µg ml^{-1} 1.994%, respectively. (Table- I)

Table- I : Reproducibility of the method

S.No	Absorbance nm
1day	0.358
2day	0.345
3day	0.363
4day	0.341
5day	0.354
6day	0.347
7day	0.355
Mean	0.351
Standard Deviation	±0.007
Relative Standard Deviation	1.994%

D. Effect of reagent concentration

It was found that 0.5 ml each of bromine water and potassium iodide solution and 0.2 ml of starch solution were sufficient for complete reaction. A few drops of formic acid were sufficient for the removal of excess of bromine. Starch solution was prepared daily.

E. Stability of Colour

The blue complex formed was stable for 48 h under optimum conditions.

F. Effect of Time and Temperature

It was found that 2 min were sufficient for bromination, and iodine was liberated immediately on addition of potassium iodide. The colour reaction was completed

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after 2 min. The effect of temperature on the colour reaction and the stability of the dye were studied. It was found that the temperature of between 15 and 25 ^{o}C was suitable for the reaction; the colour faded rapidly at higher temperatures. The colour of the complex disappeared almost completely when it was heated above 80 ^{o}C. At lower temperatures, the colour of the complex gradually faded.

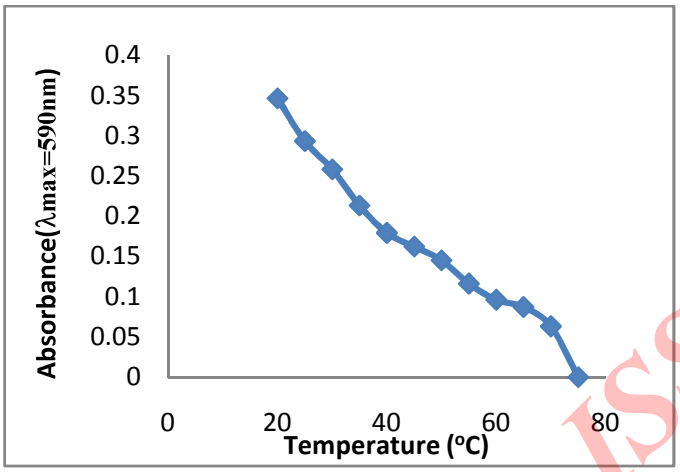

Fig:3. Effect of temperature on Blue Complex of propargite. Concentration of Propargite in µgm/10ml. Absorbance λmax=590nm

G. Effect of foreign species

The effects of various species were studied to assess the validity of the method. It was found that many compounds didn't interfere with the proposed method. The tolerance limits for various ions are given in Table II.

TABLE-II: Effect of Foreign Species
Concentration of propargite = 4µg/10ml

Foreign Species	Tolerance Limit in µg
Cypermethrin	0.336
Dicofol	0.313
Dichlorvos	0.328
Glyphosphate	0.341
Paraquat	0.352
Cu^{2+},Cl^{-}	0.332
Fe^{3+},Cl^{-}	0.344
Pb^{2+}	0.351

V. APPLICATION

A. Determination of propargite in water sample

50 ml of water sample was taken and fortified with known amount of propargite and kept for 3-4 hours, propargite was determined by the proposed as well as the method. The recoveries are shown in Table-3.

B. Determination of propargite in fruits, vegetables, cereals and biological sample

cauliflower, grapes, apple ,orange, rice, soil, beans, French beans, were weighted (5gm), crushed and spiked with known amount of propargite and kept 2-3 hours. In blood and urine (5ml) add known amount of propargite and kept for 2-3 hours. After 2-3 hours propargite was analysed by the proposed method. The recoveries are shown in Table-3. The recoveries range from 84 to 98% by the proposed method.

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VI. CONCLUSION

The proposed method is extractive, sensitive, simple and rapid spectrophotometric method for the determination of propargite. The method used less toxic reagents. The proposed method is good alternative of mostly sophisticated and costly apparatus. It can be successfully applied for the determination of propargite in water, fruits, vegetables, grains and biological samples.

Table- III: Application: Determination of Propargite in Biological and Environmental samples.

Samples (a)	Propargite added (µg) (b)	Propargite originaly found (c)	Propargite obtained in Present method * (d)	% Recovery
Grapes*	4 µg	0.040	0.383	95
Beans*	4 µg	0.093	0.415	89
Cauliflower*	4 µg	0.176	0.527	98
French Beans*	4 µg	0.080	0.393	87
Rice*	4 µg	0.128	0.447	89
Soil*	4 µg	0.211	0.513	84
Water**	4 µg	0.134	0.478	96
Apple*	4 µg	Nil	0.351	98
Orange*	4 µg	0.67	0.417	97
Blood**	4 µg	Nil	0.331	92
Urine**	4 µg	Nil	0.312	87

Mean of three replicate analysis.
* Each 5gm samples taken.
** Each 5ml samples taken.

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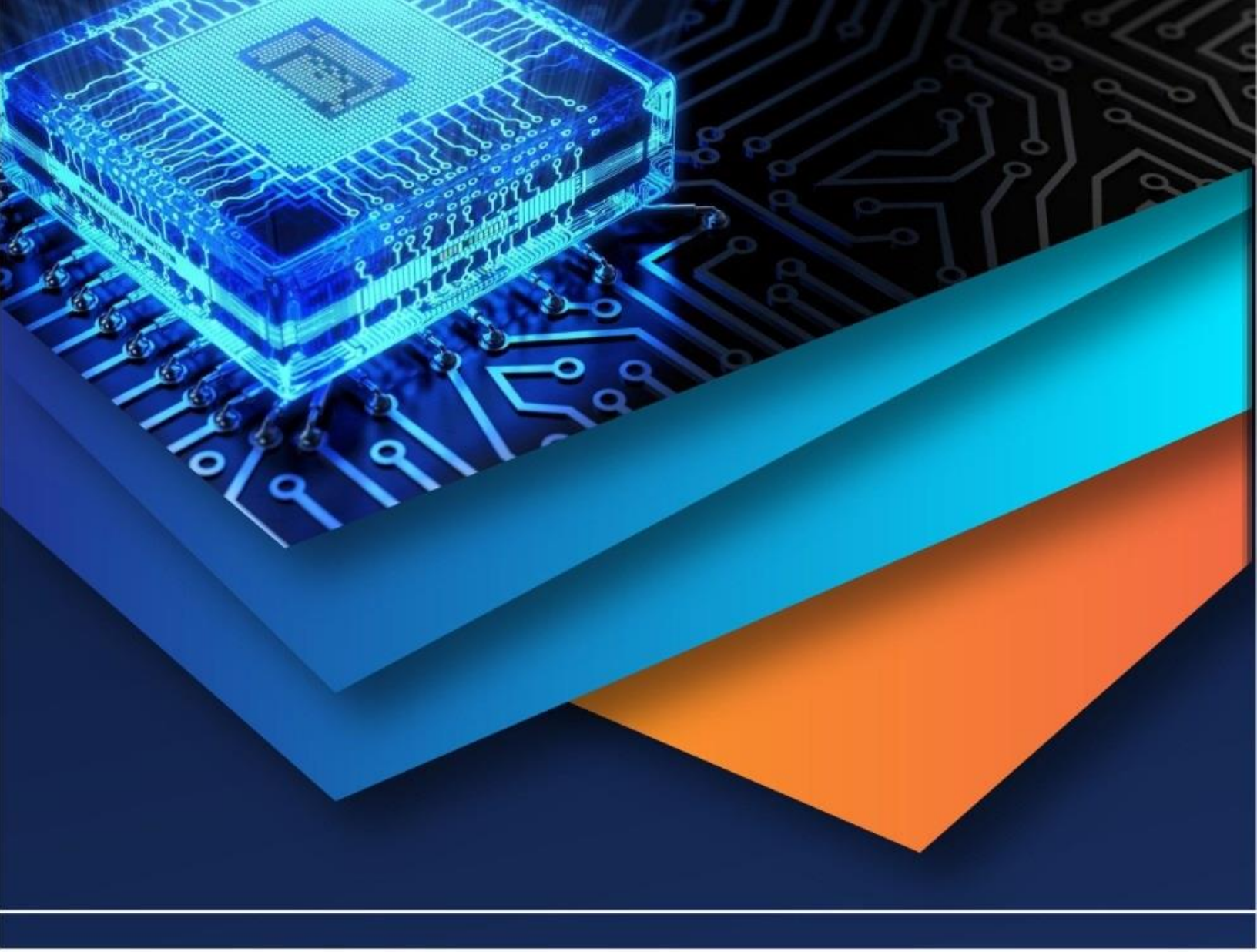

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