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ORIGINAL ARTICLE

# Spectrophotometric Estimation of Polyvinylpyrrolidone, Iodate and Iodine Simultaneously in Povidone-Iodine Complex in Pure and Pharmaceutical Preparations

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***Authors' Contributions***
*All the authors contributed equally to the research that resulted in the submitted manuscript (Conception & Study Design, Data Collection, Data Analysis, Drafting, Critical Review).*

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## ABSTRACT

A simple and selective spectrophotometric method has been proposed for estimation of polyvinylpyrrolidone (PVP), iodate ion and Iodine content simultaneously in PVP-I complex. The PVP, iodate ion and iodine content showed maximum absorbance at 240 nm, 290 nm and 360 nm, respectively. It follows Beer's and Lambert's Law at 0.5 to 8 mg concentration levels with co-relation coefficient ($r^2$) of 0.9981, 0.9988, and 0.9966, respectively. The limit of detection (LOD) of PVP, iodate ion and Iodine content were 0.1 mg, 0.11 mg and 0.9 mg accordingly at their respective wavelengths. Similarly, limit of quantification (LOQ) of PVP, iodate Ion and iodine content were 0.50 mg, 0.51 mg and 0.7 mg, respectively.

The proposed method was successfully applied for estimation of PVP, iodate ion and Iodine content in pharmaceutical preparations. It was validated according to the guidelines of USP and ICH. The accuracy, precision, robustness, specificity and ruggedness of the method were carefully studied. The optimized by studying the possible affecting parameters such as, effect of time, temperature, and pH change on the absorbance of solutions.

**Keywords:** Validation, spectrophotometric, PVP-I complex, iodate, iodine.

## INTRODUCTION

Povidone-iodine is yellow colored crystalline powder. Its 10% solution is widely used as antiseptic in operation theater of public Health [1]. It is a broad spectrum antiseptic agent. It kills not only bacteria but also viruses within 10-120 seconds. Literature survey reveals that spectrophotometric, reflectance spectroscopy and size exclusion chromatography [1-4], by MIC, potentiometric sensors [5, 6] or HPLC and Sequential Flow Injection potentiometric [7, 8] have been used for PVP determination.

There was no any simple accurate and cheap method reported. The purposed method provides simple way to estimate PVP, Iodine and its iodate content simultaneously by using spectrophotometer. PVP is versatile polymer which forms yellow colored complex with iodine through charge transfer mechanism. The chemical structure of povidone-Iodine complex is shown in Figure **1**.

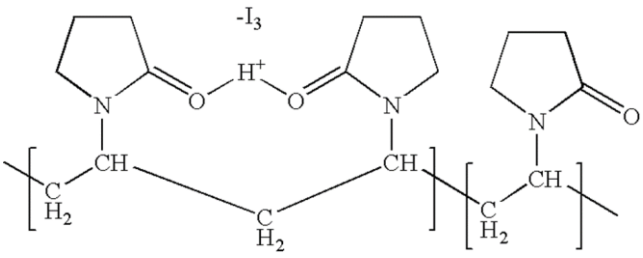


**Figure 1.** Povidone-iodine complex.

The molecular weight of Povidone-Iodine complex is 364 g/mole and meting point is 300°C.

## EXPERIMENTAL

A spectrophotometer (Model, 1601-Shimadzu Corporation, Japan) was used with 1 cm quartz cells in the experiment.

### Material and Reagents

All the materials and reagents purchased were analytical grade and used without further purification. Povidone-Iodine (99.99%) was procured from Basef Chemicals and Sodium thiosulfate (99.0%) was purchased from Merck.

### Pharmaceutical Preparations

Different brands of pharmaceutical preparations were purchased from local pharmacy and were used for analysis using the prosed method, are as follows

1. Povex® Ophth Pharma (Pvt.) Limited (Eye Drop)
2. 2-Pyiodine® Bosh Pharmaceuticals (Topical Solution)
3. Iodex® GSK (Ointment)

### Reagent Preparations

***Preparation of sodium thiosulfate (0.1 M)***: Accurately weighed 13.0 gram of sodium thiosulfate in 500 ml volumetric flask, mixed and made up volume with purified water after complete dissolution.

***Stock solution of povidone-iodine***: Accurately weighed 50 mg PVP-Iodine RS and transferred into 50 ml volumetric flask and dissolved completely in water.

***Test preparations***: Measured volume of 0.5 ml was transferred into 50 ml volumetric flask and made up volume to obtain a final concentration of 1 mg/ml.

## RESULTS AND DISCUSSION

### UV-Vib Spectra of Standard Solutions

The 1 mg/ml solution of povidone-iodine was scanned in between 190 nm to 400 nm of wavelength. Three absorptions maxima were observed at wavelength of 240, 290, and 360 nm. It was found that the wavelength of 240 nm was the indication of povidone while, wavelengths of 290 and 360 nm were the indications of iodate ion and iodine, respectively. The spectra are shown in Figure **2**.

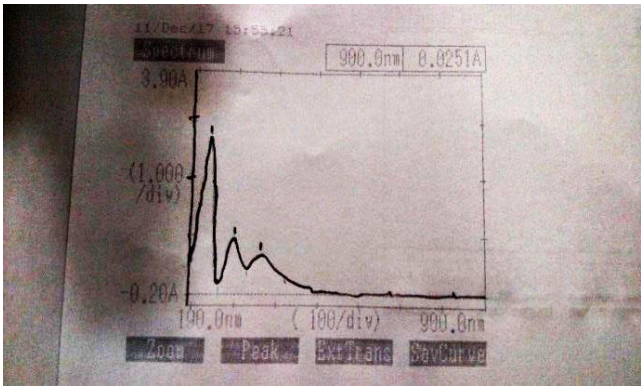


**Figure 2.** UV-Vib spectra of PVP-I complex.

Some portion of PVP-I solution was treated with 0.1 M sodium thiosulfate ($Na_2S_2O_3$) for further confirmation of absorbance maxima of povidone, free iodine and iodate ions present in the complex. After addition of 0.1 M sodium thiosulfate in the complex solution, it was re-scanned and the scanned spectra showed only one peak at wavelength of 240 nm which is shown in Figure **3**.

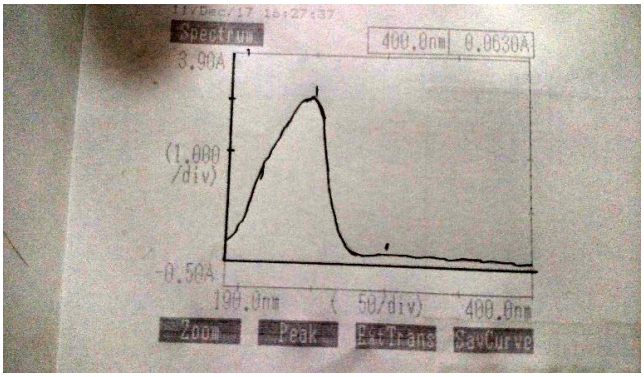


**Figure 3.** UV spectra after oxidation of iodine by $Na_2S_2O_3$ (0.1 M).

The Redox equation of free iodine and sodium thiosulfate is represented as follows:

$$I_2 + 2NaS2O_3^{-2} \text{ ----------} > 2I^- + 2Na_2S_4O_6^{-2}$$

### Optimization of Parameters

The proposed method was optimized by studying of influencing factors on the absorbance of subsequent contents present in the PVP-I complex. The possible factors which could affect the absorbance of the species present in the cited drug were effect of time, effect of pH and effect of temperature. However, the calibrated curve of povidone, iodate and iodine are shown in Figure **4**, **5** and **6**.

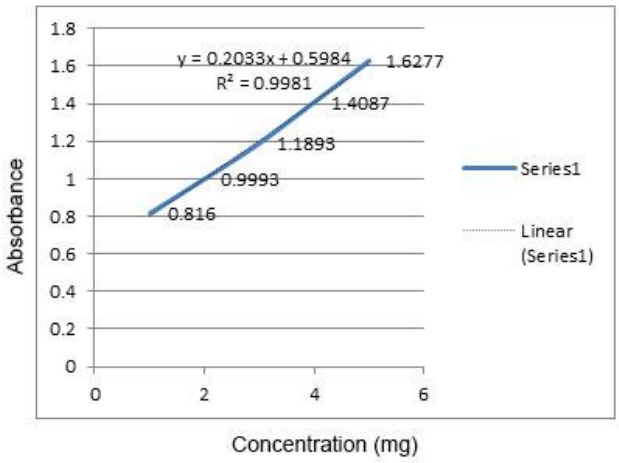


**Figure 4.** Calibration curve of povidone at 240 nm.

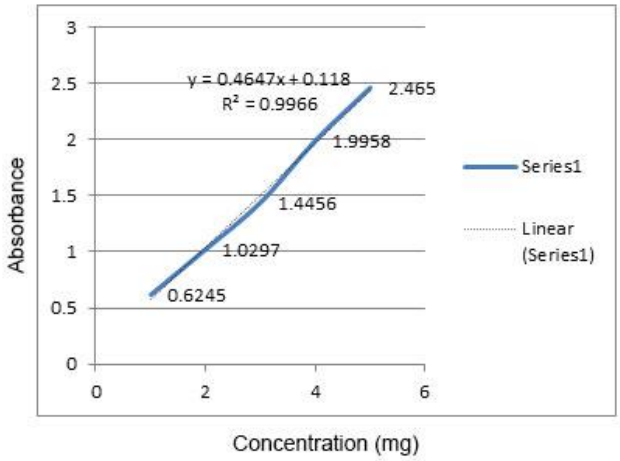


**Figure 6.** Calibration curve for iodine (free $I_2$) at 390 nm.

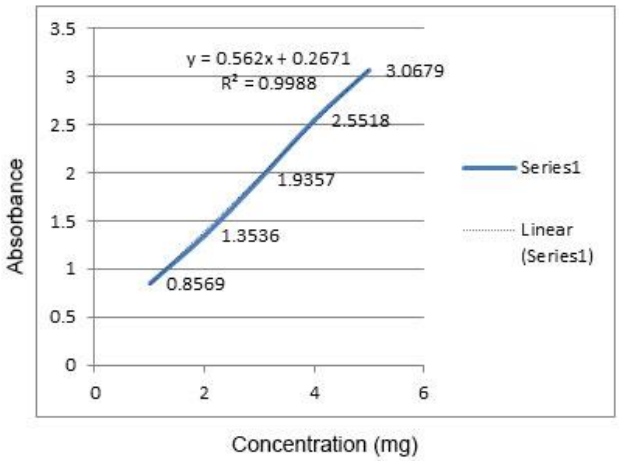


**Figure 5.** Calibration curve for $I_3^-$ ion at 290 nm.

### Time Factor on Absorbance Variation

Three replicate samples of povidone-iodine solutions with different concentrations (4, 5 and 6 mg/ml) were prepared in 10 ml volumetric flasks; the volume of flasks was constituted by distilled water. The absorbance of the solutions was recorded at 240 nm, 290 nm and 360 nm for three hours and the results are shown in Table **1**. The absorbance of solution did not show any significant change, which is the indication of reliability of the proposed method.

**Table 1. Time effect on absorbance.**

| Time hours | Absorbance 240 nm (povidone) | Absorbance 290 nm ($I_3^-$) | Absorbance 360 nm ($I_2$) | Concentration mg/ml |
|---|---|---|---|---|
| 1 | 0.8160 | 0.8569 | 0.6245 | 6 |
| 2 | 0.771 | 0.8510 | 0.6220 | 5 |
| 3 | 0.726 | 0.8453 | 0.6141 | 4 |

**Table 2. pH effect on absorbance.**

| pH | Absorbance 240 nm (povidone) | Absorbance 290 nm ($I_3^-$) | Absorbance 360 nm ($I_2$) | Concentration (mg/ml) |
|---|---|---|---|---|
| 3.6 | 0.3113 | 1.3165 | 0.9020 | 8 mg/ml |
| 4.0 | 0.3108 | 1.3201 | 0.9040 | |
| 5.2 | 0.3114 | 1.316 | 0.9021 | |
| 7.4 | 0.3362 | 1.3166 | 0.9020 | |

### Effect of pH in Absorbance of Povidone-Iodine Solution

To determine the pH effect on the absorbance of the solution (having concentration 8 mg/ml), the pH of the solution was varied from pH 3 to 7.4 by addition of sodium hydroxide (0.1 N). The absorbance was recorded after every change in pH, as represented in Table **2**.

### Effect of Temperature into Absorbance of Solution

Three replicate samples were tested at 15°C, 25°C, and 40°C to observe the variation in absorbance of the solutions. The 0.9 %RSD of the absorbance of samples shows no any reasonable variation by varying the temperature. It is the sign of good stability and reliability of proposed method. The results were tabulated in Table **3**.

## VALIDATION OF METHOD

The proposed method was validated according to guidelines of USP [9]. The parameters such as linearity, accuracy, robustness, ruggedness and specificity of method were studied.

### Linearity

For determination of linearity of povidone, iodine and iodate ion, five replicate samples were tested having concentrations of 4-8 mg/ml and the absorbances were taken at their respective wavelengths. The obtained absorbances showed good linearity, as tabulated in Table **4**. The results of linearity data for quantitative determination of povidone, iodate and iodine are shown in Table **5**.

**Table 3. Temperature effect on absorbance.**

| Temperature (°C) | Absorbance 240 nm (povidone) | Absorbance 290 nm ($I_3^-$) | Absorbance 360 nm ($I_2$) |
|---|---|---|---|
| 15 | 0.9236 | 0.8580 | 0.6252 |
| 25 | 0.9234 | 0.8534 | 0.6245 |
| 40 | 0.9231 | 0.8532 | 0.6243 |
| %RSD | 0.03% | 0.32% | 0.08% |

**Table 4. Linearity.**

| Absorbance 240 nm (povidone) | Absorbance 290 nm ($I_3^-$) | Absorbance 360 nm ($I_2$) | Concentration mg/ml |
|---|---|---|---|
| 0.8160 | 0.8569 | 0.6245 | 4 |
| 0.9993 | 1.3536 | 1.0297 | 5 |
| 1.1893 | 1.9357 | 1.4456 | 6 |
| 1.4087 | 2.5518 | 1.9958 | 7 |
| 1.6277 | 3.0679 | 2.4650 | 8 |

**Table 5. Results of linearity data for quantitative determination of povidone, iodate and iodine.**

| Statistical parameters | Povidone | Iodate | Iodine |
|---|---|---|---|
| Concentration range (mg/ml) | 4-8 mg/ml | 4-8 mg/ml | 4-8 mg/ml |
| Regression equation | Y = 0.2033x + 0.5981 | Y = 0.562x + 0.2671 | Y = 0.4648x + 0.1178 |
| Correlation coefficient (r) | 0.9981 | 0.9988 | 0.9966 |

**LOD and LOQ**

The LOD and LOQ were estimated by measuring SD of 5 times repeated intercepts of calibration curve. It was calculated according to guidelines of USP by using the following equations.

LOD = 3.3*SD/Slop of calibration curve …….. Eq. 1

LOQ = 10*SD/Slop of calibration curve ..…… Eq. 2

SD = Standard deviation of intercepts

The results obtained by using above equations for all components present in the PVP-I complex are presented in Table **6**.

**Table 6. LOD and LOQ of all components.**

| Parameters | Povidone | Iodine | Iodine |
|---|---|---|---|
| LOD | 100 ppm | 110 ppm | 90 ppm |
| LOQ | 500 ppm | 500 ppm | 700 ppm |

**Accuracy**

The accuracy of proposed method was calculated in the form of percent recovery. Five replicate samples were tested to determine the accuracy. The % recovery with 0.34% RSD is the indication of good accuracy of the proposed method. The obtained %recoveries with % RSD are shown in Table **7**.

**Precision**

Precision of the proposed method was estimated by means of interday and intraday precision of povidone, and iodine in PVP-I complex. Their percent recoveries of three replicate samples with %RSD were calculated 3 times in a day for intraday precision. Similarly, the experiment was performed 3 times in 3 different days for determination of interday precision. Their average %RSD was the indication of good precision of the proposed method. The obtained results are tabulated in Table **8**.

**Table 8. Precision of the method.**

| Precision | Povidone | Iodate | Free Iodine |
|---|---|---|---|
| Interday | 104.5% | 93.25% | 99.62% |
| Intraday | 99.93% | 99.87% | 99.82% |

**Robustness**

The robustness of the method was determined by changing the pH of the solution. The % recoveries with 0.44%, 0.16% and 0.13% RSD revealed the robustness of the method, which are shown in Table **9**.

**Ruggedness**

The ruggedness is the measure of capacity of analytical method to remain unaffected by small changes in procedure or in its reagents. The ruggedness of the method was determined at three concentration levels by addition of MeOH in small quantity. The observed percent recoveries revealed excellent stability and reliability of proposed method. The % recoveries are shown in Table **10**.

**Table 7. Accuracy in term of % age recovery of the proposed method.**

| Concentration (mg) | % Recovery of povidone | % Recovery of iodate | % Recovery of free iodine | %RDS |
|---|---|---|---|---|
| 4 | 100.44% | 105.477% | 103.2% | 2.44% |
| 6 | 147.72% | 154.60% | 150.8% | 1.99% |
| 8 | 200.356% | 210.57% | 2o4.7% | 2.44% |

**Table 9. Robustness of the method.**

| pH | Concentration mg/ml | Absorbance of povidone at 240 nm | Absorbance of iodate at 290 nm | Absorbance of iodine at 360 nm |
|---|---|---|---|---|
| 3.6 | 8 | 0.3113 | 1.3165 | 0.9020 |
| 4.0 | | 0.3108 | 1.3201 | 0.9040 |
| 7.4 | | 0.3362 | 1.3166 | 0.9020 |
| %RSD | | 0.44% | 0.16% | 0.13% |

**Table 10. Ruggedness of the method.**

| Parameters | Volume of MeOH (ml) | %Recovery of povidone at 240 nm | %Recovery of iodate at 290 nm | %Recovery of iodine at 360 nm |
|---|---|---|---|---|
| Intraday | 1.0 | 99.9% | 103.7% | 106.4% |
| | 2.0 | 98.5% | 101.4% | 102.7% |
| | 3.0 | 103.2% | 99.98% | 101.9% |
| Interday | 1.0 | 101.6% | 103.2% | 100.5% |
| | 2.0 | 98.8% | 100.75% | 98.96% |
| | 3.0 | 100.9% | 101.6% | 100.3% |

### % Recovery of Different Pharmaceutical Dosage Forms

The mentioned drug was also tested for %age recoveries in different pharmaceutical products, like, Iodex (GSK), Povex (OphthPharma), Pyodine (Bosh Pharma) and results are shown in Figure **7**.

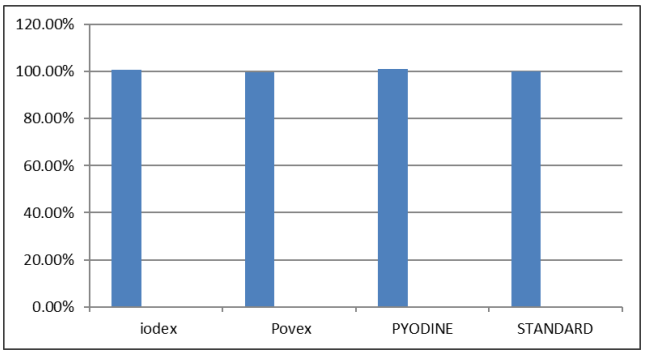


**Figure 7.** % Recoveries of cited drug in different brands and pharmaceutical dosage forms.

## CONCLUSION

The proposed method was successfully developed and validated as per ICH and USP guidelines. This is a very, cheap, simple, quick and easy method to perform. This method can be applied to estimate iodine content in water.

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