

Validation of HEDP Drop Test Kit

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I. INTRODUCTION

The compound 1-hydroxyethane 1,1-disphosphonic acid, better known as HEDP, is an organophosphonate chelating agent that is used to stabilize peracetic acid (PAA) formulations. HEDP is effective at binding metal ions that might be present in PAA formulations, thus greatly increasing the shelf-life of the mixture. Since HEDP is a necessary component for PAA formulations, it is prudent to have a validated method for quantifying its concentration. This report will focus on the validation of the Palintest® Organophosphonate drop test kit for HEDP quantification.

II. EXPERIMENTAL METHODS

A Palintest organophosphonate drop test kit (part no. AD-1582 Batch: 6064) was obtained. This test kit consisted of sample tubes, organophosphonate tablets, and an organophosphonate solution in a dropper bottle. This solution would act as the titrant, and from this experiment the concentration (mg/L) of HEDP per drop of titrant was found.

A sample of HEDP lot #4M483 was obtained. According to the certificate of analysis, the lot was 59.1% HEDP by mass with a specific gravity of 1.443. Using an electronic pipette, 1170 µL of HEDP was transferred to a 1.0L Class A volumetric flask and diluted to the mark with reverse osmosis water. This represented a 1000 mg/L (ppm) stock solution that would be used to create all further dilutions in the experiment.

For the validation study, solutions containing 0 mg/L, 2 mg/L, 5 mg/L, 10 mg/L, 15 mg/L, and 20 mg/L were made. The 0 mg/L solution contained reverse osmosis (RO) water only and would be used as a blank, or background, for the study. The other sample solutions were made from the 1000 mg/L stock solution by pipetting 2.00mL, 5.00mL, 10.00mL, 15.00mL, and 20.00mL respectively into separate 1.0L volumetric flasks. Once thoroughly mixed, 20.00 mL of each sample was transferred by electronic pipette to a sample tube. Two organophosphonate tablets were added for activation of the analyte HEDP. These solutions were titrated dropwise and number of drops were recorded. The entire process was repeated in triplicate and the results were averaged and reported herein.

The data was consolidated and analyzed using regression analysis to yield a theoretical mg/L (ppm) HEDP per drop of titrant.

III. RESULTS

The results of the each trial are displayed in Table 1.

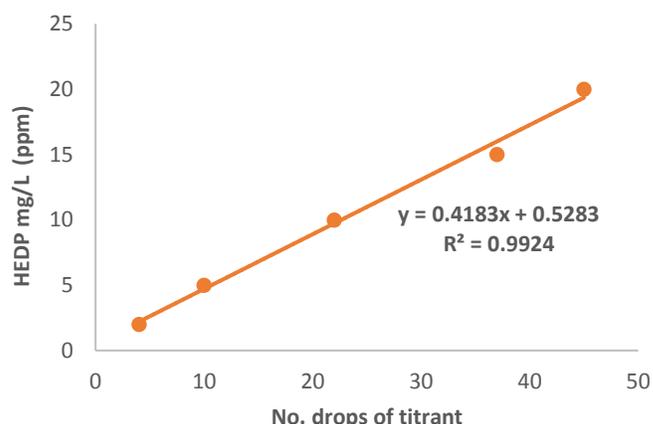
Table 1. Correction of HEDP samples using a blank.

HEDP sample mg/L (ppm)	No. drops of titrant	Corrected no. drops of titrant
0 (blank)	4	0
2	8	4
5	14	10

10	26	22
15	41	37
20	49	45

As seen in Table 1, titrating of the RO-only solution yielded a drop count of 4. Because of this, the total number of drops of titrant recorded for each sample was subtracted by 4 before a regression was calculated. These values can be found in the corrected column of Table 1 and were plotted against each sample HEDP mg/L to generate Figure 1.

Figure 1. No. drops of titrant vs. HEDP mg/L (ppm).



Using this data, a linear regression was calculated and is displayed in Fig. 1. The regression showed high linearity ($R^2 = 0.9924$) and therefore could be confidently used to validate the drop test kit.

Since the slope of the regression is HEDP (mg/L) over number of drops of titrant, this value can be used as the average mg/L (ppm) of HEDP per drop of titrant. This value, as shown in Fig. 1, was calculated as 0.4183 mg/L per drop. Uncertainty in the slope of the regression was found using regression analysis to be 0.02 mg/L. These values combined with the correction value found in Table 1 can be combined to produce the following formula:

$$\text{mg/L HEDP} = (\text{No. drops titrant} - 4) \times 0.42 \quad (1)$$

Equation 1 can be used to approximate mg/L HEDP in a given solution based on the drops of titrant required.

IV. DISCUSSION AND CONCLUSION

The Palintest organophosphonate drop test kit was validated by making several known dilutions of HEDP and titrating dropwise. After regression analysis, a value of 0.42 ± 0.02 mg/L per drop of titrant was calculated. From this calculation, equation 1 was derived and can be used in future titrations of HEDP-containing solutions, such as diluted peracetic acid solutions.