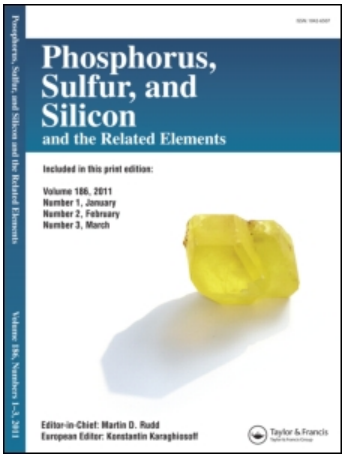

Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

Determination of Dexamethasone Sodium Phosphate in Medicament by Fluorescence Probe of Tb^{3+}-Tiron Complex

Chaobiao Huang[a]; Zhenrong Yu[b]; Libin Zhou[a]
[a] College of Chemistry and Life Science, Zhejiang Normal University, Jinhua, P. R. China [b] Deqing No. 3 High School, Zhejiang, P. R. China

To cite this Article Huang, Chaobiao , Yu, Zhenrong and Zhou, Libin(2008) 'Determination of Dexamethasone Sodium Phosphate in Medicament by Fluorescence Probe of Tb^{3+}-Tiron Complex', Phosphorus, Sulfur, and Silicon and the Related Elements, 183: 2, 730 — 734
To link to this Article: DOI: 10.1080/10426500701807699
URL: http://dx.doi.org/10.1080/10426500701807699

Phosphorus, Sulfur, and Silicon, 183:730–734, 2008

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500701807699

Taylor & Francis
Taylor & Francis Group

Determination of Dexamethasone Sodium Phosphate in Medicament by Fluorescence Probe of Tb^{3+}-Tiron Complex

Chaobiao Huang,[1] Zhenrong Yu,[2] and Libin Zhou[1]
College of Chemistry and Life Science, Zhejiang Normal University, Jinhua, P. R. China
[2]Deqing No. 3 High School, Zhejiang, P. R. China

A sensitive method by fluorescence quenching for the determination of dexamethasone sodium phosphate is proposed. The method is based on the ability of phosphate to inhibit the formation of a strong fluorescent complex of Tb^{3+} with tiron. The fluorescence intensity trends equilibrium after a 20-minutes reaction of dexamethasone sodium phosphate and Tb^{3+}-tiron complex. The optimal conditions for the determination of dexamethasone sodium phosphate are studied. Under optimal conditions, the linear range is 2.5×10^{-7}–4×10^{-6} mol/L and the detection limit is 6.2×10^{-8} mol/L, and the relative size distribution (RSD) of 11 times determination are 2.2% and 0.8% for 5×10^{-7} mol/L and 1×10^{-6} mol/L, respectively. The developed method is successfully applied to assay the dexamethasone sodium phosphate content in pharmaceutical injections.

Keywords Dexamethasone sodium phosphate; fluorescence quenching; medicament; Tb^{3+}-tiron complex

INTRODUCTION

The major composition of dexamethasone sodium phosphate injections is dexamethasone sodium phosphate, a synthetic fluorinated glucocorticoid with high glucocorticoid potency.[1] Dexamethasone sodium phosphate is a potent glucocorticoid used to treat inflammation colonopathy.[2] Normally, analytical methods for the determination of dexamethasone include ultraviolet-visible (UV-Vis) spectrophotometry and high performance liquid chromatography (HPLC). Owing to bad stability and repeatability of UV-Vis spectrophotometry, HPLC is used more regularly for well repeatability and high performance.[3,4] This

The authors' thank the Education Bureau of Zhejiang Province for funding this work and Zhejiang Normal University for providing funds for a visiting scholarship.

Address correspondence to Chaobiao Huang, College of Chemistry and Life Science, Zhejiang Normal University, Jinhua, 321004, P. R. China. E-mail: hcb@zjnu.cn

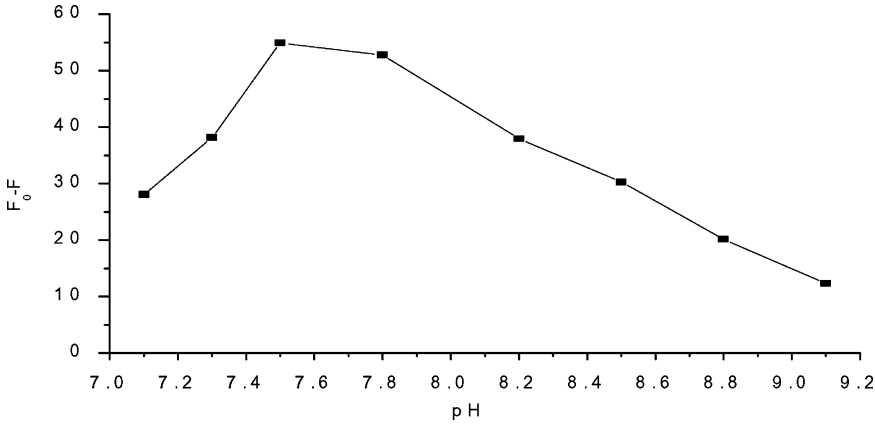

FIGURE 1 The influence of pH on the fluorescence of Tb^{3+}-tiron complex and quenching efficiency of dexamethasone sodium phosphate. (2.0 μmol/L Tb^{3+}, 2.5 μmol/L tiron, and 1.0 μmol/L dexamethasone sodium phosphate).

article describes a novel fluorescence quenching method of Tb^{3+}-tiron complex with high intensity, widely linear range, and easy manipulation.

RESULTS AND DISCUSSION

Influence of Buffer System and pH

The influence of buffer system and pH on the fluorescence of Tb^{3+}-tiron complex and the quenching efficiency of dexamethasone sodium phosphate are studied. When the HAc-NaAc (pH = 3.7∼5.9) buffer system and $Na_2B_4O_7$-NaOH (pH = 9.2∼10.8) buffer system are used, the fluorescence of Tb^{3+}-tiron complex is more weak than the Tris-HCl (pH = 7.1∼9.1) buffer system is used. And the quenching efficiency of dexamethasone sodium phosphate is more adequate. The variety of fluorescence to pH is shown in Figure 1. The Tris-HCl (0.01 mol/L, pH = 7.5) buffer system was chosen.

Influence of Other Conditions

The molar ratio of Tb^{3+}/tiron and reaction times of quenching also are studied. Customarily, Tb^{3+} and tiron form complex with 1:1 ratio,[5,6] and when the molar ratio of Tb^{3+}/tiron comes to 1:1.0∼1.5, the fluorescence can be quenched sufficiently by dexamethasone sodium phosphate. The fluorescence intensity of the system trends equilibrium after a

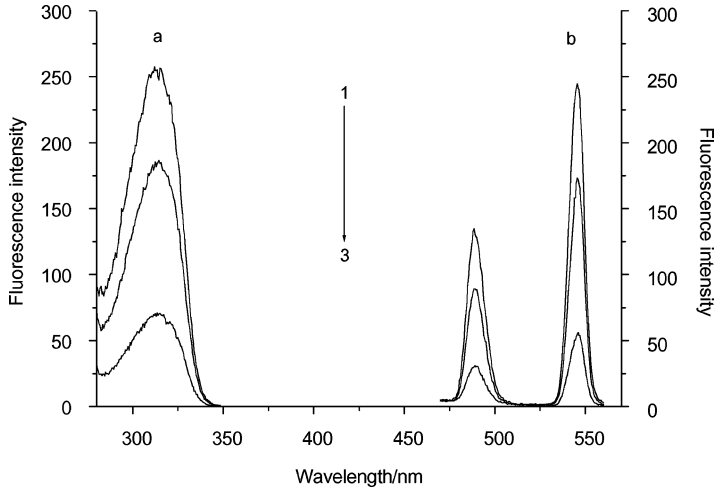

FIGURE 2 (a) Excitation and (b) emission spectra of Tb^{3+}-tiron complex under optimal condition. (1) 2.0 μmol/L Tb^{3+}, 2.5 μmol/L tiron, and 0 μmol/L dexamethasone sodium phosphate; (2) 2.0 μmol/L Tb^{3+}, 2.5 μmol/L tiron, and 1.5 μmol/L dexamethasone sodium phosphate; and (3) 2.0 μmol/L Tb^{3+}, 2.5 μmol/L tiron, and 3.0 μmol/L dexamethasone sodium phosphate.

20-minutes reaction. Dexamethasone sodium phosphate is determined under optimal condition of 2.0×10^{-6} mol/L $Tb^{3+}$and 2.5×10^{-6} mol/L tiron (pH 7.5 in 0.01 mol/L Tris-HCl buffer), with a 20-minutes reaction. Under optimal conditions, the excitation and emission spectrum of Tb^{3+}-tiron complex are shown as in Figure 2.

Analytical Performance

Under optimal conditions, the linear range of the determination is 2.5×10^{-7}–4×10^{-6} mol/L with correlation coefficient 0.9987, and the detection limit is 6.2×10^{-8} mol/L, and the RSD of 11 times determination are 2.2% and 0.8% for 5×10^{-7} mol/L and 1×10^{-6} mol/L, respectively. The quenching of the fluorescence emission of Tb^{3+}-tiron complex with addition of dexamethasone sodium phosphate is shown in Figure 3.

Application

The developed method is applied to determine dexamethasone sodium phosphate injections. Two batches of dexamethasone sodium phosphate injections are analyzed. Table I lists the results obtained with the present method. The results are in good agreement with those of

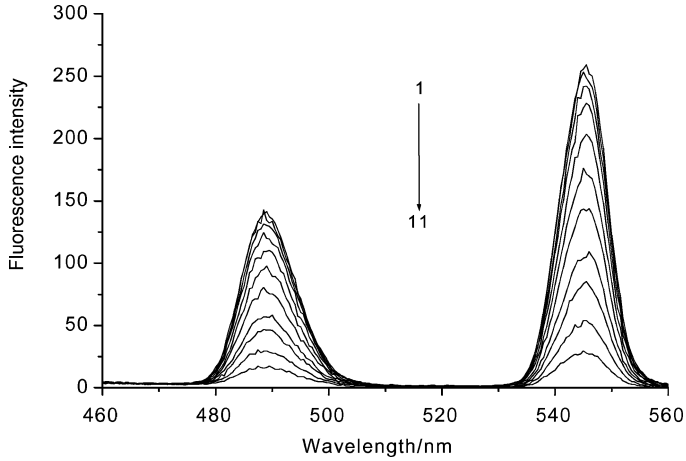

FIGURE 3 Fluorescence emission spectra of Tb^{3+}-tiron complex (Tb^{3+}: 2.0 μmol/L, tiron: 2.5 μmol/L) in the presence of dexamethasone sodium phosphate at various concentrations (μmol/L, pH = 7.5): 0, 0.25, 0.50, 0.75, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0.

labeled. The accuracy is validated by spike-recovery test with the solutions prepared from a batch of dexamethasone sodium phosphate injection, $(1.949 \pm 0.026) \times 10^{-5}$ mol/L (mean $\pm$ Standard Deviation [S.D.], n = 5) are found with the sample solution containing 9.535×10^{-6} mol/L analyte and 1.000×10^{-5} mol/L standards spiked. The recovery is 99.6 $\pm$ 2.4%.

EXPERIMENT

Apparatus

A Model LS-55 spectrofluorimeter (Perkin Elmer, USA) equipped with a Xenon lamp and a Model PHS-4 intellectual pH meter (Jiangsu Electroanalytical Instrument Factory, China) are used.

TABLE I Determination of Dexamethasone Sodium Phosphate in Pharmaceutical Preparations

Sample	Labeled[a]	Found[a] (mean $\pm$ S.D., n = 5)
I	2	2.07 $\pm$ 0.03
II	5	4.93 $\pm$ 0.06

[a]Expressed in mg per ml.

Reagent

Dexamethasone sodium phosphate standard is purchased from Sigma, Tb_2O_3, and Tiron (Sinopharm Group Chemical Reagent Co. Ltd) are analytical reagent. All chemicals used are analytical grade or better and doubly distilled water is used throughout the experiment.

Operation

2.0 ml Tris-HCl (0.1 mol/L, pH = 7.5) buffer, Tb^{3+} and tiron standard solution are added into 10 ml color comparison tube and brought to a predetermined volume with doubly distilled water. The fluorescence intensity of Tb in 545 nm is determined as F_0. The procedure above is followed, and dexamethasone sodium phosphate solution is also added. The fluorescence intensity of Tb with phosphate in 545 nm is determined as F. The transformation of fluorescence intensity $\triangle F = F_0 - F$ is defined as expression in determining of phosphate.[6] The excitation wavelength is 310 nm, the slit of excitation and emission are both 5 nm.

Sample Preparation

Dexamethasone sodium phosphate injections are diluted to 1×10^{-5} mol/L with doubly distilled water by stepwise dilution.

REFERENCES

[1] Editorial Committee of Pharmacopoeia of People's Republic of China, *Pharmacopoeia of People's Republic of China (Part II),* (Chemical Industry Press, Beijing, 2005). pp. 185–186.
[2] Y. N. Pang, Y. Zhang, Z. R. Zhang, T. L. Li, and Q. J. Pang, *Chin. Hosp. Pharm. J.*, **23,** 257 (2003).
[3] X. Y. Bi, L. P. Li, L. Li, and W. B. Yu, *Pharm. J. Chin. PLA.*, **22,** 235 (2006).
[4] Y. W. Yang, Y. Zhu, W. Q. Zhang, and R. Yuan, *J. Environ. Health.*, **23,** 264, (2006).
[5] Y. B. Zhao, D. Y. Wang, X. Q. Guo, and J. G. Xv, *Chemical Journal of Chinese Universities*, **18,** 691, (1997).
[6] Y. B. Zhao, D. Y. Wang, and L. F. Shi, *Chinese Journal of Analytical Chemistry*, **32,** 335, (2004).