

# A study of the preparation and reactivity of potassium ferrate

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## Abstract

In the context of water treatment, the ferrate ( $[\text{FeO}_4]^{2-}$ ) ion has long been known for its strong oxidizing power and for producing a coagulant from its reduced form (i.e. Fe(III)). However, it has not been studied extensively owing to difficulties with its preparation and its instability in water. This paper describes an improved procedure for preparing solid phase potassium ferrate of high purity (99%) and with a high yield (50–70%). The characteristics of solid potassium ferrate were investigated and from XRD spectra it was found that samples of the solid have a tetrahedral structure with a space group of  $D_{2h}$  (Pnma) and  $a = 7.705 \text{ \AA}$ ,  $b = 5.863 \text{ \AA}$ , and  $c = 10.36 \text{ \AA}$ . The aqueous stability of potassium ferrate at various pH values and different concentrations was investigated. It was found that potassium ferrate solution had a maximum stability at pH 9–10 and that ferrate solution at low concentration (0.25 mM) was more stable than at high concentration (0.51 mM). The aqueous reaction of ferrate with bisphenol A (BPA), a known endocrine disrupter compound, was also investigated with a molar ratio of Fe(VI):BPA in the range of 1:1–5:1. The optimal pH for BPA degradation was 9.4, and at this pH and a Fe(VI):BPA molar ratio of 5:1, approximately 90% of the BPA was degraded after 60 s.

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## 1. Introduction

The problems of water pollution continue to be of concern and as a consequence the regulated standards for drinking water supply and wastewater discharge are becoming more stringent. Hence there is a continuing interest in the application of more efficient water treatment chemical reagents to achieve higher water quality. Fe(VI) in the form of potassium ferrate ( $\text{K}_2\text{FeO}_4$ ) has been found to be a powerful oxidant over

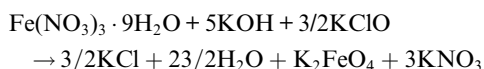
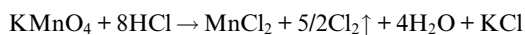
a wide pH range and many studies have considered its role as an oxidant in water and wastewater treatment (Jiang and Lloyd, 2002). During the aqueous oxidation reaction, Fe(VI) is reduced to Fe(III) or an insoluble end-product,  $(\text{Fe}[\text{OH}]_3)$  (Sharma, 2002). The standard half-cell reduction potential of ferrate has been estimated to be from +2.20 V to +0.72 V in acidic and basic solution, respectively (cf. +2.07 V and +1.24 V for ozone) (Wood, 1958). Thus the oxidation potential of potassium ferrate in acidic solution is the strongest of all the oxidants/disinfectants used in water and wastewater treatment, including chlorine, hypochlorite, chlorine dioxide, ozone, hydrogen peroxide, dissolved oxygen, and permanganate (Jiang and Lloyd, 2002). In addition, the ferric hydrolysis species including insoluble ferric

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hydroxide formed during ferrate oxidation are conventional coagulants, which mean that ferrate is a dual-function chemical reagent which has the potential to perform both oxidation and coagulation in a single treatment step (Deluca et al., 1992). The expected benefits of this combined effect in advanced water and wastewater treatment are higher water quality (particularly a lower risk of producing hazardous chemical byproducts), and lower operational and capital costs. Previous investigations have considered aspects of Fe(VI) treatment such as the inactivation of micro-organisms (Kazama, 1995), its reactivity with a wide range of aqueous contaminants, such as ammonia and heavy metals (Murrmann and Robinson, 1974) and the treatment of industrial and municipal wastewaters (Delaude and Laszlo, 1996; Johnson and Read, 1996; Norcross et al., 1997; Read et al., 1998; Sharma et al., 1999). Recently we reported on the degradation of phenol and chlorophenols by potassium ferrate (Graham et al., 2004). Owing to problems with the stability of the concentrated ferrate chemical, it is not available commercially and previous studies have required the ferrate to be freshly prepared in the laboratory. Currently, there is a need for further studies concerned with improving the method of ferrate preparation, such as increasing the stability and yield, and the reactivity of ferrate with contaminants of concern.

In general, there are three methods (Bielski and Thomas, 1987) for the synthesis of Fe(VI): (1) the electrochemical method by which anodic oxidation uses iron or alloy as the anode and NaOH/KOH as the electrolyte. The production yield is revealed to be strongly dependent on the electrolyte temperature and current density; (2) the dry method by which various iron-oxide-containing minerals are heated or melted under strong alkaline conditions and with oxygen flow. This method proves to be quite dangerous and difficult, since the synthesis process could cause detonation at elevated temperatures; (3) the wet method by which a Fe(III) salt is oxidized under strong alkaline conditions and either hypochlorite or chlorine is used as an oxidant. The third approach is widely considered to be the most practical. In 1951, Thompson et al. described the preparation of potassium ferrate by the wet method. In this case, potassium ferrate was prepared according to the following scheme:



This method of preparation gave a purity of only about 70% and a 10–15% yield.

Bisphenol A (BPA) is a commonly used name for 2,2-(4,4-dihydroxydiphenyl) propane. The structure of BPA is obtained from 2 mol of phenol with one mol of acetone. It is manufactured in high quantities, 90% or more being used as a monomer for polyester–styrene resins and flame retardants (Stoyanova et al., 2003). It is also generated during the decomposition of polycarbonate flasks. It is well known that BPA has an estrogenic activity that serves as an endocrine disrupting chemical (EDC) and causes an abnormal increase in the growth of yeast cells in polycarbonate flasks. Moreover, BPA contributes significantly to the general environmental problem of EDCs as evidenced by a number of recent studies on the effects of BPA in the “low dose” range, including its effects even at a ppt level (Inoue et al., 2003). Thus the degradation of BPA and its removal from the environment are of public concern. However, as far as is known, little has been reported on the aqueous degradation of BPA and EDCs in general by potassium ferrate. In the present study, an improved method of preparing solid phase potassium ferrate is proposed which gives a product of very high purity (99%) and a much greater yield (50–70%). The characteristics of the solid potassium ferrate were analyzed by X-ray diffraction (XRD) spectroscopy. In addition, we report the results of laboratory experiments involving the degradation of BPA by potassium ferrate in which the molar ratio of Fe(VI):BPA was in the range of 1:1–5:1.

## 2. Materials and methods

### 2.1. Chemicals

$\text{K}_2\text{FeO}_4$  was prepared with high purity (99%) by the oxidation of ferric nitrate with hypochlorite. The main chemicals used were ferric nitrate (from Acros), potassium hydroxide (from BDH), potassium permanganate (from Sigma Aldrich), and bisphenol A (from Aldrich). These chemicals were used without any further purification. The solutions were prepared with water that had been distilled and then passed through an 18 M $\Omega$  Milli-Q water purification system.

### 2.2. Experimental procedures

#### 2.2.1. Potassium ferrate preparation

One hundred and sixty five millilitres of 37% HCl was slowly reacted with 26.7 g of  $\text{KMnO}_4$  to produce chlorine. The chlorine was subsequently added to pre-chilled KOH solution (60 g of KOH in 100 ml of water) with stirring for over 2 h. A quantity of 90 g of KOH was then added into this solution and the resulting suspension was cooled. The precipitate of KCl was removed from the suspension by filtration using a GF/C filter paper, leaving a concentrated and strongly alkaline

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