Synthesis of $K_3[Fe(C_2O_4)_3] \cdot H_2O$

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§1 Reaction Theory

$Fe^{2+} \xrightarrow{C_2O_4^{2-}} FeC_2O_4 \cdot H_2O$			
Reagent	Moles	Mass	
iron(II) ammonium sulphate	0.0127 mol	5 g	
oxalic acid dihydrate	0.0396 mol	5 g	

$$FeC_2O_4 \cdot H_2O \xrightarrow[K_2C_2O_4 \cdot H_2O]{} Fe(OH)_3 + K_3Fe(C_2O_4)_3$$

Reagent	Moles	Mass or Volume
hydrogen peroxide	0.0179 mol	10 cm^3
potassium oxalate monohydrate	0.0271 mol	$3.5~\mathrm{g}$

§ 2 Experimental Method

$\S 2.1 \quad [Fe(C_2O_4)] \cdot 2H_2O$

Oxalic acid dihydrate (5 g) was dissolved in deionised water (50 cm³). Iron(II) ammonium sulphate hexahydrate (5 g) was dissolved in warm deionised water (20 cm³) and then acidified with dilute sulphuric acid (2 M, 1 cm³). The mixture was stirred rapidly. The oxalic acid solution (25 cm³) was added and heated to boiling. The mixture was allowed to settle and then decanted, it was mixed with hot deionised water (15 cm³) before being decanted again. The product was collected via Büchner filtration and washed with hot deionised water followed by acetone before being dried (2.40 g).

$\S~2.~2~K_3[Fe(C_2O_4)_3]\cdot H_2O$

The iron(II) oxalate from §2.1 was suspended in a warm solution of potassium oxalate monohydrate (3.5 g) in deionised water (10cm³). Hydrogen Peroxide was added dropwise (1.786 M, 10 cm³). The mixture was heated to boiling whilst slowly adding the oxalic acid solution (8 cm³). A further 3 cm³ of the solution was added slowly. The solution was filtered through fluted filter paper and methylated spirits (10 cm³) were added. The solution was allowed to cool and crysatalise before being collected through a Büchner filter and washed with a 1:1 methylated spirits:deionised water solution and acetone. The final product was dried in a vacuum desiccator in the dark (1.80 g).

§ 3 Interpretation

Question Nº1. What is the purpose of the H_2O_2 in the preparation of $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$

The ${\rm H_2O_2}$ oxidises the Fe(II) in the iron(II) oxalate to Fe(III), thus converting it to iron(III) oxalate.

Question Nº2. In the $(C_2O_4)^{2-}$ anion are all four C-O interatomic distances equal or are two shorter than the other two?

As the oxalate anion undergoes resonance the bond lengths will be an average of both the C=O double bond and the C-O single bond, thus every bond is the same length.