The use of ammonium succinate in protein crystallography. By HUGO L. MONACO, Department of Genetics, University of Pavia, via Abbiategrasso 207, 27100 Pavia, Italy

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Abstract

Ammonium succinate has been found to be a useful substitute for ammonium sulfate in protein crystallography.

Ammonium sulfate is one of the precipitating agents most often used in protein crystal growth because it has many advantages that are very well known (McPherson, 1982; Ducruix & Giegé, 1992). However, when preparing heavy-atom derivatives or when using other ligands of biochemical interest, it is sometimes necessary to get rid of either one or both ions, which can be incompatible with many compounds that one would like to have in the crystal mother liquor (Blundell & Johnson, 1976; Petsko, 1985). Sodium sulfate, although less soluble than ammonium sulfate, offers a valid alternative if the source of trouble is the ammonium ions. We have found that a useful alternative when the problem is the sulfate ions is ammonium succinate. A typical example is the preparation of a lead derivative of a protein crystal grown in the presence of sulfate ions. Ammonium succinate is commercially available with a high degree of purity and is soluble in water to concentrations analogous to those that can be reached by ammonium sulfate. We have found that, if one replaces an ammonium sulfate solution by an ammonium succinate solution of equal or slightly higher ionic strength, it is often possible to transfer crystals grown in the first mother liquor to the second with slight or negligible loss of isomorphism. The compound can also be used as a standard precipitant to grow crystals ex novo. A list of proteins in which this compound has proven to be useful in our laboratory includes: chicken liver (basic) fatty-acid-binding protein, hen egg-white riboflavin-binding protein, human transthyretin, the complex bovine retinol-binding protein–transthyretin and pig kidney d-amino acid oxidase.

References


Laboratory Notes

A high-temperature Guinier camera and its application to the decomposition of Co$_1$$_4$S$_4$ (jaipurite). By K. BENTE,* K. HANKE, K. MARIOLACOS, H. BARTELS and E. HENSEL, Mineralogisch-Kristallographisches Institut der Universität Göttingen, Göttingen, Germany

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In memoriam V. Kupčík

Abstract

A high-temperature Guinier camera for closed and open systems used for temperatures up to 1170 K is described. As an exemplary application, the decay of Co$_1$$_4$S$_4$ (jaipurite) to Co$_3$S$_4$ (linnaeite) and Co$_9$S$_8$ (Co-pentlandite) at 663 and 723 K under its own vapour pressure is presented.

Introduction

X-ray diffraction determinations of compounds with high fugacities such as oxides and sulfides require special apparatus for in situ high-temperature studies. Such studies are carried out for sulfides of transition metals and elements of the fourth and fifth groups of the Periodic Table. Since the sulfur vapour pressures of these phases at elevated temperatures often lead to decomposition and non-stoichiometric composition under open conditions, a device prohibiting sulfur losses or controlling the sulfur fugacity was required. Therefore, a high-temperature in situ Guinier camera for closed systems was developed, which also may be used under open conditions.

Description

The main features of our Guinier camera are the arrangement of a conventional Cu X-ray source, a graphite monochromator, a special high-temperature chamber and a film camera (diameter 114.6 mm). These items are combined to result in Guinier geometry in the subtraction position. The main problem was to construct a high-temperature chamber suitable for both open and closed sample containers. The problem for the closed operation was solved using a parallel arrangement of up to five silica capillaries with a diameter of 0.1 to 0.15 mm substituting the usual flat Guinier probe arrangement. These capillaries are sealed under vacuum and fixed in parallel in a metal tube. For open conditions, the capillaries remain open. The metal tube, with an outer diameter of 22 mm and an inner diameter of 12 mm, is introduced into a silicate carrier with a glide gear which can be turned during diffraction by...