Preparation and Crystal Structure of Tripotassium Sodium Disulfate

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The title compound, K₃Na(SO₄)₂, was isolated, and the crystal structure was determined by the single-crystal X-ray diffraction method at 90 K. It crystallizes in the trigonal space group P₃m1 with \( a = 5.6346(8) \text{Å}, \ c = 7.2543(15) \text{Å}, \ V = 199.46(6) \text{Å}^3, \ D_x = 2.767 \text{g/cm}^3, \) and \( Z = 1. \) The \( R_1 \) [\( I > 2\sigma(I) \)] and \( wR_2 \) (all data) values are 0.0244 and 0.0653, respectively, for all 217 independent reflections. The crystal consists of six-coordinated sodium ions and ten-coordinated and twelve-coordinated potassium ions, which are connected by sulfate ions.

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The chemistry of inorganic salts has attracted much attention for a long period, because these compounds can be considered to be useful inorganic materials for a constituent material of detergent; some of these have a solubility property for other constituent substances, such as sodium dodesylate.\(^1,2\) Mixed salts of sodium and potassium ions are interesting systems to make such compounds, because they have two cation-sites versus to sulfate ions. In this study, we isolated a mixed salt from a reaction of sodium sulfate and potassium carbonate, and determined the crystal structure of this salt, which is a typical example of the coexistence of sodium and potassium ions with sulfate ions, as shown in Fig. 1.

Sodium sulfate (1.00 g) was dissolved in 9 cm\(^3\) of distilled water. This solution was placed at the bottom of a dish, and an aqueous solution (9 cm\(^3\)) of potassium carbonate (1.00 g) was layered. After several hours, crystals appeared, which were collected.

X-ray diffraction data for one of these crystals were collected at 90 K on a Bruker CCD X-ray diffractometer (SMART APEX) using graphite-monochromated Mo-\( K\alpha \) radiation. Crystal data and details concerning data collection are given in Table 1. The structure was solved by direct methods and refined by full-matrix least-squares methods. All of the calculations were carried out on a Pentium IV Windows 2000 computer utilizing the SHELXTL software package. Crystallographic data have been deposited with Cambridge Crystallographic Data Centre: Deposit number CCDC-872654. Copies of the data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge, CB2 1EZ, UK; Fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

A crystal-structure analysis revealed that the present crystals are K₃Na(SO₄)₂, shown in Figs. 1 and 4S, which are known as glaserite.\(^3,4\) The asymmetric unit contains one twelfth of the K₃Na(SO₄)₂ unit. The coordination geometries around the metal ions are shown in Fig. 4.

Table 1 Crystal and experimental data

| Chemical formula: K₃Na(SO₄)₂ | Formula weight = 332.41 |
| Crystal system: trigonal | Space group: P₃m1 |
| \( a = 5.6346(8) \text{Å} \) | \( c = 7.2543(15) \text{Å} \) |
| \( V = 199.46(6) \text{Å}^3 \) | \( D_x = 2.767 \text{g/cm}^3 \) |
| \( Z = 1 \) | \( D_r \) max = 0.001 |
| \( D_r \) max = 0.365 eÅ\(^{-3} \) | \( D_r \) min = –0.474 eÅ\(^{-3} \) |

Fig. 1  Chemical structure of the title compound.

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ions, drawn by ORTEP, are shown in Fig. 2. Selected bond distances and angles are given in Table 2. Each sulfate ion is located on the crystallographic three-fold axis. The three oxygen atoms (O1, O1d, and O1q) of the sulfate ion are coordinated to potassium and sodium ions in a bidentate fashion. The other oxygen atom (O2) of the sulfate ion is located on the \( C_3 \) axis, and is coordinated to four potassium ions in a tetradentate fashion. Each sodium ion is coordinated by six sulfate-oxygen atoms to form an octahedron. The Na-O distances are 2.8314(4) Å and 3.0426(18) Å, while the K1-O distances are 2.8858(17) Å and 3.2612(5) Å. These values reflect the coordination numbers, and are also in the normal range as Shannon’s ionic radii for potassium compounds.\(^5\),\(^6\)

![ Packing diagram of the title compound viewed along the \( c \) axis.](image)

Fig. 3

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