The Crystal Structure of the Methanethiosulphonates of Divalent Sulphur, Selenium and Tellurium

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The synthesis and the unit cells and space group of these compounds were described by one of us recently. The crystals are isomorphous, with a four-molecule unit cell based on the space group \( O_{2h}^4 \) — \( P2_1/n \). The dimensions are:

\[
a, A \quad b, A \quad c, A \quad \beta
\]

\[
\begin{align*}
S(S_2O_3CH_3)_2 & = 11.33 \quad 5.21 \quad 16.14 \quad 91^\circ \\
Se(S_2O_3CH_3)_2 & = 11.38 \quad 5.23 \quad 16.23 \quad 91^\circ \\
Te(S_2O_3CH_3)_2 & = 11.43 \quad 5.29 \quad 16.32 \quad 91^\circ 
\end{align*}
\]

Weissenberg photographs were taken with CuK radiation on multiple films, and the intensities estimated visually.

A Patterson synthesis based on the \( h0l \) data for the tellurium compound indicated four possible tellurium positions. A Fourier map for one of the positions, using signs of the reflections calculated from the tellurium contributions alone, gave a clear resolution of the sulphur atoms. Inclusion of the calculated structure factors from these atoms changed the sign of 13\% of the reflections, and a second two-dimensional Fourier analysis was made, with the resulting tellurium and sulphur parameters:

\[
\begin{align*}
S_I & \quad S_{II} & \quad Te & \quad S_{IV} & \quad S_V \\
x & = 0.133 & \quad 0.123 & \quad 0.234 & \quad 0.422 & \quad 0.436 \\
z & = 0.117 & \quad -0.017 & \quad -0.068 & \quad -0.054 & \quad -0.163 
\end{align*}
\]

A Fourier analysis based on the \( h0l \) data for the sulphur compound was subsequently carried out, using signs of the reflections obtained from the tellurium compound revised by subtracting two thirds of the calculated tellurium structure factors. The five sulphur atoms were clearly resolved, and after three successive


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The Fluorometric Measurement of 4-Pyridoxic Acid in Normal Urine

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Huff and Perlzweig have isolated and identified the excretion product 4-pyridoxic acid (2-methyl-3-hydroxy-4-carboxy-5-hydroxymethylpyridine), which is supposed to be the main excretion product of vitamin B_{	ext{E}}. In the same work they described a fluorometric method for quantitative determination of 4-pyridoxic acid in urine. The fluorometric determination is based on the fact that the lactone form of 4-pyridoxic acid gives a fluorescent intensity 25 times that of the free acid, which in itself is highly fluorescent. In human urine the product occurs in the free acid form. By heating the urine in acid solution this compound is converted into its lactone form. The blank is an untreated urine sample. With this method it has been found that the average 24 hour urinary excretion in man is from 3 to 4 mg.

In a critical study of the Huff and Perlzweig method Sarrett has shown that the blank is not reliable in measurements of normal urine. Sarrett reports of unsuccessful attempts to separate the 4-pyridoxic acid from interfering substances by means of adsorption or precipitation.

Using the same method we have found that other substances than 4-pyridoxic acid present in the urine increase in fluorescence by treatment with acid. We have therefore used a blank prepared in a different way. Our technique is based on the reversion of the lactone into the free acid by heating the urine in alkaline solution.

The first step is to heat the urine with acid. By this treatment the free acid is converted into its lactone form and the fluorescence of this sample containing the


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