températures supérieures, la consommation augmente. À 50 K, elle atteint 0,41 litre de liquide par heure ce qui reste convenable et donne une autonomie d'environ quatre heures entre chaque remplissage, la capacité du réservoir étant d'environ 1,5 litres. La stabilité de la température dans ce domaine est très grande. Nous avons par exemple maintenu 10,00 ± 0,02 K pendant huit heures consécutives.

Conclusion
Nous nous sommes attachés à mettre au point un cryostat pour diffractométrie X, à température variable entre 300 et 10 K. La température est stable à mieux que 0,1 K quel que soit le point de fonctionnement. L'autonomie est toujours suffisante pour travailler dans de bonnes conditions.

Résidences

Crystal Data

Crystal data for the tetraphenylarsonium salt of the monoanion of theophylline,

$$\left[ (C_{6}H_{5})_{4}As \right] \left[ CTN_{4}O_{2}H_{2} \right] \cdot 5H_{2}O.$$  

By THOMAS J. KISTENMACHER and HENRY B. KERFOOT, Department of Chemistry, The Johns Hopkins University, Baltimore, Maryland 21218, U.S.A.

(Received 1 May 1975; accepted 23 July 1975)

The title compound crystallizes in the triclinic system with

\[ a = 13-13 (1), \quad b = 19-80 (2), \quad c = 12-916 (3) \text{ Å}, \quad \alpha = 90-01 (5), \quad \beta = 102-96 (6), \quad \gamma = 96-45 (7) \text{°}, \quad V = 3251-6 \text{ Å}^3. \]

Origin of specimens

Crystals of the tetraphenylarsonium salt of the monoanion of theophylline, 1,3-dimethyl-2,6-dioxopurine, were obtained by mixing an aqueous solution of equimolar amounts of theophylline and NaOH with an aqueous solution of hydrated tetraphenylarsonium chloride. Slow evaporation of the solvent yielded many clear crystals in the form of parallelepipeds after approximately three days. The crystalline product showed infrared bands attributable to Ph₄As⁺ and the theophylline monoanion. The crystals are air sensitive, and it was found necessary to coat them with a film of low-molecular-weight petroleum grease and seal them in thin-walled Lindemann capillaries.

Crystal data

Oscillation and Weissenberg photographs (h0l–h2l) gave no evidence of symmetry or systematically absent reflections. Unit-cell dimensions were obtained from a least-squares fit to the 2θ, ω and χ settings for 13 reflections measured on a Syntax P1 computer-controlled diffractometer. The crystal used had dimensions 0.3 x 0.4 x 0.5 mm \([100] \cdot [100], [001] \cdot [001], [010] \cdot [010], \) respectively. The density was measured by flotation methods in a mixture of cyclohexane and carbon tetrachloride. Complete crystal data are as follows:

\[ \left[ (C_{6}H_{5})_{4}As \right] \left[ CTN_{4}O_{2}H_{2} \right] \cdot 5H_{2}O \]

\[ a = 13-13 (1), \quad b = 19-80 (2), \quad c = 12-916 (3) \text{ Å}, \]

\[ \alpha = 90-01 (5), \quad \beta = 102-96 (6), \quad \gamma = 96-45 (7) \text{°}, \]

\[ V = 3251-6 \text{ Å}^3, \]

\[ Z = 4, \]

\[ D_{m} = 1-36 (1), \quad D_{c} = 1-37 \text{ g cm}^{-3}. \]

The cell reported is the Delauney reduced cell; it was searched by computer methods (Lawton & Jacobson, 1965) for higher symmetry, but no evidence for such was obtained.

Comparison with other results

We initiated our study of the tetraphenylarsonium salt of the monoanion of theophylline in an attempt to obtain accurate structural parameters for comparison with the coordinated theophylline monoanion [see Marzilli, Kistenmacher, Darcy, Szalda & Beer (1974) and references therein]. After consideration of the size of the problem, it was concluded that the anticipated results did not justify the investment. However, some general features of the crystal structure may be inferred from its similarity to the analogous Ph₄As⁺ salt of the monoanion of adenine (Kistenmacher, 1973). In the adenine monoanion structure, purine anions were found intercalated between the phenyl groups of two twofold screw-related tetraphenylarsonium cations. This feature may be retained in the theophylline anion structure.

This investigation was supported by the National Institutes of Health [Biomedical Sciences Support Grant and Public Health Service Grant No. GM 20544].

References