ACTA CHEMICA SCANDINAVICA 22 (1968) 1036–1061

Short Communications

Single Crystal Data for the Disodium Salt of Adenosine Triphosphate

M. ZEPPEZAUER, E. ZEPPEZAUER and C.-I. BRÄNDÉN

Agricultural College of Sweden, Department of Chemistry I, Uppsala 7, Sweden

Adenosine triphosphate, ATP, is a substance of considerable biochemical importance because of its function in storing and releasing chemical energy by means of its labile phosphate bond. Knowledge of the three-dimensional structure of this molecule would thus be of great biochemical importance. Unfortunately it seems to be extremely difficult to obtain single crystals of this compound suitable for a detailed structure analysis.

X-Ray powder diffraction studies of the disodium salt of ATP and its hydrates have been reported by Lomer.[1] He prepared microcrystals of this compound by the method of Berger,[2] that is by adding ethanol to an aqueous solution of Na_2ATP. He obtained three distinct X-ray powder patterns, A, B, and C, by varying the degree of hydration of the compound. The three patterns were all interpreted in terms of triclinic unit cells with small differences in the cell-dimensions.

We have now been able to obtain very thin needle-shaped single crystals of Na_2ATP which gave discrete X-ray reflections to a resolution of about 2 Å. These crystals were prepared by a modification of Bergers method using 2-methyl-2,4-pentanediol instead of ethanol. The crystals belong to an orthorhombic unit cell, space-group $P2_12_12_1$ and with the cell-dimensions given in Table 1. The space-group and approximate cell-dimensions were obtained from Weissenberg and precession photographs. Accurate values of the cell-dimensions were determined from powder photographs recorded in a camera of the Guinier-type and using silicon as an internal standard.

It was immediately apparent that our powder pattern and those reported by Lomer were very similar. We could easily index his reported powder patterns on the basis of the orthorhombic unit cell. Accurate values of the cell-dimensions of these four patterns listed in Table 1 were then obtained by a least squares refinement on the computer CD 3600 using a programme devised by P.-E. Werner.

The four patterns give almost the same length of two of the axes of the orthorhombic unit cell but there are significant differences in the third axis, the a-axis, of this cell. Pattern C which corresponds to the most hydrated specimen made by Lomer shows the longest a-axis of 31.30 Å. A regular decrease of the length of this axis is observed through the patterns B

Table 1. Cell-dimensions of the orthorhombic unit cells of Na_2ATP and its hydrates.

	Present modification	Pattern A	Pattern B	Pattern C
a (Å)	27.60 ± 0.09	29.13 ± 0.06	30.18 ± 0.11	31.30 ± 0.09
b (Å)	21.14 ± 0.06	20.78 ± 0.06	20.67 ± 0.06	21.14 ± 0.06
c (Å)	7.10 ± 0.02	7.06 ± 0.02	7.07 ± 0.02	7.10 ± 0.02

and A to our modification which has the shortest a-axis of 27.60 Å.

In order to establish the water content of our modification we made an analysis of the amount of carbon present in our sample. The observed value was 21.15 % ±0.15 and the calculated values for the anhydrous, monohydrate and dihydrate compounds are 21.80, 21.10, and 20.46, respectively.

We thus conclude that all four modifications belong to orthorhombic unit cells and that the modification studied by us corresponds to the monohydrate.

We wish to thank Professor I. Lindqvist for stimulating discussions and for the facilities placed at our disposal, and also Professor W. Kirsten at this Institute for making the carbon analysis. This work is supported by grant No. 2767—1 from the *Swedish Natural Science Research Council* and by grant No. B68-11X-37-04 from the *Swedish Medical Research Council*. Facilities for use of the computer CD 3600 were granted by the *Computing Division of the National Swedish Office for Administrative Rationalization and Economy*. These grants are gratefully acknowledged.

1. Lomer, T. R. *Acta Cryst.* **11** (1958) 108.
2. Berger, L. *Biochim. Biophys. Acta* **20** (1956) 27.

Received February 14, 1968.

trans-Dinitrotetrammine-cobalt(III) Acetate — Synthesis and Preliminary Crystal Data

THOMAS E. MacDERMOTT and BENTE SAUSTRUP KRISTENSEN

Chemistry Department A, The Technical University of Denmark, Lyngby, Denmark

We here report a direct synthesis of *trans*-dinitrotetramminecobalt(III) acetate in a highly crystalline, analytically pure form and in better than 80 % yield.

The ultra-violet-visible spectrum in aqueous solution shows the following maxima:

440 mμ (log ε = 4.20); 346 mμ (log ε = 3.61);
252 mμ (log ε = 4.20) and 193 mμ (log ε = 4.32).

This spectrum agrees well with that recorded by Carrassiti and Martelli.[1] The small deviations are attributable to the rapid aquation the compound undergoes in aqueous solution. Our spectrum was recorded within 10 min of commencing the dissolution, but a small amount of compound remained undissolved at that time.

The infra-red spectrum is useful to characterize the compound and the spectrum from 4000 to 400 cm^{-1} is shown in Fig. 1. This spectrum differs considerably from that for the chloride or iodide. These latter salts are readily prepared by treatment of the acetate in aqueous solution with sodium chloride and iodide, respectively. The spectrum of the chloride is identical with that given by Le Postollec, Mathieu and Poulet [2] for the *trans*-chloride. We conclude that Beattie and Tyrell [3] and also Blyholder and Kittila [4] have confused the isomers of this compound in their infra-red studies.

Synthesis. Cobalt(II) acetate 4-hydrate (105 g, 0.42 mole) is dissolved in a solution of sodium nitrite (105 g) in 25 % ammonia (500 ml), partly neutralised with 30 ml of 80 % acetic acid. The mixture becomes slightly warm and to the warm solution is added 28 ml of 30 % hydrogen peroxide as quickly as the effervescence will allow. The mixture is then heated on a hot plate just below its boiling point for 30 min. Much ammonia is given off and the product begins to separate from the hot solution. The heating is then stopped and the mixture cooled in ice and filtered under suction. When most of the mother liquid has been thus removed, the moist finely divided product is transferred to a beaker and stirred with 200 ml of acetone and again filtered. A second fraction which separates from the mother liquid is collected in the same way. These moist products are then transferred to a 4 l beaker and dissolved in 2.5—3 l 0.5 % acetic acid by heating to near the boiling point. The solution is allowed to cool overnight and the thin golden-yellow plates are filtered under suction, washed with ethanol and acetone and dried under the heat lamp. The yield is 81 g, 80 % based on the cobalt(II) acetate used. The compound requires no further purification.