X-ray powder diffraction data for monosodium salt azobarbituric acid dihydrate [$C_8H_5N_6O_6Na.2H_2O$]

J. Maixner,[1,a)] J. Rohlíček,[2] Č. Florián,[3] J. Vyňuchal,[3] and F. Alafid[4]
[1]*Central Laboratories, Institute of Chemical Technology Prague, Technická 5, 166 28 Prague 6, Czech Republic*
[2]*Institute of Physics AS CR,v.v.i., Na Slovance 2, 182 21 Prague 8, Czech Republic*
[3]*Synthesia a.s., Pardubice, Semtín 103, CZ-532 17 Pardubice, Czech Republic*
[4]*Faculty of Chemical Technology, Institute of Organic Chemistry and Technology, University of Pardubice, Studentská 95, 53210 Pardubice, Czech Republic*

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X-ray powder diffraction data, unit-cell parameters, and space group for a sodium azobarbituric acid dihydrate are presented [$a = 3.546$ (1) Å, $b = 9.210$ (2) Å, $c = 9.738$ (4) Å, $\alpha = 104.07$ (4)°, $\beta = 98.09$ (6)°, $\gamma = 98.80$ (2)°, unit-cell volume $V = 299.6$ Å^3, $Z = 1$, and space group $P-1$]. All the measured lines were indexed. No detectable impurities were observed. © *2014 International Centre for Diffraction Data.*
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Key words: X-ray powder diffraction, pigments, sodium azobarbituric acid dihydrate

I. INTRODUCTION

Sodium azobarbituric acid dihydrate [$C_8H_5N_6O_6Na.2H_2O$] (Figure 1) is an orange-red powder. It is used as the crucial starting material for the synthesis of the industrially important and is now worldwide discussed organic pigment – Pigment Yellow 150, which is the complex of nickel and azobarbituric acid. The pigment is used in paints and in decoration printing inks for laminates.

We have inspected the CSD database (Allen, 2002) and the PDF4+ database (ICDD, 2012) and have not found any entry for this compound in the mentioned databases. This fact is the reason why we have decided to characterize this compound by X-ray powder diffraction (XRD) technique.

II. SAMPLE PREPARATION

There is more than one way to synthesize monosodium salt azobarbituric acid, but it cannot be prepared by a common diazotization of aminobarbituric acid and the following coupling. The synthetic route using azidoformamidine as the diazo group transfer agent was chosen for the synthesis of azobarbituric acid according to the standard industrial procedure. This synthesis is one-pot reaction, in which azidoformamidine is formed from aminoguanidine and sodium nitrite in acid water media as the first step. In the second step, the diazobarbituric acid is formed and subsequently reacts with an excess of barbituric acid generating azobarbituric acid in the form of dihydrate of monosodium salt in the third step. The final product was dried under vacuum at 70 °C for the elimination of the possible decomposition. This monosodium salt is poorly soluble in water, and its solubility is in the range of tenth of a gram per liter. The thermogravimetric analysis was used to confirm crystal water in the structure of $C_8H_5N_6O_6Na.2H_2O$.

a)Author to whom correspondence should be addressed. Electronic mail: jaroslav.maixner@vscht.cz

III. POWDER DIFFRACTION DATA

The diffraction pattern for the title compound was collected at room temperature with an Empyrean powder diffractometer with transmission Debye–Scherrer geometry using Cu$K\alpha$ radiation (focusing mirror, generator setting: 45 kV, 40 mA). An ultrafast PIXCel^{3D} detector was employed to collect XRD data over the angular range from 4 to 80 °2θ with a step size of 0.013 °2θ, and a counting time of 2978,4 s step^{-1}. The sample was placed in the 0.3 mm borosilicate glass capillary. The experimental powder diffraction pattern is depicted in Figure 2. The software package HighScore Plus V3.0e (PANalytical, Almelo, Netherlands) was used to smoothen the data, to fit the background, and to eliminate the $K\alpha_2$ component; and the top of the smoothed peaks were used to determine the peak positions and intensities of the diffraction peaks (Table 1). The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

The collected data are consistent with a triclinic unit-cell parameters [$a = 3.546$ (1) Å, $b = 9.210$ (2) Å, $c = 9.738$ (4) Å, $\alpha = 104.07$ (4)°, $\beta = 98.09$ (6)°, $\gamma = 98.80$ (2)°, unit-cell volume $V = 299.6$ Å^3, $Z = 1$, and space group $P-1$]. These parameters were derived using DICVOL04 (Boultif and Louër, 2004) with the results all being within the errors indicated. The following figures of merit were achieved: $F_{20} = 45.6$ (0.0107,41) (Smith and Snyder, 1979) and $M_{20} = 26.2$ (de Wolff, 1968).

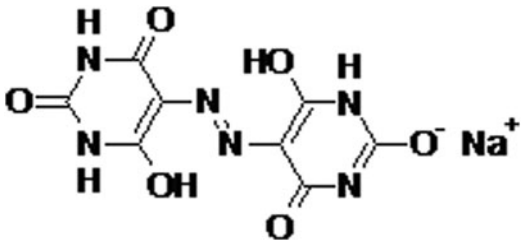

Figure 1. Structural formula of $C_8H_5N_6O_6Na.2H_2O$.

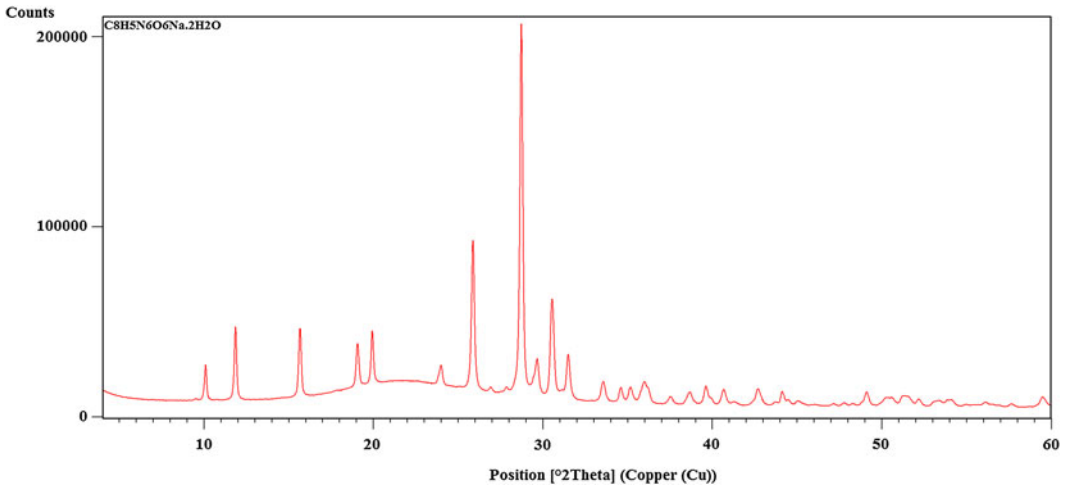

Figure 2. (Color online) X-ray powder diffraction pattern of $C_8H_5N_6O_6Na.2H_2O$ using Cu$K\alpha$ radiation (λ=1.5418 Å).

TABLE I. Indexed X-ray powder diffraction data for $C_8H_5N_6O_6Na.2H_2O$. Only the peaks with I_{rel} of 1 or greater are presented [$a = 3.546$ (1) Å, $b = 9.210$ (2) Å, $c = 9.738$ (4) Å, $\alpha = 104.07$ (4)°, $\beta = 98.09$ (6)°, $\gamma = 98.80$ (2)°, unit-cell volume $V = 299.6$ Å$^{3<>}$, $Z = 1$, and space group $P-1$]. All lines were indexed. The d-values were calculated using Cu$K\ \alpha_1$ radiation ($\lambda = 1.5406$ Å).

$2\theta_{obs}$ (deg)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (deg)	d_{calc} (Å)	$\Delta 2\theta$
9.511	9.291	1	0	0	1	9.529	9.273	−0.018
10.091	8.759	10	0	1	0	10.095	8.755	−0.004
11.858	7.457	20	0	1	−1	11.866	7.452	−0.008
15.667	5.652	18	0	1	1	15.670	5.651	−0.003
19.056	4.654	12	0	1	−2	19.059	4.653	−0.003
19.926	4.452	14	0	2	−1	19.925	4.452	0.001
23.851	3.728	3	0	2	−2	23.853	3.728	−0.002
23.976	3.709	5	0	1	2	23.993	3.706	−0.017
25.863	3.442	40	1	0	−1	25.858	3.443	0.005
26.902	3.312	1	1	−1	−1	26.892	3.313	0.010
27.848	3.201	2	0	1	−3	27.849	3.201	−0.001
28.721	3.106	100	1	1	−1	28.721	3.106	0.000
29.458	3.030	5	0	3	−1	29.457	3.030	0.001
29.653	3.010	10	1	1	0	29.691	3.006	−0.038
30.540	2.925	27	0	3	0	30.591	2.920	−0.051
31.488	2.839	12	1	−2	−1	31.469	2.841	0.019
33.561	2.668	6	1	1	1	33.529	2.671	0.032
34.590	2.591	4	1	2	−1	34.599	2.590	−0.009
35.171	2.550	4	1	0	2	35.192	2.548	−0.021
35.807	2.506	4	1	2	−2	35.768	2.508	0.039
35.967	2.495	6	1	−2	−2	35.944	2.496	0.023
36.222	2.478	4	1	−3	0	36.221	2.478	0.001
37.518	2.395	2	1	−1	−3	37.494	2.397	0.024
38.643	2.328	3	0	2	−4	38.663	2.327	−0.020
39.606	2.274	5	0	4	−1	39.610	2.274	−0.004
39.904	2.257	2	0	2	3	39.937	2.256	−0.033
40.661	2.217	4	0	3	2	40.682	2.216	−0.021
41.252	2.187	1	0	4	0	41.183	2.190	0.069
42.681	2.117	4	1	0	−4	42.731	2.114	−0.050
43.711	2.069	1	0	4	−3	43.669	2.071	0.042
44.117	2.051	4	1	−4	1	44.120	2.051	−0.003
44.445	2.037	2	1	−4	0	44.449	2.037	−0.004
45.029	2.012	1	0	4	1	44.988	2.013	0.041
45.299	2.000	1	1	3	−3	45.321	1.999	−0.022
46.008	1.971	1	1	−4	2	46.025	1.970	−0.017
47.142	1.926	1	1	1	3	47.154	1.926	−0.012
47.750	1.903	1	0	2	−5	47.730	1.904	0.020
48.246	1.885	1	0	3	3	48.259	1.884	−0.013
48.814	1.864	1	0	4	−4	48.815	1.864	−0.001
49.090	1.854	4	0	2	4	49.117	1.853	−0.027
49.925	1.825	1	1	3	−4	49.897	1.826	0.028
50.250	1.814	2	0	5	−1	50.270	1.814	−0.020
50.543	1.804	3	0	3	−5	50.594	1.803	−0.051
51.241	1.781	3	1	4	−2	51.186	1.783	0.055
51.372	1.777	3	1	0	−5	51.364	1.777	0.008
51.585	1.770	3	2	−1	−1	51.631	1.769	−0.046
52.152	1.752	2	0	5	0	52.158	1.752	−0.006
53.025	1.726	1	1	−5	1	52.987	1.727	0.038
53.356	1.716	2	2	−2	−1	53.335	1.716	0.021
53.844	1.701	2	1	−5	0	53.837	1.701	0.007
54.059	1.695	2	1	−5	2	54.101	1.694	−0.042
54.911	1.671	1	2	1	−1	54.889	1.671	0.022
55.563	1.653	1	0	4	−5	55.523	1.654	0.040
56.105	1.638	1	2	0	1	56.059	1.639	0.046
56.518	1.627	1	1	−5	−1	56.590	1.625	−0.072
57.635	1.598	1	2	1	−3	57.596	1.599	0.039
59.460	1.553	3	2	2	−2	59.466	1.553	−0.006
60.709	1.524	1	1	5	−2	60.782	1.523	−0.073
61.644	1.503	1	2	1	−4	61.646	1.503	−0.002
68.222	1.374	1	0	5	3	68.217	1.374	0.005
77.838	1.226	1	1	−6	−3	77.838	1.226	0.000

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