

X-ray powder diffraction data for Ammonium D-Gluconate,



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Abstract

The compound ammonium D-gluconate ($\text{C}_6\text{H}_{11}\text{O}_7^-\text{NH}_4^+$) has been studied by X-ray powder diffraction. The powder diffraction pattern and data obtained at room temperature are presented (cell data and powder data summary).

I. Introduction

Gluconic acid and its derivatives are commonly used in the food and beverage industries (Hustede *et al.*, 1998; Lück and Von Rymon Lipinski, 1988). A number of the salts of gluconic acid are important pharmaceutical chemicals (Theander, 1980). Recent studies also indicated that salts of gluconic acid have properties that make it usable in other applications (Labuschagné *et al.*, 2000; Labuschagné *et al.*, 2001). The crystal structure of ammonium D-gluconate has been described by Lis (1981) (see Table I) and the structure shown in Figure I. This paper is dedicated to the characterization of ammonium D-gluconate using X-ray powder diffraction.

II. Synthesis

A solution of ca. 50 % gluconic acid in water from Merck (synthesis grade) was used for the synthesis of the crystals. An excess of 10 % on mole ratio of a 35 % ammonium solution of analytic reagent grade was added to the gluconic acid. The solution was closed and stirred continuously for 24 hours. Activated carbon was used to purify the solution, as the gluconic acid tends to discolour in the presence of oxygen. The solution was evaporated at room temperature for several days in order for the ammonium gluconate to crystallise from the solution. Large crystals grew from the solution. A desiccator was used to dry and store the crystals.

III. EXPERIMENTAL PROCEDURES

Diffraction data were collected at room temperature on a Siemens D-501 automated diffractometer using a Cu target ($\lambda = 1.5406 \text{ \AA}$) operated at 40 kV and 40 mA. The instrument was equipped with a diffracted beam graphite monochromator, divergence slit of 1° , receiving slit of 0.05° and scintillation counter. A sample spinner was used. The sample was step scanned from 3° to $90^\circ 2\theta$ with steps of 0.03° with a fixed counting time of 5s at a mean temperature of 25°C . Si-powder (99% from Aldrich Chemical Company Inc.) was used as an internal standard in the first data collection run and the data used to compile Table II and (corrected accordingly) presented in Figure II. Powdering of the sample was achieved by mortar and pestle grinding and the sample was front loaded into a standard Siemens sample holder.

IV. RESULTS

X-ray powder diffraction data for ammonium D-gluconate are given in Table II and the diffraction pattern is shown in Figure I. All of the reflections were indexed successfully using the CHEKCELL program (Laugier & Bochu,). The lattice parameters were determined using the same program and the refined parameters are $a = 6.8059 (2) \text{ \AA}$, $b = 7.6275 (2) \text{ \AA}$ and $c = 17.8028 (5) \text{ \AA}$, which compare well with the values published by Lis (1981).

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Table I: Crystal Structure of ammonium D-gluconate (Lis, 1981)

Crystal System	Orthorhombic
Space Group	$P2_12_12_1$
a	6.810 (4) Å
b	7.630 (4) Å
c	17.796 (9) Å
Z	4
Volume of the unit cell	924.7 Å ³
Measured Density	1.53 mg m ⁻³

Table II. X-ray powder diffraction data for Ammonium D-Gluconate, $C_6H_{11}O_7^-NH_4^+$

I/I_0	d_{obs}	$2\theta_{obs}$	$2\theta_{calc}$	$h k l$	$\Delta 2\theta$
18	8.8425	9.995	9.937	0 0 2	0.058
4	7.0372	12.568	12.615	0 1 1	-0.047
5	6.3294	13.981	13.916	1 0 1	0.065
2	5.7902	15.290	15.289	0 1 2	0.001
6	5.4171	16.350	16.383	1 0 2	-0.033
62	5.0695	17.480	17.444	1 1 0	0.036
25	4.8784	18.170	18.147	1 1 1	0.023
23	4.6782	18.955	18.941	0 1 3	0.014
66	4.4659	19.865	19.840	1 0 3	0.025
86	3.8534	23.062	23.037	1 1 3	0.025
13	3.7295	23.840	23.839	0 2 1	0.001
4	3.5058	25.385	25.387	0 2 2	-0.002
23	3.3953	26.226	26.155	2 0 0	0.071
100	3.3446	26.631	26.639	2 0 1	-0.008
53	3.2066	27.800	27.788	0 2 3	0.012
29	3.1079	28.701	28.692	2 1 0	0.009
16	2.9609	30.158	30.120	0 0 6	0.038
44	2.9116	30.682	30.656	1 1 5	0.026
25	2.7523	32.505	32.493	2 1 3	0.012
19	2.7202	32.900	32.926	1 0 6	-0.026
3	2.6606	33.659	33.610	1 2 4	0.049
3	2.6042	34.410	34.445	0 2 5	-0.035
24	2.5586	35.043	35.017	1 1 6	0.026
18	2.5142	35.682	35.680	2 2 1	0.002
16	2.4366	36.859	36.958	1 2 5	-0.099
5	2.3799	37.770	37.757	1 0 7	0.013
8	2.3583	38.130	38.081	1 3 1	0.049
9	2.3335	38.550	38.530	2 2 3	0.020
20	2.2992	39.148	39.115	1 3 2	0.033
8	2.2724	39.629	39.626	1 1 7	0.003
20	2.2111	40.776	40.788	1 3 3	-0.012
17	2.1355	42.287	42.305	0 1 8	-0.018
37	2.1138	42.744	42.747	1 0 8	-0.003
4	2.0670	43.760	43.757	2 2 5	0.003
11	2.0404	44.360	44.319	3 1 3	0.041
10	2.0203	44.826	44.842	1 2 7	-0.016
4	1.9792	45.808	45.804	1 3 5	0.004
5	1.9486	46.572	46.524	3 2 0	0.048

6	1.9164	47.400	47.480	3 0 5	-0.080
7	1.9011	47.805	47.887	1 0 9	-0.082
2	1.8493	49.231	49.247	1 2 8	-0.016
4	1.8081	50.430	50.419	2 1 8	0.011
5	1.7539	52.106	52.107	3 1 6	-0.001
3	1.6988	53.928	53.911	1 2 9	0.017
4	1.6683	54.997	55.012	2 1 9	-0.015
5	1.6320	56.330	56.332	1 4 5	-0.002
3	1.5814	58.301	58.279	0 1 11	0.022
7	1.5604	59.162	59.157	0 3 9	0.005
<1	1.5043	61.604	61.625	0 5 2	-0.021
2	1.4875	62.378	62.331	1 4 7	0.047
2	1.4569	63.840	63.834	2 2 10	0.006
2	1.4124	66.103	66.132	1 5 4	-0.029
1	1.3864	67.505	67.459	3 4 4	0.046
<1	1.3743	68.184	68.231	1 5 5	-0.047
1	1.3469	69.765	69.773	0 1 13	-0.008
2	1.2670	74.889	74.905	4 4 1	-0.016
3	1.2530	75.868	75.849	0 1 14	0.019
1	1.2334	77.298	77.296	0 4 11	0.002
2	1.2204	78.277	78.269	3 4 8	0.008
2	1.2045	79.514	79.503	2 2 13	0.011
<1	1.1750	81.929	81.978	3 4 9	-0.049
1	1.1680	82.523	82.543	2 6 3	-0.020
1	1.1575	83.440	83.387	0 5 10	0.053
1	1.1351	85.470	85.501	6 0 0	-0.031
1	1.1203	86.881	86.894	3 2 13	-0.013

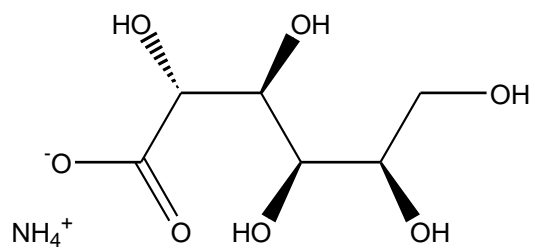


Figure I: The structure of Ammonium D-Gluconate, $C_6H_{11}O_7^-NH_4^+$.

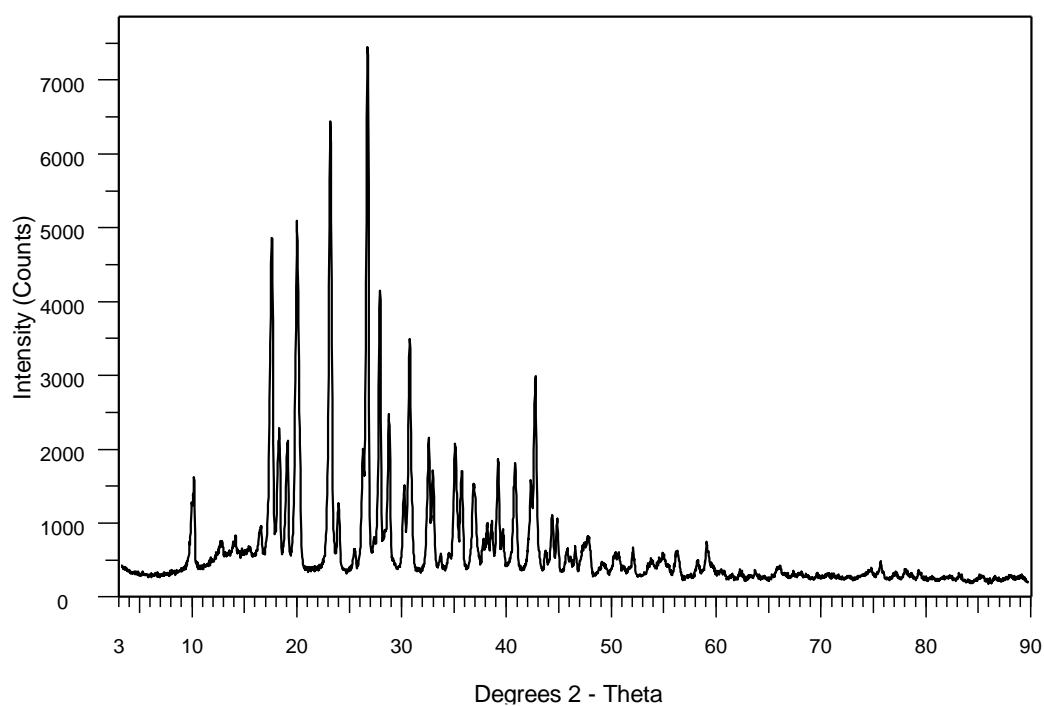


Figure II: Raw X-ray diffractogram of Ammonium D-Gluconate, $C_6H_{11}O_7^-NH_4^+$.