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DATA REPORT

X-ray powder diffraction data for niclosamide, $C_{13}H_8N_2O_4Cl_2$

Liu Qi Guo, Kai Lin Xu, Xiao Li Ma, Shan Shan Li, and Hui Li[a)]
College of Chemical Engineering, Sichuan University, Chengdu 610065, China

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Experimental X-ray powder diffraction data, unit-cell parameters, and space group for niclosamide, $C_{13}H_8N_2O_4Cl_2$, are reported [$a = 13.571(1)$ Å, $b = 7.181(8)$ Å, $c = 13.742(3)$ Å, $\beta = 97.984(7)°$, unit-cell volume $V = 1326.40$ Å^3, $Z = 4$, and space group $P2_1/c$]. All measured lines were indexed and are consistent with the $P2_1/c$ space group. No detectable impurity was observed. © *2015 International Centre for Diffraction Data*. [doi:10.1017/S0885715615000615]

Key words: X-ray powder diffraction, niclosamide

I. INTRODUCTION

Niclosamide (chemical name 2, 5-dichloro-4-nitrosalicylanilide, Figure 1, hereinafter abbreviated NCL), is a frequently-used anthelmintic drug to restrain the infestation of multiple worms (Weinbach & Garbus, 1969). It is also used for therapeutic application including antibacterial and antitumour activities (Helfman, 2011), as well as inhibits the replication of several acute respiratory syndrome coronavirus (Wu *et al.*, 2004).

The single-crystal structure of NCL ($a = 13.485(2)$ Å, $b = 7.0669(11)$ Å, $c = 13.510(2)$ Å, $\beta = 98.345(2)°$, unit-cell volume $V = 1273.9(3)$ Å^3, $Z = 4$, and space group $P2_1/c$) was first reported by Sanphui (Sanphui *et al*., 2012). But detailed X-ray powder diffraction data for NCL have not been reported in the literature. The X-ray powder diffraction data of NCL were collected, analyzed, and evaluated in this paper.

II. EXPERIMENTAL

A. Sample preparation

The title compound, NCL (99% purity), in powder form was obtained from Chengdu Aikeda Pharmaceutical Co., Ltd, its melting range is 227–229°C and density is 1.625 g cm^{-3}. It was recrystallized, dried, and then sieved by a 200-mesh screen to obtain the appropriate particle size (Figure 2). The NCL sample was characterized by high-performance liquid chromatography, Ultraviolet spectroscopy, and Fourier Transform Infrared Spectrometer.

B. Powder Diffraction data collection and reduction

The diffraction pattern for the NCL powder was collected at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with an X'celerator detector and Cu$K\alpha$ radiation (generator setting: 40 kV and 40 mA). The diffraction data were collected in the angular range from 4° to 50°2θ with a step size of 0.01313°2θ and a counting time of 100 ms step^{-1}. Data structure solution was performed using the software package Material Studio 4.2 (Accelrys Co., Ltd., CA, USA).

Through analyzing the peak positions in the powder X-ray diffraction (PXRD) pattern by X-Cell method from "Powder Indexing", the preliminary unit-cell parameters were obtained. The indexing results were then refined with the type of Pawley (Pan *et al.*, 2012), which involves assigning the Miller indices (*h, k, l*) to each observed peak in the experimental PXRD pattern (Harris, 2012). In order to obtain detailed X-ray powder diffraction data, variables defining the structural model and the powder diffraction profiles from the results of Powder Indexing and the experiment powder diffraction pattern were recurrently refined by Pawley refinement (Pawley, 1981) techniques. After Pawley refinement, the final R_{wp} of the spectrogram was converged to 4.20%.

III. RESULTS

The diffraction patterns of recrystallized NCL matched a pattern collected which corresponds to a pattern simulated using the single-crystal structure data (Figure 3). Indexing results confirmed that NCL is monoclinic with space group $P2_1/c$ and unit-cell parameters: $a = 13.571(1)$ Å, $b = 7.181(8)$ Å, $c = 13.742(3)$ Å, $\beta = 97.984(7)°$, unit-cell volume $V = 1326.40$ Å^3, $Z = 4$, and space group $P2_1/c$ (Table I). Using Pawley refinement, the detailed X-ray powder diffraction data of NCL was successfully determined.

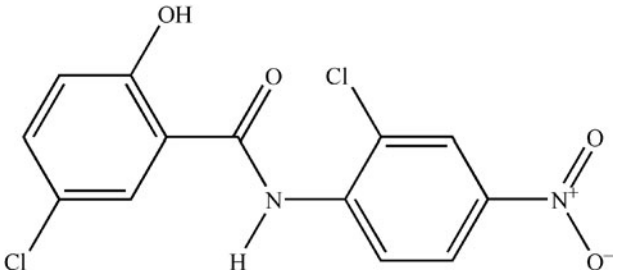


Figure 1. Structural formula of NCL.

[a)]Author to whom correspondence should be addressed. Electronic mail: lihuilab@sina.com

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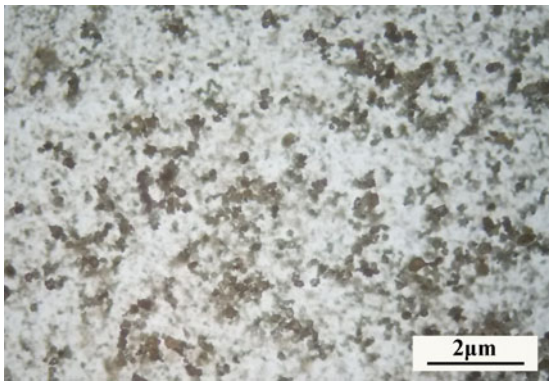


Figure 2. (Color online) Optical photomicrograph of NCL.

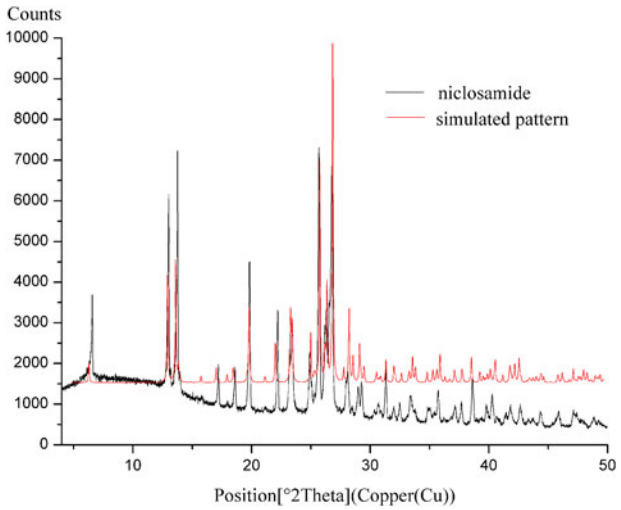


Figure 3. (Color online) Comparison of the powder diffraction pattern of NCL recrystallized in methanol (black) with the pattern simulated from the single-crystal data (red).

TABLE I. X-ray powder diffraction data of NCL ($C_{13}H_8N_2O_4Cl_2$).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
6.5538	13.4754	48	1	0	0	6.5714	13.4395	−0.0175
13.0271	6.7903	84	0	0	2	12.9997	6.8046	0.0274
13.1715	6.7162	26	2	0	0	13.1714	6.7162	0.0001
13.7624	6.4291	99	1	0	−2	13.7345	6.4421	0.0279
17.2288	5.1426	26	2	0	−2	17.1955	5.1525	0.0333
18.5943	4.7679	26	2	1	−1	18.5744	4.7730	0.0199
19.8286	4.4738	62	2	1	1	19.8304	4.4734	−0.0019
22.1920	4.0024	45	3	0	−2	22.1717	4.0060	0.0203
23.2030	3.8303	32	0	1	3	23.1720	3.8353	0.0311
23.4000	3.7985	40	3	1	0	23.3843	3.8010	0.0157
24.7918	3.5883	19	0	2	0	24.7736	3.5909	0.0182
24.8837	3.5752	30	1	1	3	24.8582	3.5788	0.0255
25.6847	3.4655	100	1	2	0	25.6572	3.4692	0.0275
26.1705	3.4023	40	0	0	4	26.1706	3.4023	−0.0001
26.3018	3.3856	46	1	2	−1	26.2599	3.3909	0.0418
26.5381	3.3560	48	4	0	0	26.5068	3.3599	0.0313
26.7613	3.3285	94	1	2	1	26.7230	3.3332	0.0384
27.9431	3.1904	22	4	0	−2	27.9114	3.1939	0.0317
28.1006	3.1728	25	2	1	3	28.0870	3.1743	0.0136
28.9804	3.0785	20	1	1	−4	28.9411	3.0826	0.0393
29.2824	3.0474	21	1	2	2	29.2886	3.0468	−0.0062
30.7004	2.9098	14	3	0	−4	30.6850	2.9112	0.0154
31.2913	2.8562	27	4	0	2	31.2587	2.8591	0.0325
31.7509	2.8155	9	0	2	3	31.7550	2.8155	−0.0042
32.0135	2.7934	12	3	2	−1	32.0397	2.7912	−0.0262
32.4730	2.7549	14	3	1	3	32.4547	2.7564	0.0183
33.0770	2.7060	10	1	2	3	33.0360	2.7092	0.0411
33.4053	2.6801	17	2	2	−3	33.4007	2.6805	0.0046
34.8365	2.5732	13	4	0	−4	34.8128	2.5749	0.0237
35.0072	2.5611	12	1	1	−5	34.9897	2.5623	0.0175
35.7162	2.5118	18	3	2	2	35.6691	2.5150	0.0471
36.0576	2.4888	8	2	1	−5	36.0447	2.4897	0.0129
36.5303	2.4577	8	4	2	−1	36.5195	2.4584	0.0108
36.5828	2.4543	8	4	2	0	36.5967	2.4534	−0.0139
37.1343	2.4191	13	5	1	1	37.1246	2.4197	0.0097
37.6989	2.3842	15	4	2	−2	37.6607	2.3865	0.0382
38.6049	2.3303	23	1	3	−1	38.5781	2.3318	0.0268
39.4189	2.2840	9	2	1	5	39.4203	2.2839	−0.0014
39.7997	2.2630	13	3	2	−4	39.8293	2.2614	−0.0296
40.2987	2.2361	17	4	2	2	40.2880	2.2367	0.0106
41.3360	2.1824	10	1	1	−6	41.3594	2.1812	−0.0235

Continued

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
41.8218	2.1582	14	6	1	−1	41.8509	2.1567	−0.0291
42.6359	2.1188	14	5	2	−2	42.5982	2.1206	0.0377
43.3055	2.0876	9	5	1	3	43.2774	2.0889	0.0281
43.6863	2.0703	9	6	1	1	43.6762	2.0707	0.0100
44.3165	2.0423	11	3	2	−5	44.2995	2.0430	0.0171
45.8659	1.9768	11	5	0	4	45.8231	1.9786	0.0428
46.3255	1.9583	7	0	3	4	46.3367	1.9578	−0.0113
47.0870	1.9284	11	6	1	−4	47.0609	1.9294	0.0261
47.4153	1.9158	11	1	3	4	47.4184	1.9157	−0.0032

The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.540\ 56$ Å).

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SUPPLEMENTARY MATERIALS

For supplementary material for this article, please visit http://dx.doi.org/10.1017/S0885715615000615

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