DATA REPORT

X-ray powder diffraction data for deferasirox, $C_{21}H_{15}N_3O_4$

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X-ray powder diffraction data, unit-cell parameters, and space group for deferasirox, $C_{21}H_{15}N_3O_4$, are reported [$a = 8.821(7)$ Å, $b = 26.798(2)$ Å, $c = 7.540(4)$ Å, $\alpha = 90°$, $\beta = 94.655(2)°$, $\gamma = 90°$, unit-cell volume $V = 1776.7(3)$ Å^3, $Z = 4$, $\rho_{cal} = 1.396$ g cm^{-3}, 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.
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Key words: X-ray powder diffraction, deferasirox

I. INTRODUCTION

Deferasirox (Exjade®, ICL670, Figure 1), chemical name 4-[3,5-bis (2-hydroxyphenyl)-1,2,4- triazol-1-yl] benzoic acid, is an oral, once-daily iron chelator widely approved for the treatment of transfusional chronic iron overload (Yang *et al*., 2007), and also as salvage therapy for a patient with progressive rhinocerebral mucormycosis (Reed *et al*., 2006). Deferasirox was the first oral iron chelator approved by the Food and Drug Administration (FDA). Compared with other iron chelators, deferasirox can rapid access to intracellular iron pools (Neufeld, 2006), and with less side effects. Therefore, deferasirox has become one of the most promising iron chelator drugs.

The single-crystal structure of deferasirox [$a = 8.900(2)$ Å, $b = 26.946(5)$ Å, $c = 7.558(2)$ Å, $\beta = 94.77(3)°$, unit-cell volume $V = 1806.3(7)$ Å^3, $Z = 4$, and space group $P2_1/c$] was first reported by Steinhauser *et al*. (2004). But detailed X-ray powder diffraction data for deferasirox have not been reported. In this report, the X-ray powder diffraction data of deferasirox were collected and analyzed.

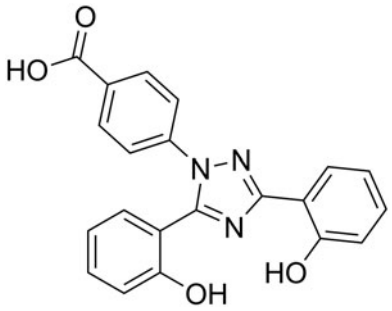


Figure 1. Structural formula of deferasirox.

II. EXPERIMENTAL

A. Sample preparation

The title compound, deferasirox (≥98% purity), in powder form was obtained from Wuhan E-ternity Technologies Co., Ltd. (Shanghai, China), and was characterized by FT-IR measurements (Thermo Fisher Scientific, Madison, USA, online Figure S1). The melting point and measured density of deferasirox are 262.48 °C and 1.40 g cm^{-3}. The compound was re-crystallized by slow evaporation of a methanol solution at room temperature. Then the crystallized product with a needle-like shape was dried and ground into powder.

B. Powder diffraction data collection and reduction

X-ray powder diffraction measurement was performed at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with a PIXcel 1D detector and Cu$K\alpha$ radiation (generator setting: 40 kV and 40 mA). The diffraction data were collected over the angular range from 4° to 51°2θ with a step size of 0.013 13°2θ and a counting time of 50 ms step^{-1}. Figure 2 shows the powder X-ray diffraction (XRD) pattern of the compound.

The software package Material Studio 8.0 (Accelrys Co., Ltd., CA, USA) was used to process the data in the Analytical & Testing Center (Sichuan University, Chengdu, China). The powder XRD pattern was pretreated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component (Wang *et al*., 2016). Automatic indexing results were obtained

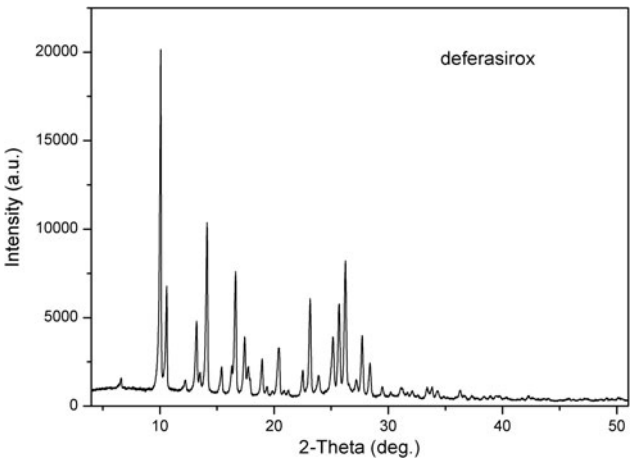


Figure 2. Powder XRD pattern of deferasirox.

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TABLE I. X-ray powder diffraction data for deferasirox, $C_{21}H_{15}N_3O_4$. The *d*-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$ (°)
6.6132	13.3545	8	0	2	0	6.5912	13.3991	0.0220
10.0796	8.7683	100	1	0	0	10.0517	8.7926	0.0279
10.5917	8.3445	34	1	1	0	10.5804	8.3544	0.0113
12.2330	7.2293	7	0	1	1	12.2209	7.2364	0.0121
13.2046	6.6994	24	0	4	0	13.2043	6.6996	0.0003
13.4804	6.5630	9	0	2	1	13.4972	6.5549	−0.0168
14.1238	6.2654	51	1	3	0	14.1219	6.2663	0.0019
15.2004	5.8240	6	1	1	−1	15.2245	5.8148	−0.0241
15.4105	5.7450	11	0	3	1	15.3948	5.7509	0.0157
16.2902	5.4367	11	1	2	−1	16.2711	5.4431	0.0192
16.6316	5.3259	38	1	4	0	16.6221	5.3289	0.0095
17.4195	5.0868	19	1	2	1	17.4236	5.0856	−0.0041
17.7346	4.9971	11	0	4	1	17.7205	5.0010	0.0141
17.8921	4.9534	8	1	3	−1	17.8831	4.9559	0.0090
18.9557	4.6778	13	1	3	1	18.9407	4.6815	0.0150
19.3759	4.5773	5	1	5	0	19.3797	4.5764	−0.0038
19.8486	4.4694	4	0	6	0	19.8620	4.4664	−0.0135
20.1637	4.4002	6	2	0	0	20.1818	4.3963	−0.0182
20.4525	4.3387	16	2	1	0	20.4545	4.3383	−0.0019
20.8727	4.2523	4	1	4	1	20.8865	4.2495	−0.0138
21.2535	4.1770	5	2	2	0	21.2524	4.1772	0.0011
22.3039	3.9826	4	1	6	0	22.3069	3.9821	−0.0030
22.5140	3.9459	10	2	3	0	22.5223	3.9445	−0.0083
22.8291	3.8921	5	2	1	−1	22.8137	3.8947	0.0155
23.1574	3.8377	30	0	6	1	23.1462	3.8395	0.0112
23.6695	3.7558	5	0	0	2	23.6570	3.7578	0.0125
23.8796	3.7233	9	0	1	2	23.8918	3.7214	−0.0122
25.1532	3.5375	19	2	2	1	25.1559	3.5372	−0.0027
25.3633	3.5087	6	1	7	0	25.3535	3.5100	0.0097
25.6784	3.4664	29	1	6	1	25.6784	3.4664	0.0000
25.6915	3.4646	29	0	3	2	25.6979	3.4638	−0.0064
26.2430	3.3931	41	2	3	1	26.2475	3.3925	−0.0045
26.5056	3.3600	6	1	0	2	26.5191	3.3583	−0.0135
27.1753	3.2787	7	0	4	2	27.1863	3.2774	−0.0110
27.7136	3.2163	20	2	4	1	27.7084	3.2168	0.0052
28.3570	3.1447	11	1	4	−2	28.3569	3.1447	0.0001
28.3964	3.1405	12	1	7	1	28.3867	3.1415	0.0097
29.4731	3.0281	5	2	5	1	29.4886	3.0266	−0.0155
30.2346	2.9536	4	2	6	−1	30.2385	2.9532	−0.0039
30.4710	2.9312	3	3	0	0	30.4744	2.9309	−0.0035
30.7204	2.9080	3	2	2	−2	30.7265	2.9074	−0.0060
31.0881	2.8744	5	0	6	2	31.0766	2.8754	0.0115
31.2063	2.8638	5	3	2	0	31.2131	2.8632	−0.0068
31.2325	2.8614	5	1	8	1	31.2432	2.8605	−0.0107
31.3901	2.8474	4	1	5	2	31.4083	2.8458	−0.0182
31.6527	2.8244	4	2	3	−2	31.6407	2.8254	0.0120
31.7052	2.8198	4	1	9	0	31.7006	2.8203	0.0046
32.0860	2.7872	4	3	3	0	32.1149	2.7848	−0.0289
32.3092	2.7685	3	0	9	1	32.3124	2.7682	−0.0032
33.3990	2.6806	5	0	10	0	33.4092	2.6798	−0.0102
33.8192	2.6483	5	2	7	1	33.8194	2.6482	−0.0002
34.2131	2.6187	4	1	9	1	34.2202	2.6181	−0.0071
34.3838	2.6061	4	1	7	−2	34.3631	2.6076	0.0206
36.2351	2.4770	4	2	6	−2	36.2214	2.4779	0.0137
36.2614	2.4753	4	2	8	1	36.2921	2.4733	−0.0307
36.3139	2.4718	4	3	4	1	36.3256	2.4711	−0.0117
36.5897	2.4538	3	1	1	−3	36.6149	2.4522	−0.0252
36.7341	2.4445	3	1	10	−1	36.7440	2.4439	−0.0099
37.3118	2.4080	3	3	0	−2	37.3156	2.4078	−0.0038
38.3097	2.3475	3	0	4	3	38.3273	2.3465	−0.0175
38.4016	2.3421	3	2	6	2	38.4251	2.3408	−0.0235
38.9137	2.3125	3	1	4	−3	38.9335	2.3114	−0.0197
39.4521	2.2822	3	1	3	3	39.4703	2.2811	−0.0182
39.7541	2.2655	3	3	7	−1	39.7696	2.2647	−0.0155
42.2882	2.1354	3	4	3	0	42.3079	2.1345	−0.0197
42.6165	2.1197	2	3	6	−2	42.6233	2.1194	−0.0068

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$ (°)
44.0739	2.0530	2	4	4	−1	44.0592	2.0536	0.0147
49.1028	1.8538	2	3	8	2	49.1124	1.8535	−0.0096
50.1664	1.8170	2	4	7	1	50.1505	1.8175	0.0159
50.4421	1.8077	2	0	13	2	50.4532	1.8073	−0.0111

by the DICVOL91 method (Boultif and Louër, 1991). The indexing results were then refined with the type of Pawley (1981), which involves assigning the Miller indices (*h, k, l*) to each observed peak in the experimental powder XRD pattern (Xu *et al*., 2014).

III. RESULTS

Pawley refinement results confirmed that deferasirox is monoclinic with space group $P2_1/c$ and unit-cell parameters: $a = 8.821(7)$ Å, $b = 26.798(2)$ Å, $c = 7.540(4)$ Å, $\alpha = 90°$, $\beta = 94.655(2)°$, $\gamma = 90°$, unit-cell volume $V = 1776.7(3)$ Å^3, $Z = 4$, and $\rho_{cal} = 1.396$ g cm^{-3}. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, and $\Delta 2\theta$ are listed in Table I. The figures of merit were achieved: $F_{24} = 47.5$ (0.0071, 71) (Smith and Snyder, 1979) and $M_{24} = 22.9$ (de Wolff, 1968). The comparison of this pattern with the calculated pattern of single-crystal data reported by Steinhauser *et al*. (2004) is shown in online Figure S2. The results showed that both single-crystal and powder diffraction methods can get similar structure data.

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SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at http://dx.doi.org/10.1017/S0885715616000476

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