X-ray powder diffraction data for trelagliptin succinate, $C_{18}H_{20}FN_5O_2•C_4H_6O_4$

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Experimental X-ray powder diffraction data, unit-cell parameters, and space group for trelagliptin succinate, $C_{18}H_{20}FN_5O_2•C_4H_6O_4$, are reported [$a = 18.481(6)$, $b = 8.120(4)$, $c = 7.903(4)$ Å, $\alpha = 89.972(0)°$, $\beta = 97.722(4)°$, $\gamma = 90.120(0)°$, unit-cell volume $V = 1175.37$ Å^3, $Z = 2$, and space-group $P1$]. All measured lines were indexed and are consistent with the $P1$ space group. No detectable impurity was observed. © *2016 International Centre for Diffraction Data*. [doi:10.1017/S0885715615001025]

Key words: X-ray powder diffraction, trelagliptin succinate

I. INTRODUCTION

Trelagliptin succinate [chemical name, 2-[[6-[(3R)-3-amino-1-piperidinyl]-3,4-dihydro-3-methyl-2,4-dioxo-1(2H)-pyrimidinyl]methyl]-4-fluorobenzonitrile succinic acid salt (Figure 1), hereinafter abbreviated TS], is a novel once-weekly oral DPP-4 inhibitor for type 2 diabetes (Inagaki *et al.*, 2014, 2015).

The crystal structure of TS has not been reported so far. The powder X-ray diffraction (XRD) data of TS were collected, analyzed, and evaluated in this literature.

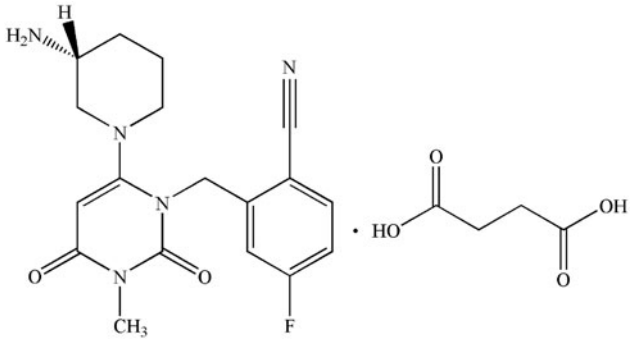


Figure 1. Structural formula of TS.

II. EXPERIMENTAL

A. Sample preparation

The title compound, TS (99 % purity), in powder form was obtained from J&K Chemical Co., Ltd., and its melting range is 184–185 °C. It was recrystallized, dried, and then sieved by a 200-mesh screen to obtain the appropriate particle size. The TS sample was characterized by the melting point and Fourier transform infrared spectroscopy measurements.

B. Powder diffraction data collection and reduction

The diffraction pattern for the TS powder was collected at room temperature using an X' Pert PRO diffractometer (PANalytical Co., Ltd., The 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 0.013 13°2θ and a counting time of 30 ms step^{-1}. Data structure solution was performed using the software package Material Studio 4.2 (Accelrys Co., Ltd. USA) in the State Key Laboratory of Polymer Materials Engineering (Sichuan University, China).

Through analyzing the peak positions in the powder XRD pattern by the 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 powder XRD pattern (Harris, 2012). To obtain the detailed powder XRD 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 techniques (Pawley, 1981). After the Pawley refinement, the final R_{wp} of the spectrogram was converged to 6.28%.

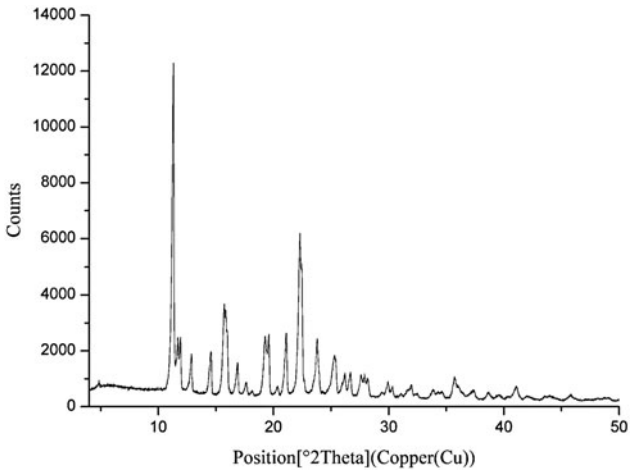


Figure 2. Powder XRD pattern of TS using Cu$K\alpha$ radiation ($\lambda = 1.540\,56$ Å).

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TABLE I. Powder XRD data of TS, $C_{18}H_{20}FN_5O_2$ $C_4H_6O_4$. 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)	$\Delta 2\theta$
4.8469	18.2166	8	1	0	0	4.8179	18.3260	0.0290
11.3201	7.8101	100	0	0	1	11.2861	7.8336	0.0340
11.7009	7.5568	20	1	0	−1	11.6645	7.5803	0.0364
11.9373	7.4077	20	1	1	0	11.9099	7.4247	0.0274
12.8826	6.8661	15	1	0	1	12.8619	6.8771	0.0207
14.5896	6.0664	16	2	1	0	14.5709	6.0742	0.0187
15.7450	5.6237	30	0	1	−1	15.7021	5.6390	0.0430
15.8632	5.5821	28	2	0	1	15.8269	5.5948	0.0363
15.9551	5.5502	22	1	−1	−1	15.9606	5.5483	−0.0055
16.8874	5.2458	13	1	−1	1	16.8700	5.2512	0.0173
17.6489	5.0211	7	2	1	−1	17.6523	5.0202	−0.0033
18.1479	4.8842	5	3	−1	0	18.1303	4.8889	0.0176
19.2640	4.6037	20	2	1	1	19.2509	4.6068	0.0131
19.5922	4.5273	21	3	0	1	19.5750	4.5312	0.0172
20.3538	4.3596	6	3	1	−1	20.3756	4.3549	−0.0218
21.1285	4.2014	21	4	0	−1	21.0918	4.2087	0.0367
22.2970	3.9838	50	4	1	0	22.2767	3.9874	0.0203
22.4415	3.9585	42	3	1	1	22.4578	3.9557	−0.0163
23.8070	3.7344	20	4	1	−1	23.8113	3.7338	−0.0043
25.2908	3.5186	15	3	0	−2	25.2891	3.5188	0.0017
26.2099	3.3973	10	4	1	1	26.2112	3.3971	−0.0013
26.6826	3.3381	10	5	1	0	26.6802	3.3384	0.0024
27.6279	3.2260	9	3	1	−2	27.6188	3.2271	0.0091
27.9037	3.1948	10	4	0	−2	27.8873	3.1966	0.0163
28.1400	3.1685	8	2	1	2	28.1573	3.1666	−0.0173
29.3874	3.0368	4	4	2	0	29.3843	3.0371	0.0031
29.5450	3.9551	4	3	2	1	29.5175	3.0237	0.0275
29.9520	2.9808	7	4	−1	−2	29.9910	2.9770	−0.0390
30.3328	2.9442	6	5	1	1	30.3318	2.9443	0.0010
31.0943	2.8738	4	5	0	−2	31.0734	2.8757	0.0210
31.6721	2.8227	5	0	2	2	31.6886	2.8213	−0.0166
31.9609	2.7979	6	4	0	2	31.9663	2.7974	−0.0054
32.5255	2.7506	4	4	2	1	32.5226	2.7508	0.0030
33.8780	2.6438	5	4	1	2	33.8700	2.6444	0.0080
34.2719	2.6143	5	0	0	3	34.3141	2.6112	−0.0423
34.6526	2.5865	5	6	−1	1	34.6761	2.5848	−0.0235
35.7162	2.5118	9	5	0	2	35.6964	2.5132	0.0198
37.4100	2.4019	5	5	−1	2	37.4110	2.4018	−0.0010
38.6574	2.3272	4	3	−3	1	38.6617	2.3270	−0.0043
39.5634	2.2760	4	5	0	−3	39.5639	2.2760	−0.0005
41.0734	2.1957	6	0	2	−3	41.0627	2.1963	0.0107
41.9662	2.1511	3	1	−2	3	41.9377	2.1525	0.0286
43.5812	2.0750	3	4	−2	−3	43.5594	2.0760	0.0219
44.0408	2.0544	3	8	1	1	44.0654	2.0533	−0.0246
44.7630	2.0229	2	6	3	0	44.7217	2.0247	0.0413
45.8003	1.9795	4	6	−2	2	45.7870	1.9801	0.0133
49.0566	1.8555	3	3	−4	1	49.0624	1.8553	−0.0059

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results confirmed that TS is triclinic with space-group *P*1 and unit-cell parameters: $a = 18.481(6)$, $b = 8.120(4)$, $c = 7.903(4)$ Å, $\alpha = 89.972(0)°$, $\beta = 97.722(4)°$, $\gamma = 90.120(0)°$, unit-cell volume $V = 1175.37$ Å^3, $Z = 2$, and space-group *P*1 (Table I). Using the Pawley refinement, the detailed powder XRD data of TS was successfully determined.

SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit http://dx.doi.org/10.1017/S0885715615001025

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