# X-ray powder diffraction data for 7-ethyl-14-nitro-camptothecin, $C_{22}H_{19}N_3O_6$

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X-ray powder diffraction (XRD) data, unit-cell parameters and space group for 7-ethyl-14-nitro-camptothecin, $C_{22}H_{19}N_3O_6$, are reported [$a = 10.987(5)$ Å, $b = 10.941$ (9) Å, $c = 8.438$ (2) Å, $\alpha = 71.321(6)°$, $\beta = 96.145(0)°$, $\gamma = 95.139(3)°$, unit-cell volume $V = 953.87$ Å$^3$, $Z = 2$, $\rho_{cal} = 1.467$ g cm$^{-3}$, and space group $P$-1]. All measured lines were indexed and are consistent with the $P$-1 space group. No detectable impurities were observed. © *2018 International Centre for Diffraction Data*. [doi:10.1017/S0885715618000684]

Key words: X-ray powder diffraction, 7-ethyl-14-nitro-camptothecin

## I. INTRODUCTION

7-Ethyl-14-amino-camptothecin was reported as a promising candidate for tumor treatment based on its superiority to traditional camptothecin derivatives, including excellent efficacy, acceptable safety, significant brain penetration and poor substrate properties toward the major drug-resistant pumps (Duan *et al*., 2011; Cheng *et al*., 2015). The title compound, 7-ethyl-14-nitro-camptothecin (ENC, Figure 1), is an intermediate in the synthesis of 7-ethyl-14-amino-camptothecin from 7-ethylcamptothecin (Duan *et al*., 2011). Therefore, it is very important to design the crystallization techniques of ENC as well as to be certain of its crystal structure.

To date, detailed X-ray powder diffraction (XRD) data for ENC have not been reported.

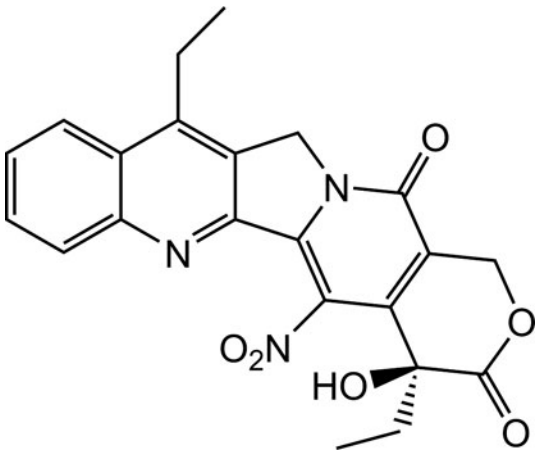


Figure 1. Molecular diagram for 7-ethyl-14-nitro-camptothecin.

## II. EXPERIMENTAL

### A. Sample preparation

ENC was obtained from Sichuan Sinovation Biotech Co., Ltd., China. It was re-crystallized in methanol–dichloromethane (1 : 1, v/v), then dried and ground into powder. The sample was characterized by melting point (306.9 °C), density (1.459 g cm$^{-3}$), FT-IR (Supplementary Figure S1) and MS ($[M + H]^+ = 422.1$, Supplementary Figure S2). Further, a purity of 98.5% was checked by HPLC (Supplementary Figure S3).

### B. Diffraction data collection and reduction

XRD measurements were performed at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with a PIXcel one-dimensional detector and Cu$K\alpha$ radiation ($\lambda = 1.5406$ Å, generator voltage and current were set at 40 kV and 40 mA). The diffraction data were collected over the angular range from 4° to 50° 2$\theta$ with a step size of 0.013 13° 2$\theta$ and a counting time of 29.07 ms per step (Figure 2).

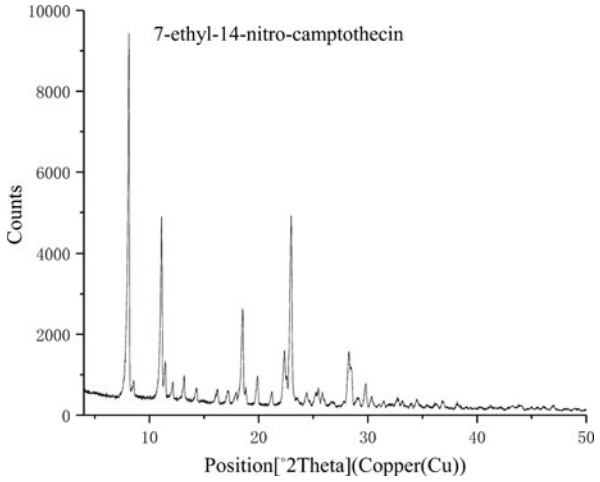


Figure 2. XRD pattern of 7-ethyl-14-nitro-camptothecin using Cu$K\alpha$ radiation.

Data evaluation was performed using the software package Material Studio 8.0 (Accelrys Co., Ltd., San Diego, California, USA) in the Analytical & Testing Center

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TABLE I. Indexed XRD data for 7-ethyl-14-nitro-camptothecin, $C_{22}H_{19}N_3O_6$. The *d*-values were calculated using Cu$K\alpha$ radiation ($\lambda$ = 1.540 56 Å).

| $2\theta_{obs}$ (°) | $d_{obs}$ (Å) | $I_{obs}$ | $h$ | $k$ | $l$ | $2\theta_{cal}$ (°) | $d_{cal}$ (Å) | $\Delta 2\theta$ |
|---|---|---|---|---|---|---|---|---|
| 8.1263 | 10.8765 | 100 | 1 | 0 | 0 | 8.1006 | 10.9055 | 0.0257 |
| 8.5465 | 10.3427 | 9 | 0 | 1 | 0 | 8.5380 | 10.3477 | 0.0085 |
| 11.1201 | 7.9542 | 52 | 0 | 0 | 1 | 11.0977 | 7.9661 | 0.0224 |
| 11.4352 | 7.7357 | 14 | 1 | −1 | 0 | 11.4280 | 7.7366 | 0.0072 |
| 12.1180 | 7.3013 | 9 | 1 | 1 | 0 | 12.1214 | 7.2956 | −0.0034 |
| 13.1815 | 6.7145 | 10 | 1 | 0 | −1 | 13.1969 | 6.7033 | −0.0154 |
| 14.2845 | 6.1984 | 7 | 1 | 0 | 1 | 14.2912 | 6.1924 | −0.0067 |
| 16.0045 | 5.5359 | 5 | 0 | 1 | −1 | 16.0159 | 5.5292 | −0.0114 |
| 16.2409 | 5.4559 | 7 | 2 | 0 | 0 | 16.2421 | 5.4527 | −0.0012 |
| 17.1600 | 5.1657 | 6 | 0 | 2 | 0 | 17.1239 | 5.1739 | 0.0361 |
| 17.2388 | 5.1422 | 6 | 0 | 2 | 1 | 17.2470 | 5.1372 | −0.0082 |
| 17.9741 | 4.9335 | 6 | 2 | −1 | 0 | 17.9221 | 4.9452 | 0.0520 |
| 18.2235 | 4.8665 | 7 | 1 | −2 | −1 | 18.2323 | 4.8618 | −0.0088 |
| 18.5124 | 4.7912 | 28 | 1 | −2 | 0 | 18.5292 | 4.7845 | −0.0168 |
| 18.8275 | 4.7117 | 7 | 2 | 1 | 0 | 18.8200 | 4.7112 | 0.0075 |
| 19.9042 | 4.4592 | 10 | 1 | 2 | 1 | 19.8947 | 4.4591 | 0.0095 |
| 21.2304 | 4.1836 | 6 | 0 | 1 | 2 | 21.2221 | 4.1831 | 0.0083 |
| 22.3464 | 3.9771 | 17 | 2 | −2 | −1 | 22.3918 | 3.9672 | −0.0454 |
| 22.5828 | 3.9360 | 10 | 2 | 1 | −1 | 22.5803 | 3.9345 | 0.0025 |
| 22.9767 | 3.8694 | 52 | 2 | −2 | 0 | 22.9717 | 3.8683 | 0.0050 |
| 23.5413 | 3.7779 | 5 | 1 | 1 | 2 | 23.5898 | 3.7683 | −0.0485 |
| 24.4079 | 3.6457 | 6 | 2 | 2 | 0 | 24.3811 | 3.6478 | 0.0268 |
| 24.6705 | 3.6074 | 4 | 1 | 2 | −1 | 24.6700 | 3.6057 | 0.0005 |
| 25.2351 | 3.5280 | 6 | 1 | −3 | −1 | 25.2237 | 3.5278 | 0.0114 |
| 25.4715 | 3.4958 | 7 | 3 | −1 | 0 | 25.4686 | 3.4944 | 0.0029 |
| 25.8391 | 3.4469 | 6 | 3 | −1 | −1 | 25.8472 | 3.4441 | −0.0081 |
| 26.0360 | 3.4213 | 4 | 3 | 0 | −1 | 26.0637 | 3.4213 | −0.0277 |
| 26.8107 | 3.3241 | 4 | 1 | 3 | 1 | 26.7848 | 3.3256 | 0.0259 |
| 27.8480 | 3.2026 | 4 | 3 | 0 | 1 | 27.7862 | 3.2080 | 0.0618 |
| 28.2813 | 3.1545 | 17 | 2 | 1 | 2 | 28.2697 | 3.1542 | 0.0116 |
| 28.4914 | 3.1317 | 13 | 3 | 1 | 1 | 28.4853 | 3.1309 | 0.0061 |
| 29.0035 | 3.0776 | 5 | 3 | 1 | −1 | 29.0157 | 3.0748 | −0.0122 |
| 29.1479 | 3.0627 | 5 | 3 | −2 | 0 | 29.1582 | 3.0601 | −0.0103 |
| 29.8044 | 2.9967 | 8 | 2 | −3 | 0 | 29.8013 | 2.9955 | 0.0031 |
| 30.3690 | 2.9423 | 5 | 2 | 2 | 2 | 30.3362 | 2.9439 | 0.0328 |
| 31.0518 | 2.8791 | 3 | 2 | 3 | 1 | 31.0410 | 2.8787 | 0.0108 |
| 31.4457 | 2.8439 | 4 | 2 | 3 | 0 | 31.4673 | 2.8406 | −0.0216 |
| 32.2861 | 2.7718 | 3 | 1 | −1 | −3 | 32.2442 | 2.7739 | 0.0419 |
| 32.6931 | 2.7382 | 5 | 0 | 2 | 3 | 32.6938 | 2.7368 | −0.0007 |
| 32.8113 | 2.7286 | 4 | 0 | 4 | 1 | 32.8482 | 2.7243 | −0.0369 |
| 33.1395 | 2.7023 | 4 | 1 | −4 | −1 | 33.1396 | 2.7010 | −0.0001 |
| 33.1921 | 2.6982 | 4 | 3 | −3 | −1 | 33.1990 | 2.6963 | −0.0069 |
| 33.9536 | 2.6394 | 3 | 1 | 1 | 3 | 33.9321 | 2.6397 | 0.0215 |
| 34.5182 | 2.5975 | 4 | 2 | −1 | −3 | 34.5240 | 2.5958 | −0.0058 |
| 36.0939 | 2.4876 | 3 | 1 | 4 | 0 | 36.1220 | 2.4845 | −0.0281 |
| 36.8029 | 2.4413 | 4 | 1 | 4 | 2 | 36.8041 | 2.4400 | −0.0012 |
| 36.9211 | 2.4338 | 4 | 3 | 3 | 0 | 36.9324 | 2.4319 | −0.0113 |
| 38.1684 | 2.3571 | 4 | 4 | 2 | 0 | 38.1731 | 2.3556 | −0.0047 |
| 41.2147 | 2.1896 | 3 | 2 | 3 | 3 | 41.2081 | 2.1889 | 0.0066 |
| 43.1579 | 2.0954 | 2 | 2 | −3 | 2 | 43.1400 | 2.0952 | 0.0179 |
| 44.0902 | 2.0532 | 3 | 1 | 5 | 2 | 44.1288 | 2.0505 | −0.0386 |
| 47.0314 | 1.9314 | 3 | 1 | 0 | 4 | 47.0192 | 1.9310 | 0.0122 |

(Sichuan University, Chengdu, China). The XRD pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Automatic indexing results were obtained with DICVOL91 (Boultif and Louër, 1991). The following figures of merit were achieved: $F_{27} = 26.6$ (0.0132, 77) (Smith and Snyder, 1979) and $M_{27} = 11.1$ (de Wolff, 1968). The indexing results were then refined using Pawley refinement (Pawley, 1981), which involves assigning the Miller indices ($h$, $k$, $l$) to each observed peak in the experimental XRD pattern.

## III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results showed that ENC is triclinic, space group $P$-1 and unit-cell parameters: $a = 10.987(5)$ Å, $b = 10.941$ (9) Å, $c = 8.438$ (2) Å, $\alpha = 71.321(6)°$, $\beta = 96.145(0)$ °, $\gamma = 95.139(3)°$, unit-cell volume $V = 953.87$ Å$^3$, $Z = 2$, $\rho_{cal} = 1.467$ g cm$^{-3}$. The values of $2\theta_{obs}$, $d_{obs}$, $I_{obs}$, $h$, $k$, $l$, $2\theta_{cal}$, $d_{cal}$, $I_{cal}$, $\Delta 2\theta$ are listed in Table I. All measured lines were indexed and are consistent with the $P$-1 space group. No detectable impurities were observed.


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## Supplementary material

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715618000684.


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