X-ray powder diffraction data for trimethylene carbonate

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X-ray powder diffraction data, unit-cell parameters and space group for trimethylene carbonate, $C_4H_6O_3$, are reported [$a = 6.145(1)$ Å, $b = 11.328(3)$ Å, $c = 6.900(2)$ Å, $\beta = 99.067(4)°$, unit-cell volume $V = 474.33$ Å^3, $Z = 4$ and space group $P2_1/n$]. All measured lines were indexed and are consistent with the $P2_1/n$ space group. No detectable impurity was observed. © *2012 International Centre for Diffraction Data.* [doi:10.1017/S0885715612000486]

Key words: X-ray powder diffraction, trimethylene carbonate

I. INTRODUCTION

Trimethylene carbonate, systematic name 1,3-dioxane-2-one, shortly TMC (Figure 1), is an important monomer for synthesis of biocompatible polymers – poly(trimethylene carbonate) and poly(ester carbonates), most importantly copolymers of TMC with L-lactide, glycolide or ε-caprolactone (Nair and Laurencin, 2007). These polymers are very useful materials for application in orthopedics and in soft tissue engineering. Depending on the copolymer structure they are used as flexible suture materials, orthopaedic tacks and screws, and they are good candidates for elastic scaffold materials, e.g. for production of vascular grafts and other hollow smooth muscle organs.

The crystal structure of TMC was solved by Kataeva *et al.* (1988).

II. EXPERIMENTAL

A. Sample preparation

TMC was obtained as a gift from Labso Chimie Fine, France. It was sublimated at 40 °C and 700 Pa. TMC was characterized by DSC using DSC Q100 instrument (TA Instruments) at a heating rate 5 °C min^{-1} and nitrogen purge 50 cm^3 min^{-1}. The sublimated TMC exhibited two melting endotherms at 38 and 51 °C with corresponding heats of fusion 11 and 100 J g^{-1}.

B. Diffraction data collection and reduction

The diffraction pattern of TMC was collected at room temperature using an X'Pert PRO θ-θ powder diffractometer with parafocusing Bragg–Brentano geometry and Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å, generator setting: 40 kV, 30 mA). An ultrafast X'Celerator detector was employed to collect XRD data over the angular range from 4 to 60° 2θ with a step size of 0.017° 2θ and a counting time of 20.32 s per step. The experimental powder diffraction pattern is depicted in Figure 2. The software package HighScore Plus V 3.0d (PANalytical, Almelo, Netherlands) was used to smooth the data, to fit the background and to eliminate the $K\alpha_2$ component. The top of the smoothed peak method was used to determine the peak positions and intensities of the diffraction peaks.

Automatic indexing of the experimental XRD pattern was performed using DICVOL04 (Boultif and Louër, 2004).

III. RESULTS

Automatic indexing results obtained by DICVOL04 show that TMC is monoclinic with space group $P2_1/n$ and unit-cell parameters: $a = 6.145(1)$ Å, $b = 11.328(3)$ Å, $c = 6.900(2)$ Å, $\beta = 99.067(4)°$, unit-cell volume $V = 474.33$ Å^3 and $Z = 4$. The figures of merits are $F_{20} = 117.6(0.0036, 47)$ (Smith and Snyder, 1979) and $M_{20} = 69.8$ (de Wolf, 1968). All lines were indexed and are consistent with the $P2_1$ space group.

A comparison of unit cell parameters from powder data at 293 K (this article) and from single crystal data at 113 K (Kataeva *et al.*, 1988) displays a significant shortening of all unit cell parameters at 113 K with the most profound effect on c parameter and even more substantial increase in the monoclinic angle from 99 to 102.6°. The unit-cell volume contracts to 441.7 Å^3.

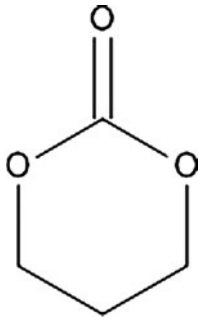


Figure 1. Structural formula of TMC.

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TABLE I. Indexed X-ray powder diffraction data of TMC, $C_4H_6O_3$. Only the peaks with I_{rel} of 1 or greater are presented reported [$a = 6.145(1)$ Å, $b = 11.328(3)$ Å, $c = 6.900(2)$ Å, $\beta = 99.067(4)°$, unit-cell volume $V = 474.33$ Å^3, $Z = 4$ and space group $P2_1/n$]. All measured lines were indexed and are consistent with the $P2_1/n$ space group. The d-values were calculated using Cu$K\,\alpha_1$ radiation ($\lambda = 1.5406$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{calc}$ (°)	d_{calc} (Å)	$\Delta 2\theta$
15.634	5.663	14	0	2	0	15.633	5.664	−0.0017
16.559	5.349	68	1	1	0	16.560	5.349	0.0003
17.962	4.935	4	1	0	−1	17.962	4.934	0.0008
19.605	4.525	9	1	1	−1	19.608	4.524	0.0031
20.375	4.355	41	0	2	1	20.372	4.356	−0.0028
21.074	4.212	15	1	0	1	21.066	4.214	−0.0076
21.442	4.141	14	1	2	0	21.443	4.141	0.0014
22.499	3.949	16	1	1	1	22.494	3.949	−0.0048
23.912	3.718	1	1	2	−1	23.898	3.721	−0.0137
26.138	3.406	100	0	0	2	26.134	3.407	−0.0042
27.800	3.207	1	1	3	0	27.805	3.206	0.0047
29.029	3.073	8	1	1	−2	29.028	3.074	−0.0009
29.774	2.998	3	1	3	−1	29.769	2.999	−0.0041
30.602	2.919	12	0	2	−2	30.596	2.920	−0.0056
31.310	2.855	10	2	1	−1	31.310	2.855	−0.0002
31.574	2.831	4	0	4	0	31.566	2.832	−0.0084
31.789	2.813	3	1	3	1	31.795	2.812	0.0058
33.062	2.707	21	1	1	2	33.050	2.708	−0.0123
34.257	2.616	3	0	4	−1	34.261	2.615	0.0046
34.953	2.565	2	1	4	0	34.934	2.566	−0.0183
35.093	2.555	4	2	1	1	35.096	2.555	0.0035
35.865	2.502	2	1	2	2	35.860	2.502	−0.0052
36.550	2.456	4	1	4	−1	36.554	2.456	0.0034
36.824	2.439	1	1	3	−2	36.832	2.438	0.0077
37.277	2.410	3	2	1	−2	37.270	2.411	−0.0062
38.011	2.365	3	2	3	0	38.014	2.365	0.0036
38.271	2.350	3	1	4	1	38.260	2.351	−0.0106
38.706	2.324	2	2	3	−1	38.704	2.325	−0.0024
39.821	2.262	2	2	2	−2	39.821	2.262	0.0005
41.429	2.178	2	0	4	2	41.427	2.178	−0.002
42.589	2.121	3	1	5	0	42.559	2.123	−0.0298
42.870	2.108	1	0	2	−3	42.863	2.108	−0.0067
44.305	2.043	1	2	4	−1	44.303	2.043	−0.0023
44.646	2.028	1	3	0	−1	44.641	2.028	−0.0047
45.389	1.997	2	3	1	−1	45.390	1.996	0.0016
45.503	1.992	3	3	1	0	45.517	1.991	0.0146
46.675	1.944	2	2	1	−3	46.679	1.944	0.0038
49.255	1.848	1	3	1	−2	49.252	1.849	−0.0033
50.217	1.815	2	2	5	0	50.216	1.815	−0.0006
50.598	1.803	2	1	6	0	50.590	1.803	−0.0079
50.751	1.797	3	2	5	−1	50.770	1.797	0.019
53.403	1.714	2	2	5	1	53.399	1.714	−0.0049
53.764	1.704	2	0	0	4	53.768	1.704	0.004
54.972	1.669	1	3	3	1	54.969	1.669	−0.0028
56.549	1.626	1	1	6	−2	56.584	1.625	0.0344
59.084	1.562	1	3	4	−2	59.068	1.563	−0.0161

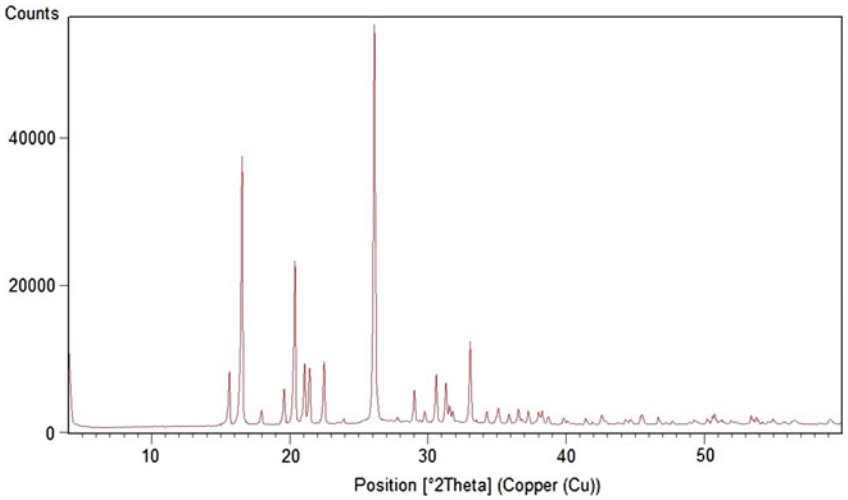


Figure 2. X-ray powder diffraction pattern of TMC using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

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Boultif, A. and Louër, D. (**2004**). "Powder pattern indexing with the dichotomy method," J. Appl. Crystallogr. **37**, 724–731.

de Wolff, P. M. (**1968**). "A simplified criterion for the reliability of a powder pattern indexing," J. Appl. Crystallogr. **1**, 108–113.

Kataeva, O. N., Litvinov, I. A., and Naumov, V. A. (**1988**). "Molecular structure of trimethylene carbonate in the crystalline phase," Zh. Strukt. Khim. (J. Struct. Chem.) **29**, 653–655 (in Russian).

Nair, L. S. and Laurencin, C.T. (**2007**). "Biodegradable polymers as biomaterials," Progr. Polym. Sci. **32**, 762–798.

Smith, G.S. and Snyder, R. L. (**1979**). "F_N: a criterion for rating powder diffraction patterns and evaluating the reliability of powder indexing," J. Appl. Crystallogr. **12**, 60–65.

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