

The chemical preparation as well as crystallization is described in the main body of the article.

The measured sample of the deposited structure was prepared after immersion of the crystal into the liquid nitrogen, letting to obtain room temperature followed by mechanical separation of a uniform crystal block from the batch of the crystals. The data collection was carried out in different temperatures from the room temperature down to 95 K which was followed by the increase of the temperature and by repeated data collection. Here is reported the second measurement at the room temperature after previous cooling and heating.

This crystal is different from that which served for the data collection of the phases II and III which are reported in the main body of the article.

Experimental details:

	Phase I
Crystal data	
Chemical formula	$C_{18}H_{30}BaCa_2O_{12}$
M_r	655.9
Crystal system, space group	Orthorhombic, <i>Pnma</i>
Hall symbol	- <i>P 2ac 2n</i>
a, b, c (Å)	18.1858(6), 18.1858(6), 18.1858(6)
V (Å ³)	6014.5(3)
Z	8
$F(000)$	2640
D_x (Mgm ⁻³)	1.4488
Radiation type	Mo $K\alpha$
Number of diffractions for determination of the unit-cell parameters	7090
Θ range (°)	3.16-24.16
μ (mm ⁻¹)	1.709
Temperature (K)	281

Habitus, colour	Plate, colourless
Crystal size (mm)	0.285 × 0.145 × 0.083
Data collection	
Diffractometer	SuperNova, Dual, Cu at home/near, AtlasS2
Radiation source	X-ray tube
monochromatization	mirror
Measurement mode	φ and ω scans
Absorption correction	multi-scan <i>CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018)</i>
T_{\min} , T_{\max}	0.75096, 1.00
No. of measured, independent and observed [$I > 3\sigma(I)$] reflections	11099, 270, 245
R_{int}	0.028
θ_{max} (°)	24.55
h,k,l range	$h = -20 \rightarrow 20$ $k = -20 \rightarrow 20$ $l = -20 \rightarrow 20$
Refinement on F^2	
$R[I > 3\sigma(I)]$, $wR(I)$, S	0.0217, 0.0533, 2.31
No. of reflections	270
No. of parameters	38
No. of restraints	4
No. of constraints	8
Special treatment	C2 and C3 were treated unharmonically up to the 3 rd order
H-atom treatment	The methylene H constrained, the methyl hydrogens undetermined
Weighting scheme	$1/(\sigma^2(I) + 0.0004I^2)$

$(\Delta/\sigma)_{\max}$	0.0495
$\Delta\rho_{\max}, \Delta\rho_{\min}$ ($e \text{ \AA}^{-3}$)	0.25, -0.20
Extinction correction	-
Extinction coefficient	-
Absolute structure	-
Absolute structure parameter:	-

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018) *SHELXT* (Sheldrick, 2015), *JANA2006* (Petříček *et al.*, 2014).

Refinement:

The positions of the non-hydrogen atoms were determined by the program *SHELXT* (Sheldrick, 2015) and by the calculations of the subsequent difference electron density maps which determined the positions of the disordered atoms more reliably. The refinement was carried out by the program *JANA2006* (Petříček *et al.*, 2014). C2 and C3 were treated anharmonically up to the third order (Petříček & Dušek, 2000).

The distances between the carboxyl carbon- C^α and C^α - C^β were restrained to 1.506 and 1.480 Å, respectively, with the weight 0.002 Å. O1-C1 distance was restrained to 1.245(2). This value was derived from the structure determinations of bis(guanidinium) oxalate monohydrate (Andrews, 2019, refcode KORDED) and of 3,5-dimethyladamantan-1-aminium hydrogen ethanedioate (Bhardwaj *et al.*, 2020, refcode KUBSAE). The angle $C_{\text{carboxyl}}-C^\alpha-C^\beta$ was restrained to the value 109.47° with the weight 0.01. The positions of the methylene hydrogens were calculated and constrained by the value $C_{\text{methylene}}-H_{\text{methylene}} = 0.97 \text{ \AA}$ while $U_{\text{iso}}(H_{\text{methylene}}) = 1.2U_{\text{eq}}(C_{\text{methylene}})$. The methyl hydrogens were not determined.

(The refcodes are pertinent to the Cambridge Structural Database, Groom *et al.*, 2016; version 5.41 from November 2019 with updates until August 2020.)

References regarding the supplementary material:

Andrews, L.C. (2019). CSD communication (private communication).

Bhardwaj V., Shaiwale, M., Lakhani, B. & Ballabh, A. (2020). *J. Mol. Struct.* **1206**, 127672.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.

Petříček, V., Dušek, M. (2000). Manual for the Crystallographic Computing System JANA98, p. 122. Institute of Physics, Academy of Sciences of the Czech Republic.

Petříček, V., Dušek, M. & Palatinus, L. (2014). *Z. Kristallogr.* **229**, 345-352.

Rigaku, Oxford Diffraction (2019). *CrysAlisPro*, version 1.171.40.53.

Sheldrick, G. M. (2015). *Acta Cryst.* **A71**, 3-8.