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Crystal structure of bis(acridin-10-ium) 2,5-dihydroxyterephthalate – 2,5-dihydroxyterephthalic acid (1/1), C₂₁H₁₅NO₆

Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.26 × 0.23 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	25.5°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10752, 3060, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2499
$N(\text{param})_{\text{refined}}$:	256
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

<https://doi.org/10.1515/ncrs-2019-0377>

Received May 28, 2019; accepted August 14, 2019; available online September 17, 2019

Abstract

C₂₁H₁₅NO₆, triclinic, $P\bar{1}$ (no. 2), $a = 9.3591(4)$ Å, $b = 9.4371(5)$ Å, $c = 10.1106(5)$ Å, $\alpha = 102.581(4)^\circ$, $\beta = 92.929(4)^\circ$, $\gamma = 102.573(4)^\circ$, $V = 846.09(7)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0452$, $wR_{\text{ref}}(F^2) = 0.1135$, $T = 288.7(3)$ K.

CCDC no.: 1947088

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

In a typical experiment, 2,5-dihydroxyterephthalic acid (0.02 g, 0.1 mmol) is dissolved in 5 mL of absolute ethanol, acridine (0.02 g, 0.1 mmol) is dissolved in another 5 mL of absolute ethanol. The two solutions are mixed. The mixture was left at room temperature for 10 minutes to get red block crystals of the title compound.

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Experimental details

The hydrogen atoms were placed at calculated positions with the SHELX program (AFIX options: 43 and 147).

Comment

Over the past few decades, the design and assembly of organic cocrystals molecular materials has gained considerable interest [4–6] due to their potential applications such as luminescence and polarized emission [7, 8], luminescent sensors [9, 10] and tunable mechanical properties [11]. Synthesis methods of organic cocrystals mainly include solution processes [12, 13], vacuum sublimation [14] and external stimuli [15]. The most frequently used method for preparing cocrystals is a solution process and single crystals are grown by slow evaporation of the solvents. But it is still difficult to predict the generation of a new cocrystal. In order to further study the formation conditions of cocrystals, in this article, we use 2,5-dihydroxyterephthalic acid (H₂diph) and acridine (AD) to synthesize a cocrystal.

The asymmetric unit of the cocrystal contains one acridinium cation, one half of a 2,5-dihydroxyterephthalic acid molecule and one half 2,5-dihydroxyterephthalate dianion. In the cocrystal, π - π intermolecular interactions between benzene rings of AD molecules and pyridine rings of AD molecules (3.722 Å), benzene rings of H₂diph molecules and benzene rings of AD molecules (3.593 Å), and hydrogen bonding from O(5)–H(5)···O(1)^a (distance: 2.49 Å, angle: 174°), C(6)–H(6)···O(3) (distance: 3.16 Å, angle: 133°), and N(1)–H(1)···O(2)^b (distance: 2.67 Å, angle: 169°) form a

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
N1	0.41853(16)	0.38719(18)	0.79478(15)	0.0292(4)
H1	0.4915	0.3939	0.7463	0.035*
C1	0.4759(2)	0.6554(2)	0.8570(2)	0.0345(5)
H1A	0.5537	0.6609	0.8032	0.041*
C2	0.4447(2)	0.7815(2)	0.9303(2)	0.0420(5)
H2	0.5022	0.8739	0.9262	0.050*
C3	0.3272(3)	0.7760(2)	1.0126(2)	0.0433(6)
H3	0.3092	0.8643	1.0627	0.052*
C4	0.2404(2)	0.6430(2)	1.0191(2)	0.0385(5)
H4	0.1628	0.6408	1.0731	0.046*
C5	0.2671(2)	0.5063(2)	0.94356(18)	0.0296(4)
C6	0.1825(2)	0.3661(2)	0.94405(19)	0.0326(5)
H6	0.1010	0.3589	0.9933	0.039*
C7	0.2163(2)	0.2362(2)	0.87274(19)	0.0309(5)
C8	0.1334(2)	0.0897(2)	0.8707(2)	0.0430(5)
H8	0.0504	0.0780	0.9177	0.052*
C9	0.1739(3)	−0.0322(3)	0.8013(2)	0.0479(6)
H9	0.1182	−0.1270	0.8002	0.057*
C10	0.3009(3)	−0.0164(2)	0.7303(2)	0.0451(6)
H10	0.3284	−0.1015	0.6846	0.054*
C11	0.3831(2)	0.1201(2)	0.7276(2)	0.0371(5)
H11	0.4662	0.1287	0.6804	0.045*
C12	0.3410(2)	0.2495(2)	0.79748(18)	0.0287(4)
C13	0.3882(2)	0.5152(2)	0.86381(18)	0.0277(4)
O1	0.60291(14)	0.34549(17)	0.44697(14)	0.0433(4)
O2	0.66247(14)	0.43920(16)	0.67028(14)	0.0384(4)
O3	0.92840(15)	0.57414(19)	0.76347(13)	0.0462(4)
H3A	0.8400	0.5432	0.7666	0.069*
C14	0.89348(19)	0.4251(2)	0.39358(18)	0.0268(4)
H14	0.8218	0.3749	0.3217	0.032*
C15	0.85271(19)	0.45881(19)	0.52453(17)	0.0236(4)
C16	0.6944(2)	0.4114(2)	0.54846(19)	0.0282(4)
C17	0.9612(2)	0.5355(2)	0.63228(17)	0.0264(4)
O4	−0.13293(16)	0.18322(16)	0.67900(16)	0.0454(4)
H4A	−0.2104	0.1351	0.6972	0.068*
O5	0.33512(14)	0.26655(15)	0.47314(15)	0.0378(4)
H5	0.4230	0.2864	0.4628	0.057*
O6	0.33634(17)	0.05419(18)	0.32518(18)	0.0578(5)
C18	0.13156(19)	0.0637(2)	0.45407(18)	0.0267(4)
C19	0.0605(2)	0.1523(2)	0.54319(19)	0.0284(4)
H19	0.1009	0.2545	0.5717	0.034*
C20	−0.0696(2)	0.0910(2)	0.59015(19)	0.0290(4)
C21	0.2764(2)	0.1284(2)	0.4111(2)	0.0320(5)

three-dimensional structure. The cocrystal structure is similar to the structures of cocrystals reported by Christian Wolf's group [16]. They reported a series of cocrystals by acridine and dicarboxylic acids. All these cocrystal molecules are stabilized by π -stacking and hydrogen bonding.

Acknowledgements: This work was supported by key scientific research projects of higher education of Henan Province (16 A150016) and Henan Province Natural Science Foundation (182300410166).

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