Crystal structure of (*S*)-2-(1,3-dioxo-1,3-dihydro-isoindol-2-yl)-propionic acid methyl ester, C₈H₄NO₂[CH(CH₃)COOCH₃]

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Table 1. Data collection and handling.

Crystal:	colorless plate, size 1.05 x 0.65 x 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.00 cm ⁻¹
Diffractometer, scan mode:	AXS BRUKER P4, ω
$2\theta_{\max}$:	55°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	5519, 5263
Criterion for F_{obs} , $N(hkl)_{\text{gt}}$:	$F_{\text{obs}} > 3 \sigma(F_{\text{obs}})$, 4307
$N(\text{param})_{\text{refined}}$:	306
Program:	SHELXTL-plus [5]

Abstract

C₁₂H₁₁NO₄, monoclinic, $P12_11$ (No. 4), $a = 11.533(1)$ Å, $b = 7.012(1)$ Å, $c = 15.438(2)$ Å, $\beta = 111.68(1)^\circ$, $V = 1160.2$ Å³, $Z = 4$, $R_g(F) = 0.064$, $R_w(F) = 0.064$, $T = 293$ K.

Source of material

The title compound was prepared from (*S*)-*N*-phthaloyl alanine [1] by refluxing in methanol in the presence of catalytic amounts of sulfuric acid [2]. Extraction and recrystallization from acetone yielded 91% of colorless crystals.

Discussion

This compound was prepared in context with diastereoselectivity problems in intermolecular photoreactions between enantiomeric pure *N*-phthaloyl amino acid methyl esters and potassium carboxylates [3]. In analogy to the intramolecular photoreactions [4] photodecarboxylation is the dominant process for product formation.

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(14)	2a	0.3362(3)	0.9414(7)	0.3614(3)	0.08
H(15)	2a	0.4728(3)	0.9217(8)	0.2828(3)	0.08
H(16)	2a	0.4037(4)	0.9524(9)	0.1242(4)	0.08
H(17)	2a	0.1928(3)	1.0171(8)	0.0330(3)	0.08
H(110)	2a	-0.1640(3)	1.0406(7)	0.2446(3)	0.08
H(11A)	2a	-0.5096(4)	0.756(1)	0.0307(4)	0.08
H(11B)	2a	-0.4077(4)	0.617(1)	0.0954(4)	0.08
H(11C)	2a	-0.3998(4)	0.688(1)	0.0014(4)	0.08
H(11D)	2a	-0.3074(4)	1.2248(9)	0.1425(4)	0.08
H(11E)	2a	-0.2239(4)	1.2440(9)	0.0826(4)	0.08
H(11F)	2a	-0.1783(4)	1.3294(9)	0.1835(4)	0.08
H(24)	2a	0.2061(3)	1.4586(8)	0.4190(3)	0.08
H(25)	2a	0.0291(3)	1.5125(8)	0.2825(3)	0.08
H(26)	2a	0.0429(3)	1.5304(8)	0.1376(3)	0.08
H(27)	2a	0.2328(3)	1.4854(7)	0.1202(3)	0.08
H(210)	2a	0.6890(3)	1.3463(8)	0.4764(3)	0.08
H(21A)	2a	1.0112(4)	1.650(1)	0.4502(4)	0.08
H(21B)	2a	0.9130(4)	1.774(1)	0.4730(4)	0.08
H(21C)	2a	0.8997(4)	1.743(1)	0.3692(4)	0.08
H(21D)	2a	0.7953(4)	1.1767(9)	0.4109(4)	0.08
H(21E)	2a	0.6853(4)	1.1917(9)	0.3140(4)	0.08
H(21F)	2a	0.6663(4)	1.0823(9)	0.3963(4)	0.08

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(11)	2a	-0.0308(2)	1.0309(7)	0.1888(2)	0.050(1)	0.076(2)	0.061(2)	-0.002(1)	0.018(1)	0.001(2)
C(12)	2a	0.0711(3)	1.0004(7)	0.2709(2)	0.061(2)	0.062(2)	0.057(2)	-0.012(2)	0.016(2)	-0.000(2)
O(12)	2a	0.0661(2)	0.9918	0.3471(2)	0.086(2)	0.116(3)	0.058(1)	-0.014(2)	0.029(1)	0.009(2)
C(13)	2a	0.1806(3)	0.9856(6)	0.2422(2)	0.057(2)	0.046(2)	0.057(2)	-0.005(2)	0.013(1)	-0.002(2)

Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(14)	2a	0.3066(3)	0.9546(7)	0.2948(3)	0.058(2)	0.064(2)	0.077(2)	−0.006(2)	0.004(2)	0.008(2)
C(15)	2a	0.3857(3)	0.9437(8)	0.2485(3)	0.044(2)	0.073(3)	0.112(3)	−0.004(2)	0.011(2)	−0.002(3)
C(16)	2a	0.3444(4)	0.9635(9)	0.1542(4)	0.081(3)	0.084(3)	0.143(4)	0.005(3)	0.067(3)	−0.008(3)
C(17)	2a	0.2205(3)	0.9986(8)	0.0992(3)	0.076(2)	0.089(3)	0.077(2)	0.001(2)	0.036(2)	−0.007(2)
C(18)	2a	0.1408(3)	1.0051(7)	0.1467(2)	0.056(2)	0.062(2)	0.059(2)	−0.003(2)	0.017(2)	−0.003(2)
C(19)	2a	0.0014(3)	1.0336(7)	0.1109(2)	0.058(2)	0.077(2)	0.051(2)	−0.002(2)	0.009(2)	0.001(2)
O(19)	2a	−0.0691(2)	1.0504(8)	0.0327(2)	0.072(2)	0.165(3)	0.057(2)	0.002(2)	0.004(1)	0.005(2)
C(110)	2a	−0.1601(3)	1.0395(7)	0.1835(3)	0.058(2)	0.072(2)	0.076(2)	−0.003(2)	0.026(2)	−0.007(2)
C(111)	2a	−0.2254(4)	0.8597(7)	0.1360(3)	0.055(2)	0.070(3)	0.086(3)	0.001(2)	0.024(2)	−0.000(2)
O(111)	2a	−0.1751(3)	0.7140(7)	0.1311(3)	0.061(2)	0.076(2)	0.163(3)	0.010(2)	0.024(2)	−0.022(2)
O(112)	2a	−0.3490(2)	0.8853(7)	0.1008(2)	0.050(1)	0.083(2)	0.139(3)	−0.002(1)	0.028(2)	−0.025(2)
C(112)	2a	−0.4227(4)	0.723(1)	0.0531(4)	0.068(3)	0.112(4)	0.215(7)	−0.017(3)	0.051(4)	−0.062(4)
C(113)	2a	−0.2232(4)	1.2265(9)	0.1444(4)	0.061(3)	0.084(3)	0.140(4)	0.000(2)	0.029(3)	0.002(3)
N(21)	2a	0.5304(2)	1.3990(7)	0.3676(2)	0.045(1)	0.093(2)	0.065(2)	−0.008(2)	0.014(1)	0.001(2)
C(22)	2a	0.4526(3)	1.4120(8)	0.4178(3)	0.058(2)	0.083(3)	0.061(2)	−0.009(2)	0.016(2)	−0.001(2)
O(22)	2a	0.4858(3)	1.3982(8)	0.5017(2)	0.071(2)	0.151(3)	0.058(2)	−0.010(2)	0.011(1)	−0.005(2)
C(23)	2a	0.3242(3)	1.4459(7)	0.3472(2)	0.052(2)	0.061(2)	0.061(2)	−0.006(2)	0.011(1)	−0.003(2)
C(24)	2a	0.2126(3)	1.4657(8)	0.3588(3)	0.062(2)	0.082(3)	0.082(2)	−0.008(2)	0.032(2)	−0.018(2)
C(25)	2a	0.1092(3)	1.4969(8)	0.2777(3)	0.052(2)	0.078(3)	0.093(3)	−0.002(2)	0.020(2)	−0.020(2)
C(26)	2a	0.1168(3)	1.5065(8)	0.1914(3)	0.055(2)	0.068(3)	0.091(3)	0.006(2)	0.003(2)	−0.010(2)
C(27)	2a	0.2277(3)	1.4821(7)	0.1809(3)	0.068(2)	0.062(2)	0.063(2)	0.002(2)	0.007(2)	0.003(2)
C(28)	2a	0.3330(3)	1.4526(7)	0.2600(2)	0.053(2)	0.059(2)	0.057(2)	−0.004(2)	0.010(1)	−0.002(2)
C(29)	2a	0.4661(3)	1.4235(7)	0.2706(3)	0.055(2)	0.070(2)	0.071(2)	0.000(2)	0.021(2)	0.005(2)
O(29)	2a	0.5114(2)	1.4184(7)	0.2129(2)	0.076(2)	0.123(3)	0.075(2)	0.000(2)	0.039(1)	0.014(2)
C(210)	2a	0.6645(3)	1.3681(8)	0.4106(3)	0.050(2)	0.097(3)	0.075(3)	−0.007(2)	0.011(2)	0.003(2)
C(211)	2a	0.7285(3)	1.5510(8)	0.3967(3)	0.050(2)	0.088(3)	0.090(3)	−0.003(2)	0.021(2)	−0.007(2)
O(211)	2a	0.6817(3)	1.7008(8)	0.3709(4)	0.074(2)	0.077(2)	0.239(5)	0.011(2)	0.038(3)	0.007(3)
O(212)	2a	0.8511(2)	1.5185(7)	0.4248(2)	0.049(1)	0.102(2)	0.104(2)	−0.002(2)	0.023(1)	0.015(2)
C(212)	2a	0.9248(4)	1.685(1)	0.4296(4)	0.073(3)	0.113(4)	0.177(6)	−0.015(3)	0.056(3)	0.003(4)
C(213)	2a	0.7066(4)	1.1889(9)	0.3802(4)	0.068(3)	0.083(3)	0.116(4)	0.002(2)	0.009(3)	−0.001(3)

References

1. Nefkens, G. H. L.; Tesser, G. I.; Nivard, R. J. F.: A simple preparation of phthaloyl amino acids via a mild phthaloylation. *Recl. Trav. Chim. Pays-Bas*. **79** (1960) 688–698.
2. Crippa, G. B.; Galimberti, P.: Sulla ftaloilazione. *Gazz. Chim. Ital.* **63** (1933) 81–96.
3. Oelgemöller, M.: Dissertation planned for 1999, University of Cologne, Germany
4. Griesbeck, A. G.; Henz, A.; Kramer, W.; Nerowski, F.; Oelgemöller, M.; Peters, K.; Peters, E.-M.: Synthesis of medium- and large-ring compounds initiated by photochemical decarboxylation of ω -phthalimido alkylcarboxylates. *Helvetica Chim. Acta* **80** (1997) 912–933.
5. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.