Crystal and molecular structure of (exo)-2-methoxycarbonyl-5-ethoxycarbonyl-4,5,6,11b-tetrahydroisoxazolidino-[2,3-a]-β-carboline, C₁₈H₂₀N₂O₅

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Abstract

The structure of the title compound, C₁₈H₂₀N₂O₅, was determined by X-rays at T = 290 K. Mᵣ = 344.366, monoclinic, space group P2₁/c, a = 13.7850(8), b = 8.8951(7), c = 15.1603(11) Å, β = 111.410(6)°, Vᵣ = 1730.7 Å³, Z = 4, Dₓ = 1.322 Mgm⁻³. Cu Kα radiation (graphite crystal monochromator, λ = 1.54178 Å), μ(Cu Kα) = 7.69 cm⁻¹. Final conventional R-factor = 0.057, R_w = 0.076 for 2160 "observed" reflections and 271 variables. The structure was solved using MULTAN.

Introduction

The cycloaddition reaction of a nitrone to a variety of alkenes was investigated recently (Plate et al., 1986), one of these being methyl acrylate. This reaction gave a mixture of four stereomers in a ratio of 12:52:31:5. For a mechanistic rationale of this cycloaddition in terms of regio- and stereoselectivity we had to determine the structure of these isomers. The structure of the major isomer (52%, denoted PEDRCC) is reported in a foregoing paper (Smits et al., 1986a) and we refer to this paper for a more extensive introduction.
Here we report the crystal structure of another isomer (31% to be denoted PEDROC). It was isolated and recrystallized from ethyl acetate/n-hexane (mp 165–167°C). This isomer could be assigned structure 1.

![Chemical Structure](image)

**Experimental**

All measurements were made at $T = 290$ K. An irregularly shaped crystal of approximately $0.12 \times 0.16 \times 0.55$ mm was used for the measurements. Throughout the experiment Cu Kα radiation was used with a graphite crystal monochromator on a Nonius CAD4 single-crystal diffractometer ($\lambda = 1.54178$ Å). The unit cell dimensions, $a = 13.7850(8)$, $b = 8.8951(7)$, $c = 15.1603(11)$ Å, $\beta = 111.410(6)^\circ$, $V_c = 1730.7$ Å$^3$, were determined from the angular settings of 25 reflections with $23 < \theta < 33^\circ$. The space group was determined to be $P2_1/c$ from the systematic absences $0k0: k = 2n + 1$, $h0l: l = 2n + 1$ and the structure determination, $Z = 4$, $D_x = 1.322$ Mg m$^{-3}$, $F(000) = 728$. The intensity data of 7018 reflections (half a sphere up to $\theta = 70^\circ$) were measured, using the $\omega-2\theta$ scan technique, with a scan angle of 1.50° and a variable scan rate with a maximum scan time of 15 s per reflection. The intensity of the primary beam was checked throughout the data collection by monitoring three standard reflections every 30 min. The final drift correction factors were between 1.00 and 1.03. On all reflections profile analysis was performed (Lehman and Larsen, 1974; Grant and Gabe, 1978); empirical absorption correction was applied, using psi-scans (North et al., 1968), $\mu(Cu K\alpha) = 7.69$ cm$^{-1}$ (correction factors were in the range 0.95–1.00). Lorentz and polarization corrections were applied and the data were reduced to $|F_o|$ values. Symmetry-equivalent reflections were averaged, $R_{int} = \Sigma(F - \langle F \rangle)/\Sigma F = 0.032$ for all reflections and 0.015 for the observed reflections only, resulting in 3284 unique reflections of which 2160 were observed with $F > 3\sigma(F)$.

The structure was solved using MULTAN (Main et al., 1980), which gave all the nonhydrogen atoms. Isotropic least-squares refinement, using SHELX