CRYSTAL STRUCTURE OF THE PHOTOCYCLOADDUCT OF 1-[4'-BROMOPHENYL]-2-PHENYL-3-ETHOXYCARBONYL-Δ^2-PYRROLINE-4,5-DIONE WITH STYRENE[1]

Takehiro Sano and Yoshisuke Tsuda*
Showa College of Phamaceutical Sciences, Setagaya-ku, Tokyo-154, Japan

Haruo Ogura* and Kimio Furuhata
School of Phamaceutical Sciences, Kitasato University, Minato-ku, Tokyo-108, Japan

Yoichi Iidaka
Faculty of Pharmaceutical Sciences, University of Tokyo, Bunkyo-ku, Tokyo-103, Japan

The structure of the cycloadduct of 1-[4'-bromophenyl]-2-phenyl-3-ethoxycarbonyl-Δ^2-pyrroline-4,5-dione with styrene was determined by X-ray analysis using a heavy atom method.

In preceding paper we showed that 2-phenyl-3-ethoxycarbonyl-Δ^2-pyrroline-4,5-dione reacts with the olefins carrying electron rich substituents to give [2+2] cycloadducts[2]. For determination of regio- and stereochemistry of the products, we chose the cycloadduct of 1-[4'-bromophenyl]-2-phenyl-3-ethoxycarbonyl-Δ^2-pyrro-

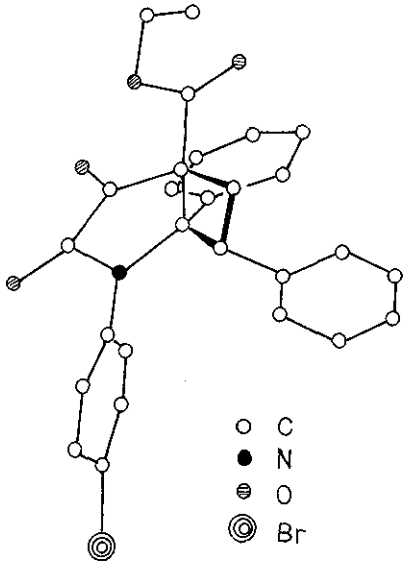
line-4,5-dione with styrene and undertook its X-ray analysis. The desired cycloadduct was obtained in about 20% yield by irradiation of the two reactants with 300W high pressure mercury lamp in dioxane-ether (1:1) for 60 min and formed single crystals by crystallization from methanol.

The crystals are colorless prisms. The cell dimensions were obtained by means of the least-squares method. The crystal data were collected on a Rigaku Denki Computer controlled four-circle diffractometer, using CuKα radiation. All the intensities were obtained from a crystal with dimention 0.25 × 0.20 × 0.20 mm. Reflections with 2θ values up to 140° of (hkl), (hk$\bar{l}$), ($\bar{h}$kl), and ($\bar{h}$k$\bar{l}$) were measured by ω-2θ scanning technique at a scan rate of 2° per minute. The scan range for each reflection was calculated by the formula $\omega = 1.10^{\circ} + 0.5^{\circ} \tan\theta$ and the backgrounds were measured at both ends of the scan range for 10.0 seconds. The instensity data were corrected for the background count and for the usual Lorentz and polarization effects. A total of 1982 independent non-zero reflections were measured visually.

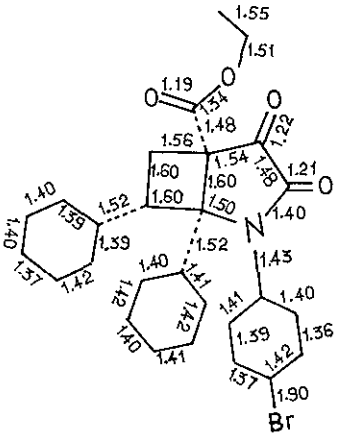
The crystal belongs to the triclinic system, with two formula units in a cell with dimensions of; a=11.384, b=11.851, c=9.321 Å, α=109.49, β=91.91, γ=103.06°. The space group is p $\bar{1}$. The structure has been determined by the Patterson synthesis, a series of the Fourier synthesis and the least-squares methods. The final R value is 0.085 for 1929 observed reflections.

From the determined structure of the cycloadduct we can not

elucidate the stereochemical course of this reaction whether it would proceed in 2a+2s manner with gaining maximum overlapping of two reactants or in 2s+2s manner by steric repulsion between phenyl group and dioxopyrroline ring. We however prefer the former process since the reaction seems to be governed by donor-acceptor interaction[2,3].



C-axis projected



Bond distances (Å)

REFERENCES

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2 See Part II.

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