

Versuche zur Messung des Betrages der Zirkularpolarisation sollen durchgeführt werden. Lösungen der Verbindung in $C_2H_4(OH)_2$ sind optisch inaktiv.

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cis-1-Acetyl-4-[1-(4-bromophenyl)-2-phenyl]-vinyl-3-pyrrolin-2-one

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Abstract. Monoclinic, $P2_1/c$, $a = 15.15$ (8), $b = 13.82$ (8), $c = 8.26$ (3) Å, $\beta = 90.5$ (2)°. $D_{\text{obs}} = 1.478$; $D_{\text{calc}}(C_{20}H_{16}BrNO_2, n = 4) = 1.467$ g cm⁻³.

Introduction. Three-dimensional data were collected to $\sin \theta/\lambda = 0.66$ Å⁻¹ on a card-controlled Enraf–Nonius three-circle diffractometer with nickel-filtered Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å) (*cf.*, Matthews, Stenkamp & Colman, 1973). Of 3729 reflections measured, 1429 were considered to have significant intensity [$I > 2\sigma(I)$]. The crystal measured approximately 0.1 mm in each direction and had an absorption coefficient $\mu(\text{Mo } K\alpha) = 25.3$ cm⁻¹.

Systematic absences determined from Weissenberg and Buerger precession photographs were $h0l$ for l odd, and $0k0$ for k odd. A Patterson map provided starting coordinates for the bromine atom, and a Fourier synthesis, calculated with heavy-atom phases, clearly showed the remainder of the non-hydrogen atoms.

Refinement by block-diagonal least-squares calculations, with isotropic temperature factors, resulted in an R value of 0.24. Anisotropic temperature factors improved R to 0.145 for the reflections with $I > 2\sigma(I)$. The maximum and average final shifts were 0.0014 and 0.0008 Å respectively for the bromine, and 0.019 and 0.005 Å respectively for the remaining non-hydrogen atoms. A difference map, using low-angle data ($\sin \theta/\lambda < 0.32$ Å⁻¹), did not clearly show all the hydrogen positions, and had positive and negative features up to 1 e Å⁻³ in the vicinity of the bromine atom, presumably

due to the neglect of absorption, but within these limitations confirmed the structure determination.

Discussion. In a study of the intramolecular Diels–Alder reaction, *N*-(*p*-bromophenyl)propargyl)-*cis*-cinnamamide in acetic anhydride gave two crystalline products (Hwang, 1973). The structure of one product, resulting from [4 + 2] cycloaddition has been characterized (McGuire, 1968), while the n.m.r. infrared, ultraviolet and mass spectra of the other product, arising from [2 + 2] cycloaddition, are consistent with at least four plausible structures. The X-ray structure determination has shown that the other product of the reaction is *cis*-1-acetyl-4-[1-(4-bromophenyl)-2-phenyl]-vinyl-3-pyrrolin-2-one. Presumably formation is achieved by the [2 + 2] cycloaddition of two π bonds [$\pi 2a + \pi 2s$], followed by conrotatory opening of the cyclobutene ring (Hwang, 1973). Additional details of the reaction will be described elsewhere.

Positional and thermal parameters for the non-hydrogen atoms are given in Table 1.* Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962).

The bond lengths and angles are illustrated in Fig. 1 which also gives the atomic numbering. The estimated

* The table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30555 (16 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional coordinates* ($\times 10^4$)The thermal parameters, B_{ij} ($\times 10^4$), are the coefficients in $\exp[-\sum B_{ij}h_ih_j]$.

	<i>x</i>	<i>y</i>	<i>z</i>	B_{11}	B_{22}	B_{33}	B_{33}	B_{13}	B_{12}
Br	10984 (2)	1675 (2)	10058 (4)	59 (1)	66 (2)	272 (5)	-6 (6)	-14 (4)	-2 (3)
C(2)	10050 (17)	1309 (16)	8524 (34)	53 (12)	36 (14)	255 (53)	-22 (39)	-110 (42)	-17 (19)
C(3)	10270 (12)	1227 (17)	6926 (31)	16 (8)	59 (15)	24 (50)	-119 (43)	38 (31)	12 (17)
C(4)	9571 (12)	1050 (20)	5793 (35)	10 (7)	76 (18)	29 (58)	-40 (51)	51 (32)	-42 (20)
C(5)	8746 (15)	928 (14)	6223 (23)	62 (12)	23 (10)	77 (30)	6 (27)	-53 (31)	-3 (18)
C(6)	8530 (12)	996 (17)	7928 (28)	23 (8)	55 (14)	161 (41)	-8 (38)	16 (28)	-11 (18)
C(7)	9233 (14)	1186 (16)	9074 (28)	34 (10)	46 (13)	163 (40)	-11 (38)	-42 (31)	-14 (18)
C(8)	8000 (13)	722 (15)	5155 (23)	37 (9)	48 (13)	84 (31)	6 (32)	-42 (27)	17 (18)
C(9)	7968 (16)	1160 (17)	3732 (35)	42 (12)	40 (13)	259 (55)	12 (44)	-1 (40)	1 (19)
C(10)	7376 (12)	1052 (15)	2346 (23)	23 (8)	51 (13)	86 (30)	58 (32)	34 (24)	22 (17)
C(11)	7088 (16)	106 (15)	1775 (24)	69 (14)	35 (12)	81 (32)	-78 (32)	3 (33)	17 (21)
C(12)	6452 (20)	26 (20)	564 (30)	76 (17)	69 (18)	130 (43)	-40 (44)	22 (42)	-42 (28)
C(13)	6116 (18)	827 (20)	-246 (29)	68 (15)	71 (17)	121 (42)	-38 (41)	-14 (38)	7 (27)
C(14)	6376 (14)	1796 (19)	221 (20)	39 (10)	113 (20)	-3 (22)	10 (34)	88 (23)	-86 (24)
C(15)	7001 (13)	1845 (17)	1454 (28)	25 (9)	58 (14)	16 (41)	-46 (37)	82 (29)	16 (18)
C(16)	7310 (13)	19 (13)	5629 (21)	44 (10)	26 (10)	70 (27)	-35 (27)	4 (26)	44 (16)
C(17)	6452 (16)	133 (16)	5597 (28)	54 (12)	40 (13)	157 (41)	-66 (36)	-75 (36)	55 (21)
C(18)	6019 (12)	-741 (18)	6273 (29)	20 (8)	76 (17)	214 (47)	-60 (44)	-100 (33)	52 (19)
N(19)	6716 (11)	-1364 (11)	6749 (18)	45 (8)	36 (10)	66 (23)	6 (23)	4 (22)	-10 (14)
C(20)	7584 (16)	-974 (16)	6360 (28)	60 (13)	38 (12)	159 (41)	52 (36)	-97 (38)	-40 (21)
C(21)	6674 (14)	-2290 (17)	7509 (23)	50 (12)	58 (14)	66 (31)	26 (34)	-14 (30)	-21 (21)
C(22)	5724 (17)	-2704 (21)	7763 (37)	44 (12)	68 (18)	269 (60)	-116 (54)	2 (42)	6 (24)
O(23)	7343 (12)	-2662 (13)	8032 (24)	63 (10)	54 (11)	277 (41)	-77 (34)	-87 (32)	-5 (17)
O(24)	5235 (12)	-898 (16)	6499 (23)	52 (9)	112 (16)	193 (34)	96 (38)	-17 (28)	4 (21)

standard deviation in the Br-C(2) bond length is 0.025 Å and the average for bonds between non-hydrogen atoms is 0.031 Å. We attribute the relatively large uncertainties in the bond lengths partly to the small volume of the crystal [only 38% of the reflections had $I > 2\sigma(I)$], and also to the neglect of absorption. Since the primary objective of the study has been achieved, further refinement with the data available does not seem worth while. None of the intermolecular approaches are unusual. Neither the benzene, nor bromobenzene nor pyrrolinone ring is significantly aplanar; in fact no ring atom departs from the best-fit least-squares plane by more than a standard deviation, which is typically less than 0.025 Å. O(24) is 0.029 Å below, C(22) 0.045 Å above, and O(23) 0.255 Å below the plane through the five pyrrolinone ring atoms indicating that the acetyl group is 8° from parallel to the ring, this slight deviation presumably being due to steric interference between the methyl group at C(22) and the keto oxygen O(24) (distance 2.80 Å). The apparent resonance involving O(23), C(21), N(19), C(18) and O(24) is reflected in the shortening of the C(21)-N(19) and N(19)-C(18) bonds (1.427 and 1.415 Å), whereas the N(19)-C(20) bond (1.460 Å) retains a length close to 1.472 Å expected for a C-N single bond (*International Tables for X-ray Crystallography*, 1962).

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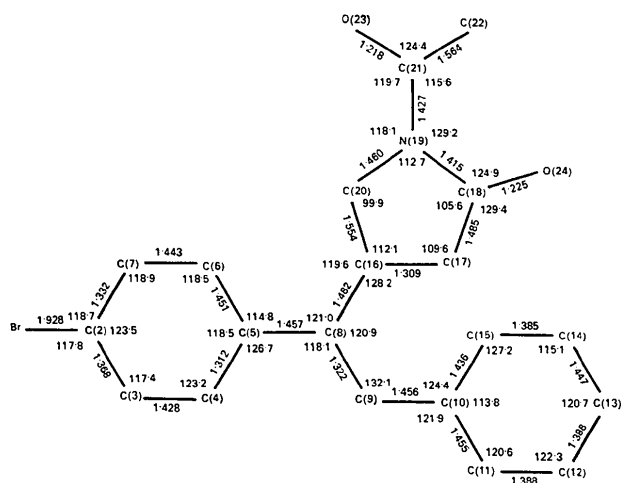


Fig. 1. Schematic diagram of the structure including atom numbering, bond lengths and bond angles.

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