STRUCTURE OF N-BENZYL-(2-CYANO-3-(2' FURYL)--ACRYLAMIDE)

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RESUMEN. La estructura de la N-benzil-(2-ciano-3-(2' furil)-acrilamida se determinó a partir de los datos de difracción de rayos X. Los cristales pertenecen al sistema triclínico, grupo espacial P1̄, con parámetros cristalográficos: a =9.8 05(2), b =27 .542(3), c =5.0 10(4) Å, α =95 .64(2), β =10 4.83(2) y γ =8 8.52(2)°,V =1 301.5(1.6) Å^3, Mm =25 2.3 g, μ =0.6 7 mm^{-1},T =2 93 K, siendo el número de unidades estructurales por celda Z =4. La estructura es de doble contenido con dos moléculas cristalográficamente independientes y químicamente equivalentes. La estructura fue resuelta por métodos directos y refinada a matriz completa por mínimos cuadrados utilizando el programa SHELXTL-Plus(1), alcanzando un valor final de R =0.074 (Rw =0.079) para 3 495 reflexiones y 209 parámetros refinados. Las moléculas en el cristal se empaquetan por fuerzas de van der Waals.

ABSTRACT. The structure of N-benzyl-(2-cyano-3-(2' furyl)-acrylamide has been determined from X-ray diffraction. Crystal are triclinic, space group P1̄ with a =9 .805(2), b = 27.542(3), c =5 .010(4) Å, α =95 .64(2), β =1 04.83(2) and γ =8 8.52(2)°, V =1 301.5(1.6) Å^3, Mw =25 2.3 g, m =0.6 7 mm^{-1},T= 293 K and Z =4. There are two crystallographically independent but chemically equivalent molecules present in the asymmetric unit. The structure was solved by direct methods and refined by full-matrix least-squares analysis using SHELXTL-Plus(1), with final Rb =0 .074 (wRb =0.0 79) for 3 495 reflections and 209 refined parameters. The molecules are packed at normal van der Waals distances.

INTRODUCTION

There is a great interest in studing the structure and properties of furanic derivatives because of their proven pharmacological activity and their use in chemical industry.[1] Although the biological activity of the title compound has not been fully tested, the substituents bound to the skeleton causes this compound to be structurally similar to compounds that are known to possess antitumoral activity.[2] In order to understand better the varied pharmacological activity of these compounds, The structural characteristics of a series of furanic derivatives were studied.[3]

The structure of the title compound is reported in the present paper. The compound was obtained using methods previously described.[6] The single crystal used in X-ray analysis was obtained by slow evaporation, at room temperature, from a mixture of ethanol and acetone 1:1.

EXPERIMENTAL

The colourless crystal of $C_{15}H_{12}N_2O_2$ to be used in the X-ray diffraction study were grown from slow evaporation, at room temperature, from a mixture of ethanol and acetone, 1:1. The crystal selected for data collection was a transparent, dimensions: (0.20X0.20X0.25) mm, and was mounted on a SIEMENS P3/PC diffractometer. Ni-filtered CuKα radiation was used. The unit-cell parameters were determined by least-squares fit of setting angles of 25 reflections. The 2θ/θ scan technique, $\theta_{max.}$ =5 5°, two control reflections were monitored every 100 measurements and showed no systematic variation of intensity. The number of the measured reflections including the controls and equivalents was 3 667 with -10 < h < 10, -29 <k<2 9a nd 0<l<5 . ($R_{int.}$= 0.059). The 3 658 reflections with $I \geq 3\sigma(I)$ and were used in the structure refinement. Only Lorentz-polarization corrections were applied.

The structure was solved by direct methods using the program SHELXTL-Plus (1).[5] The non-H atoms were refined anisotropically. The function minimized was $\Sigma w(|F_o| - |Fc|)^2$, where $w = (\sigma F^2 + 0.1185\ F^2)^{-1}$, final R = 0.074, weighed factor-of-merit (wR = 0.079) and goodness-of-fit (S = 0.95). For 209 parameters refined, the highest peak on the final dFmap was 0.72(3) eÅ^{-3}, the minimum -0.65(2) eÅ^{-3} . Computer programs used: Data collection: SIEMENS, cell refinement: SHELXTL-Plus(1),[5] data reduction SHELXTL-Plus(1),[5] program used to refine structure: SHELXTL-Plus(1).[5] Most of the calculation were performed on a Pentium-60c omputer, atomic scattering factors were taken from international tables for x-ray crystallography..[6]

RESULTS AND DISCUSSION

The fractional coordinates derived from the last cycle of refinement are presented in Table I. The thermal ellipsoids of each molecule with the atomic numbering scheme are shown in figure 1. Bond lengths are present in Table II, bond angles involving non-H atoms are given in Table III.

The packing of the molecules in the crystal is shown in Fig. 2. All rings are planar, the mean out-of plane e.s.d. being 0.004 Å for phenyl rings and 0.005 Å for furanic rings. In every single molecule the furanic rings is about 90° from phenyl rings.

Tables of anisotropic thermal parameters, hydrogen atoms coordinates, complete tables of geometrical details and observed and calculated structure factor are available on request by the author. The molecules are packed in the crystal by van der Waals forces.

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TABLE I

Atom coordinates (· 10^4) and temperature factors (Å^2 · 10^3)

Atom	x	y	z	U
C(1)a	8 183(8)	1 486(3)	3 113(17)	87(3)*
C(2)a	8 337(10)	1 052(3)	4 368(24)	103(4)*
C(3)a	9 362(13)	1 010(3)	6 813(27)	119(5)*
C(5)a	10 236(9)	1 382(3)	8 015(19)	97(3)*
C(6)a	10 089(6)	1 812(2)	6 740(13)	69(2)*
C(4)a	9 108(6)	1 874(2)	4 339(12)	62(2)*
C(13)a	8 693(6)	4 383(2)	13 392(13)	68(2)*
C(14)a	9 523(6)	4 802(2)	14 633(15)	75(2)*
C(15)a	10 683(7)	4 760(2)	13 599(15)	83(3)*
O(2)a	106 29(4)	4 374(1)	1 1752(9)	67(1)*
C(12)a	9 386(5)	4 140(2)	11 630(12)	58(2)*
N(1)a	9 587(5)	2 763(2)	4 651(9)	58(1)*
C(10)a	10 965(5)	3 602(2)	7 687(11)	51(2)*
C(9)a	9 642(5)	3 452(2)	8 124(10)	51(2)*
O(1)a	7 843(4)	2 855(1)	6 879(9)	67(1)*
C(8)a	8 959(5)	2 998(2)	6 477(11)	53(2)*
C(11)a	8 999(5)	3 702(2)	9 910(11)	55(2)*
C(7)a	8 952(7)	2 333(2)	2 926(12)	68(2)*
N(2)a	11 996(5)	3 695(2)	7 210(11)	64(2)*
C(1)b	15 922(6)	4 975(2)	6 093(13)	74(2)*
C(2)b	15 820(8)	5 405(2)	4 868(16)	85(3)*
C(3)b	14 800(7)	5 446(2)	2 414(16)	81(3)*
C(5)b	13 923(7)	5 070(3)	1 245(15)	83(3)*
C(6)b	14 008(5)	4 635(2)	2 474(13)	65(2)*
C(4)b	15 037(5)	4 585(2)	4 933(11)	57(2)*
C(13)b	15 507(6)	2 013(2)	–3 657(15)	73(2)*
C(14)b	14 737(8)	1 576(3)	–4 601(20)	100(3)*
C(15)b	13 662(8)	1 615(3)	–3 534(20)	98(3)*
O(2)b	13 645(4)	2 029(2)	–1 881(10)	80(2)*
C(12)b	14 841(5)	2 281(2)	–1 986(12)	57(2)*
O(1)b	16 227(4)	3 620(1)	2 387(8)	66(1)*
C(9)b	14 496(4)	2 992(2)	1 312(10)	49(2)*
N(1)b	14 525(5)	3 690(2)	4 657(9)	59(2)*
C(8)b	15 165(5)	3 459(2)	2 816(11)	54(2)*
C(11)b	15 154(5)	2 751(2)	–420(11)	57(2)*
C(7)b	15 179(6)	4 122(2)	6 394(11)	65(2)*
N(2)b	12 166(5)	2 750(2)	2 386(12)	76(2)*
C(10)b	13 230(6)	2 839(2)	1 889(11)	58(2)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U(i,j) tensor.

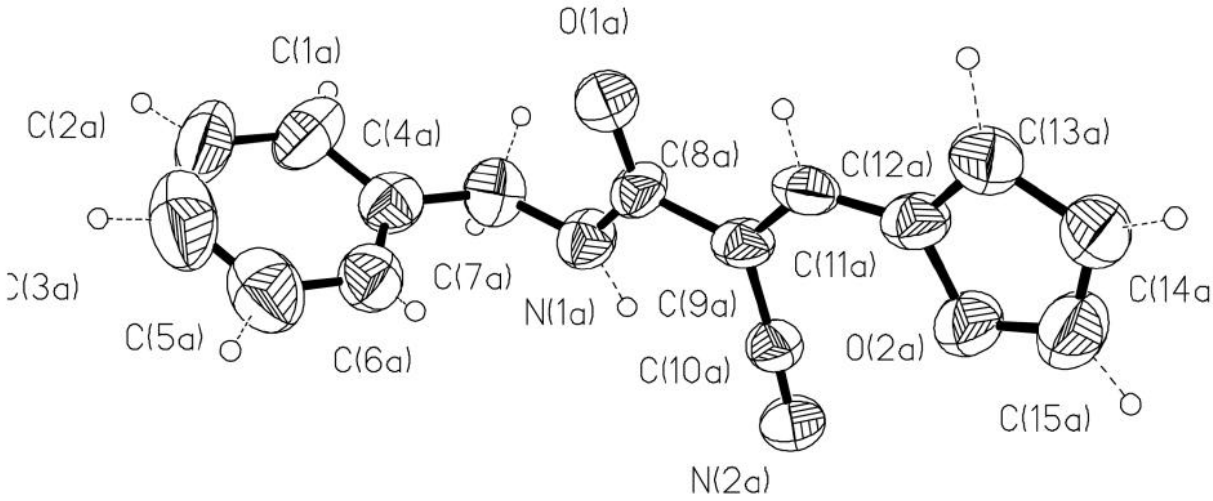

Fig. 1. View of the molecule A showing the numbering sheme adopted (1). Ellipsoids are scale to enclose 50 % probability. H atoms are represented as spheres of arbitrary radii.

TABLE II
Bond lengths (Å)

Atoms	Distances	Atoms	Distances
C(1)a-C(2)a	1.39(1)	C(1)a-C(4)a	1.406(8)
C(2)a-C(3)a	1.38(2)	C(3)a-C(5)a	1.34(1)
C(5)a-C(6)a	1.39(1)	C(6)a-C(4)a	1.357(7)
C(4)a-C(7)a	1.494(8)	C(13)a-C(14)a	1.425(8)
C(13)a-C(12)a	1.362(9)	C(14)a-C(15)a	1.36(1)
C(15)a-O(2)a	1.333(8)	O(2)a-C(12)a	1.378(6)
C(12)a-C(11)a	1.412(7)	N(1)a-C(8)a	1.336(7)
N(1)a-C(7)a	1.449(7)	C(10)a-C(9)a	1.446(7)
C(10)a-N(2)a	1.138(7)	C(9)a-C(8)a	1.500(6)
C(9)a-C(11)a	1.348(7)	O(1)a-C(8)a	1.243(7)
C(1)b-C(2)b	1.38(1)	C(1)b-C(4)b	1.382(8)
C(2)b-C(3)b	1.38(1)	C(3)b-C(5)b	1.350(9)
C(5)b-C(6)b	1.390(9)	C(6)b-C(4)b	1.395(7)
C(4)b-C(7)b	1.517(8)	C(13)b-C(14)b	1.412(9)
C(13)b-C(12)b	1.345(9)	C(14)b-C(15)b	1.30(1)
C(15)b-O(2)b	1.343(9)	O(2)b-C(12)b	1.394(7)
C(12)b-C(11)b	1.443(7)	O(1)b-C(8)b	1.219(7)
C(9)b-C(8)b	1.501(6)	C(9)b-C(11)b	1.326(8)
C(9)b-C(10)b	1.427(8)	N(1)b-C(8)b	1.344(7)
N(1)b-C(7)b	1.463(7)	N(2)b-C(10)b	1.170(8)

TABLE III
Bond angles (°)

Atoms	Angles	Atoms	Angles
C(2)a-C(1)a-C(4)a	118.7(7)	C(1)a-C(2)a-C(3)a	120.3(7)
C(2)a-C(3)a-C(5)a	121.2(9)	C(3)a-C(5)a-C(6)a	118.3(8)
C(5)a-C(6)a-C(4)a	123.2(6)	C(1)a-C(4)a-C(6)a	118.3(6)
C(1)a-C(4)a-C(7)a	118.1(5)	C(6)a-C(4)a-C(7)a	123.7(5)
C(14)a-C(13)a-C(12)a	106.3(5)	C(13)a-C(14)a-C(15)a	104.8(6)
C(14)a-C(15)a-O(2)a	112.9(6)	C(15)a-O(2)a-C(12)a	105.3(5)
C(13)a-C(12)a-O(2)	110.6(4)	C(13)a-C(12)a-C(11)a	128.5(5)
O(2)a-C(12)a-C(11)a	120.9(5)	C(8)a-N(1)a-C(7)a	121.0(5)
C(9)a-C(10)a-N(2)a	175.6(5)	C(10)a-C(9)aC(8)a	117.7(4)
C(10)a-C(9)a-C(11)a	123.2(4)	C(8)a-C(9)a-C(11)a	119.1(4)
N(1)a-C(8)a-C(9)a	118.4(5)	N(1)a-C(8)a-O(1)a	122.8(4)
C(9)a-C(8)a-O(1)a	118.8(5)	C(12)a-C(11)a-C(9)a	130.5(5)
C(4)a-C(7)a-N(1)a	115.3(4)	C(2)b-C(1)b-C(4)b	121.7(5)
C(1)b-C(2)b-C(3)b	118.7(6)	C(2)b-C(3)b-C(5)b	120.5(7)
C(3)b-C(5)b-C(6)b-	121.2(6)	C(5)b-C(6)b-C(4)b	119.2(5)
C(1)b-C(4)b-C(6)b	118.6(5)	C(1)b-C(4)b-C(7)b	119.5(5)
C(6)b-C(4)b-C(7)b	121.9(5)	C(14)b-C(13)b-C(12)b	107.5(6)
C(13)b-C(14)b-C(15)b	105.5(7)	C(14)b-C(15)b-O(2)b	114.0(6)
C(15)b-O(2)b-C(12)b	104.4(5)	C(13)b-C(12)b-O(2)b	108.6(4)
C(13)b-C(12)b-C(11)b	132.1(5)	O(2)b-C(12)b-C(11)b	119.3(5)
C(8)b-C(9)b-C(11)b	116.7(5)	C(8)b-C(9)b-C(10)b	117.8(4)
C(11)b-C(9)b-C(10)b	125.5(4)	C(8)b-N(1)b-C(7)b	119.7(5)
O(1)b-C(8)b-C(9)b	121.7(5)	O(1)b-C(8)b-N(1)b	122.0(4)
C(9)b-C(8)b-N(1)b	116.3(5)	C(12)b-C(11)b-C(9)b	129.8(5)
C(4)b-C(7)b-N(1)b	115.0(4)	C(9)b-C(10)b-N(2)b	175.1(5)

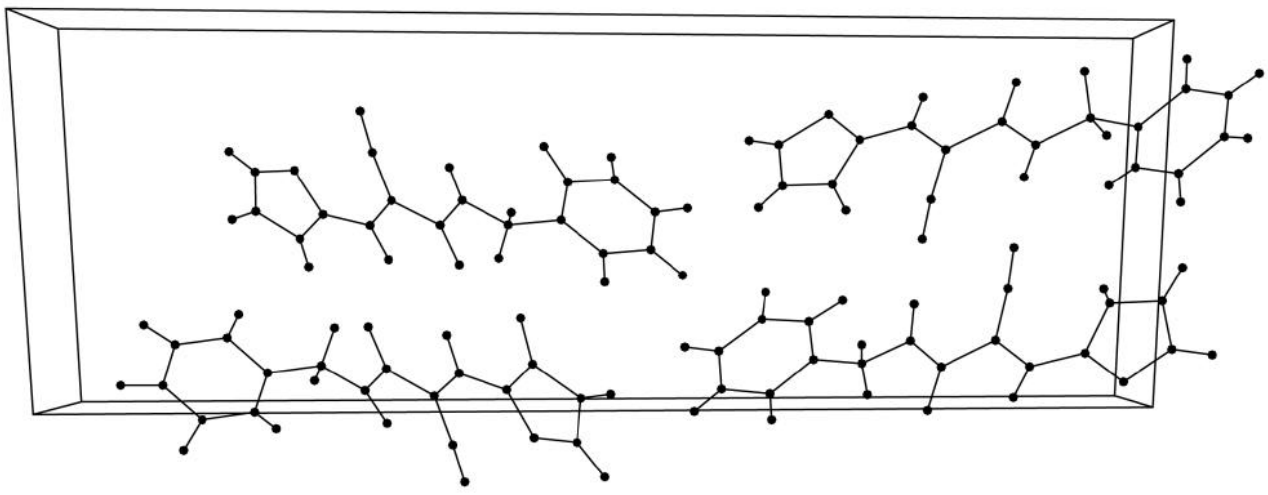

Fig. 2. A projection of the molecules of the title compound in the ab plane.

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