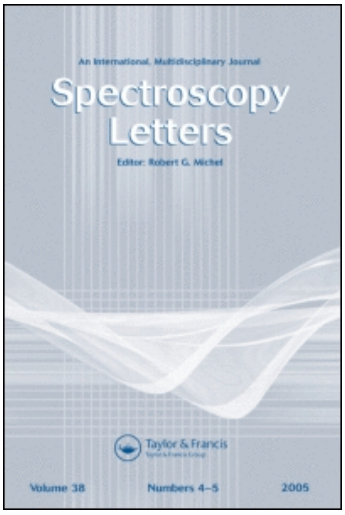

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STRUCTURE OF QUINOLINE-4-CARBOXALDEHYDE-2,4-DINITROPHENYL-N METHYLHYDRAZONE

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Abstract

The crystal structure of the title compound, $C_{17}H_{13}N_5O_4$, has been determined by single crystal x-ray diffraction at room temperature.The molecule is not planar, with dihedral angles of 7.2(1)° between the quinoline ring and N-methylhydrazone group, and 17.45(2)° between the N-methylhydrazone group and the phenyl ring. The crystal parameters of this compound are as follows: monoclinic P 2 1/n, a=9.525(2) Å, b=15.192(2) Å, c= 11.302(2) Å, β = 94.722(3)°, V = 1629.8(6) $Å^3$, Z = 4, Dx = 1.432 g/cm^3, F(000) = 728, λ (MoKα) = 0.71070 Å, μ = 0.106 mm^{-1}, R_{int}= 0.017. The structure was solved by SHELXS-86 and refined by SHELXL-93. R = 0.07 for 2438 observed reflections with $I > 2\sigma(I)$.

Introduction

Recently, scientists have begun to study on the conjugated unsaturated ring systems containing nitrogen as biologically important compounds that may be active against human cell representing major tumor types and the human immunodeficiency virus (HIV).

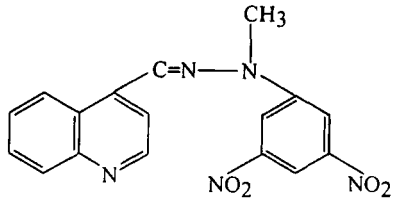
As part of investigations of the synthesis of organic compound having biological activity properties, (I), title compound was obtained and its structure was analyzed by standart analytical techniques (microanalysis, NMR and IR). In order to obtain information about the stereochemistry of the molecule and to confirm the assigned structure, the x-ray analysis of (I) was undertaken.

Experimental

The title compound was synthesized as in the reference [1].Pale yellow and prismatic-shaped crystals were grown from ethanol solution.

Intensities were measured on an Enraf - Nonius CAD - 4 four - circle diffractometer (graphite - monochromated MoKα x-rays). Crystal dimensions are approximately 0.5 x 0.4 x 0.2 mm^3. Accurate unit - cell parameters and the orientation matrix were determined by least squares using 25 reflections in the range $9.97° < \theta < 18.11°$. Data collection was used $w/2\theta$ scan. A total of 3064 reflections ($-11 < h < 0, 0 < k < 18, -14 < l < 14$) was considered non - zero at the $2\sigma(I)$ significance level out of 2438 possible reflections in the θ range 2.53° - 26.37°. Three standart reflections were checked with interval 120 min. for orientation.

Data were corrected for Lorentz, Polarization factors. This structure was solved by direct methods using SHELXS-86[2] and refined by full matrix least squares using SHELXL - 93[3]. H atoms were isotropically, other non-H atoms anisotropically were refined. H atoms attached to the phenyl ring, quinoline ring and hydrazone moiety were fixed geometrically and allowed to ride on their respective parent atoms. The title crystal was of poor quality so the R and wR indexs are not good enough. Final R = 0.07, wR = 0.13



(I)

with weighting scheme $w^{-1} = [\sigma^2 (F_o^2) + (0.151P)^2 + 0.2350P]$, $P = (F_o^2 + 2 F_c^2) / 3$. Max. and min. heights in final difference Fourier map are 0.50 and -0.49 e Å^{-3}.

Result and Discussion

Table-1. lists final atomic coordinates of non-H atoms and selected bond lengths and angles are given in Table-2, Table-3, respectively. An ORTEPII [4] plot of the title molecule is shown in Fig.1. The structure of title compound consists of three almost planar moiety which are N-methylhydrazone group, quinoline and phenyl rings.The dihedral angle between the quinoline ring and N-methylhydrazone group is 7.2(1)°. The maxsimum deviation from the plane defined by ten atoms of the quinoline ring is 0.021(1)Å for C12. The dihedral angle between the two six-membered rings in the quinoline group is 1.45(1)°. In this group bond alteration is observed as short in the C9-C17, C10-C11, C11-C12, C13-C14 bonds; and the others are long.The N-methylhydrazone group is planar and at an angle of 17.45(1)° with respect to the phenyl ring. Single bond distances in the N-methylhydrazone group C1-N3,C7-N3 and N3-N4 (1.375(2) Å, 1.446(3) Å and 1.375(2) Å, respectively) is comparable to values reported in the literature [5, 6]. The C8=N4 bond is typical double-bond length (C8=N4, 1.272(2) Å).The dihedral angles of between the planar phenyl ring and nitro groups in the 2 and 4 positions are 5.83(3)° and 60.16(2)°, respectively. An analysis of the nitro group geometry shows that C-NO_2 bond distances are characteristic single bond distances and similar to the C-NO_2 bond

Table 1. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 x10^3$) for $C_{17}H_{13}N_5O_4$.

$$U_{eq} = \left(\frac{1}{3}\right)\sum_i \sum_j U_{ij} a_i a_j a_i^* a_j^*$$

Atom	x	y	z	U_{eq}
N1	930(2)	954(1)	5674(2)	68(1)
N2	-3089(2)	535(2)	7925(2)	73(1)
N3	1141(2)	2793(1)	6517(2)	49(1)
N4	2347(2)	2381(1)	6219(2)	46(1)
N5	6996(2)	1617(2)	4620(2)	70(1)
O1	1628(2)	321(1)	5970(3)	108(1)
O2	839(2)	1270(2)	4670(2)	89(1)
O3	-3095(2)	-220(2)	7580(2)	106(1)
O4	-3979(2)	827(2)	8540(2)	96(1)
C1	77(2)	2247(1)	6816(2)	44(1)
C2	35(2)	1348(1)	6538(2)	50(1)
C3	-950(2)	783(2)	6929(2)	57(1)
C4	-2000(2)	1127(2)	7570(2)	56(1)
C5	-2055(2)	2011(2)	7820(2)	56(1)
C6	-1027(2)	2563(1)	7460(2)	50(1)
C7	1026(2)	3743(1)	6511(2)	60(1)
C8	3378(2)	2840(1)	5922(2)	48(1)
C9	4641(2)	2407(1)	5530(2)	47(1)
C10	5723(2)	2940(1)	5098(2)	45(1)
C11	5551(2)	3827(1)	5105(2)	48(1)
C12	6544(2)	4309(2)	4676(3)	72(1)
C13	7763(2)	3966(2)	4229(3)	72(1)
C14	7940(2)	3094(2)	4208(2)	57(1)
C15	6908(2)	2533(1)	4642(2)	47(1)
C16	5967(2)	1113(2)	5039(2)	63(1)
C17	4785(2)	1510(2)	5509(2)	57(1)

Table 2. Selected bond lengths [Å] for $C_{17}H_{13}N_5O_4$.

N1 - O1	1.202(3)	N1 - O2	1.229(3)	N1 - C2	1.475(3)
N2 - O3	1.211(3)	N2 - O4	1.223(3)	N2 - C4	1.454(3)
N3 - C1	1.373(2)	N3 - N4	1.375(2)	N3 - C7	1.446(3)
N4 - C8	1.272(2)	N5 - C16	1.359(3)	N5 - C15	1.394(3)
C8 - C9	1.471(2)	C9 - C17	1.370(3)	C9 - C10	1.427(3)
C10 - C11	1.358(3)	C10 - C15	1.420(2)	C11 - C12	1.319(3)
C12 - C13	1.404(3)	C13 - C14	1.337(4)	C14 - C15	1.419(3)
C16 - C17	1.418(3)				

Table 3. Selected bond and torsion angles[°] for $C_{17}H_{13}N_5O_4$.

O1 - N1 - O2	124.4(2)	O1 - N1 - C2	118.5(2)
O2 - N1 - C2	116.9(2)	O3 - N2 - O4	122.8(2)
O3 - N2 - C4	118.8(2)	O4 - N2 - C4	118.4(2)
C1 - N3 - N4	115.7(2)	C1 - N3 - C7	123.2(2)
C8 - N4 - N3	119.6(2)	N4 - C8 - C9	120.2(2)
C1 - N3 - N4 - C8	179.5(2)	N4 - N3 - C1 - C6	161.3(2)
C7 - N3 - C1 - C6	-18.6(3)	O1 - N1 - C2 - C3	-59.4(3)
O4 - N2 - C4 - C5	-6.4(4)	N3 N4 - C8 - C9	-176.2(2)
N4 - C8 - C9 - C10	174.2(2)		

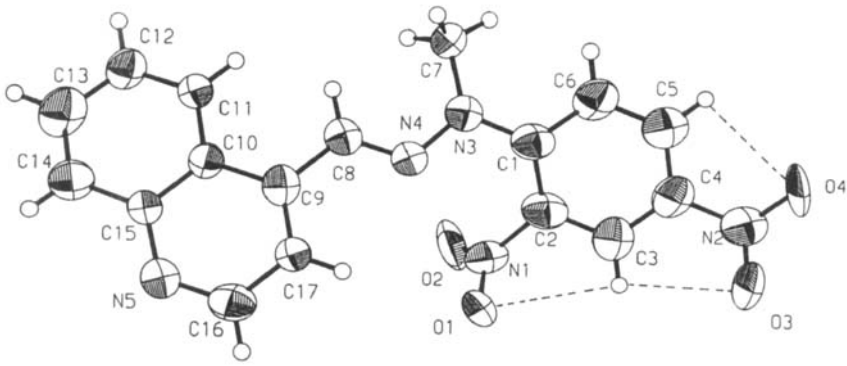


Figure 1. ORTEPII view of the molecule and atomic numbering. The displacement ellipsoids are at the %50 probability level. H atoms are shown as spheres of arbitrary size.

length in nitrobenzene derivatives [7]. The N-O bond distances are in the range of 1.203(3) Å, 1.229(3) Å. The O1 and O3 atoms have high atomic displacement parameters. Therefore N1-O1 and N2-O3 bond distances are slightly shorter than other N-O bond distances in the molecule. In addition, as shown in Fig.1 that the following possible C-H...O intramolecular hydrogen bonds (<3.5 Å) are observed: C3...O1 2.851(3) Å, C3-H3...O1 92.3(2)°; C3...O3 2.699(3) Å, C3-H3...O3 97.3(2)°; C5...O4 2.738(3) Å, C5-H5...O4 97.3(2)°.

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