

Postdoctoral Research Fellowship and HKF would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R & D No. 190-9609-2801.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1423). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 1672–1673

N-(4-Chloro-2-iodophenyl)-*N*-methylcinnamamide

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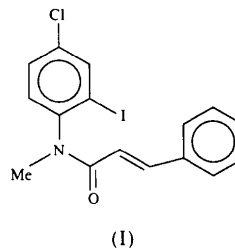
(Received 13 May 1999; accepted 8 June 1999)

Abstract

In the title compound, C₁₆H₁₃ClINO, the cinnamamide group is almost planar. The phenyl rings are almost perpendicular, making an interplanar angle of 83.1 (1)° with one another. The molecules are packed as dimers through C—H···O hydrogen bonds.

Comment

We are interested in the synthetic potential of the title compound, (I), and of other acyclamides, which can react with a wide range of nucleophilic compounds and therefore present new possibilities in the synthesis of heterocyclic compounds (Augustin *et al.*, 1980). We have undertaken the X-ray structure analysis of (I) as an extension of studies on the molecular packing of cinnamamide derivatives.



The bond lengths and angles of the cinnamamide group in (I) are comparable with reported values (Schmidt, 1964; Iwamoto & Kashino, 1990; Iwamoto *et al.*, 1989). In the cinnamoyl part of the molecule, the C1—C7, C7—C8 and C8—C9 bond distances of 1.461 (5), 1.326 (4) and 1.493 (4) Å, respectively, are comparable with reported values of 1.467 (2), 1.329 (2) and 1.485 (2) Å, respectively, for the analog *N*-methyl-*N*-(2-nitrophenyl)cinnamamide (Subramanian *et al.*, 1999). The widening of the C1—C7—C8 angle [126.8 (3)°] is due to intramolecular repulsion between C2 and C8 [H2A···H8A = 2.29 and C2···C8 = 3.025 (6) Å]. Structural studies reveal that the N11 atom is *sp*² hybridized, in spite of the lack of coplanarity of ring *B* and the cinnamoyl moiety.

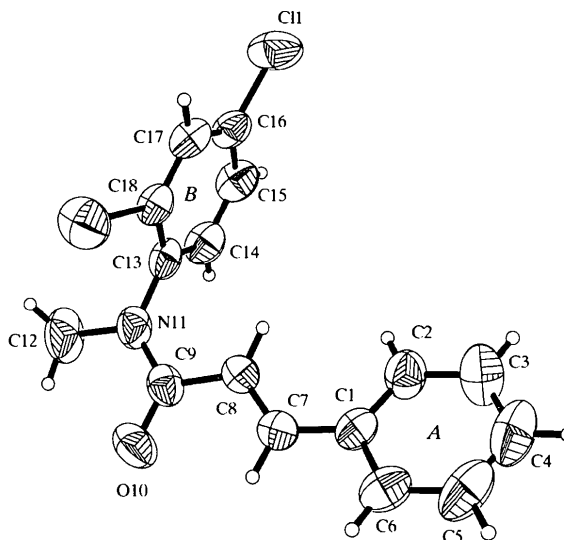


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Phenyl ring *B* and the carbonyl group are *trans* to one another with respect to the C9—N11 bond [C13—N11—C9—O10 = 177.2 (3)°]. Phenyl rings *A* and *B* (Fig. 1) are almost perpendicular, making an interplanar angle of 83.1 (1)° with one another. The molecules exist as dimers through C—H...O hydrogen bonding [C14...O10(2 - *x*, -*y*, 1 - *z*) = 3.309 (6) Å] in the lattice.

Experimental

Cinnamoyl chloride (0.2 mol in hexane) was added dropwise to a solution of *p*-chloro-*o*-iodoaniline (0.2 mol) in dry benzene (50 ml) and pyridine (0.2 mol). The mixture was heated on a water bath at 333–343 K for 5 h and then poured into water (80–100 ml). The benzene layer was separated and washed repeatedly with water (3–4 times), dried with magnesium sulfate and the benzene was then evaporated off to obtain the anilide. For methylation of the anilide, NaH (0.2 mol) was placed in a round-bottomed flask. A solution of the anilide (0.2 mol) in dry dimethylformamide (DMF, 50 ml) was added dropwise. The reaction mixture was stirred for 15 min and then methyl iodide was added and stirring continued for 3 h. The reaction mixture was poured into water and extracted with ethyl acetate and then washed with water repeatedly to remove the DMF. The compound was recrystallized from dichloromethane.

Crystal data

C₁₆H₁₃ClINO

M_r = 397.62

Triclinic

*P*1

a = 9.0587 (2) Å

b = 10.2616 (2) Å

c = 10.3295 (2) Å

α = 79.104 (1)°

β = 64.898 (1)°

γ = 65.456 (1)°

V = 790.85 (3) Å³

Z = 2

D_x = 1.670 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 6563 reflections

θ = 2.18–28.37°

μ = 2.188 mm⁻¹

T = 293 (2) K

Block

0.48 × 0.40 × 0.28 mm

Colorless

Data collection

Siemens SMART CCD area-detector diffractometer

ω scans

Absorption correction:

empirical (*SADABS*;

Sheldrick, 1996)

T_{min} = 0.393, *T_{max}* = 0.542

5477 measured reflections

3771 independent reflections

3193 reflections with

I > 2σ(*I*)

R_{int} = 0.019

θ_{max} = 28.28°

h = -12 → 12

k = -13 → 8

l = -13 → 13

Refinement

Refinement on *F*²

R [*F*² > 2σ(*F*²)] = 0.041

wR (*F*²) = 0.114

S = 1.042

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.699 e Å⁻³

Δρ_{min} = -0.705 e Å⁻³

Extinction correction: none

3771 reflections

181 parameters

H atoms constrained

w = 1/[σ²(*F_o*²) + (0.0507*P*)² + 0.5358*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

Scattering factors from
*International Tables for
Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

N1—C18	2.086 (3)	C7—C8	1.326 (4)
C1—C16	1.736 (4)	C8—C9	1.493 (4)
C1—C7	1.461 (5)	C9—O10	1.220 (4)
C5—C6—C1	119.8 (4)	N11—C9—C8	116.7 (3)
C8—C7—C1	126.8 (3)	C9—N11—C13	123.9 (2)
C7—C8—C9	120.8 (3)	C9—N11—C12	119.4 (3)
O10—C9—N11	122.1 (3)	C13—N11—C12	116.7 (3)
O10—C9—C8	121.2 (3)		

Table 2. Hydrogen-bonding geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C14—H14A...O10 ⁱ	0.93	2.40	3.309 (6)	166

Symmetry code: (i) 2 - *x*, -*y*, 1 - *z*.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

SR and ES thank M. Raja, Scientist, SPIC Science Foundation, Chennai, and Professor K. K. Balasubramanian, Indian Institute of Technology, Chennai, for the synthesis and provision of the title compound. HKF thanks the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. SSSR thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1426). Services for accessing these data are described at the back of the journal.

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