

Journal of Molecular Structure 641 (2002) 101-107



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# Influence of quadrupolar interactions on the crystal packing of organic compounds: the pentafluorophenyl ester of a tricyclic $\alpha$ , $\beta$ -unsaturated- $\gamma$ -amino acid

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Received 25 April 2002; revised 1 July 2002; accepted 1 July 2002

# Abstract

The single crystal X-ray diffraction structure of (-)-pentafluorophenyl(*Z*,*S*,*S*)-2-(6-oxo-1,3,4,6,11,11a-hexahydro-2*H*-pyrido[1,2-*b*]isoquinolin-11-yliden)acetate (**1**) is reported. We have found that the crystal packing of the  $\alpha$ , $\beta$ -unsaturated- $\gamma$ -amino acid derivative **1** is mainly determined by the interaction between the two aromatic rings, namely the benzenic type and the pentafluorinated ring. The crystal packing of **1** shows a herringbone (zig-zag) structure, that is quite typical of aromatic compound with intermolecular arene–arene interactions. The benzenic and the fluorinated phenyl rings in **1** are in a matched face-to-face arrangement. The origin of this interaction can be ascribed to a favourably quadrupolar interaction between both types of aromatic ring. The results reported in the present paper indicate that the pentafluorophenyl ester functionality can be a useful building block in crystal engineering. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Arene-arene interaction; Quadrupolar interaction; Crystal structure; Polyannular heterocycle; α,β-Unsaturated-γ-amino acid

# 1. Introduction

Intermolecular interactions play key roles in the structure, reactivity, and properties of organic compounds [1,2]. The strength of the interaction depends on the type (electrostatic, dispersion, charge-transfer, etc) as well as on the relative orientation of the interacting species [3,4]. Aromatic compounds possess several properties (electron charge distribution, non-vanishing

electrostatic multipole moment, magnetic ring current, etc) [5-7], that render them very active to participate in numerous interactions. Aromatic moieties have been reported to interact with other chemical entities, including other aromatics (arene-arene interactions) [8-12], electron-donating groups [13], hydrogen-bond donors [14,15], hydrocarbonated fragments [16,17], amides [18, 19], halogens [20,21], and cations [22-24].

The structure (both molecular and supramolecular), the reactivity, and the properties (physical, physico-chemical, technological, and biological) of the aromatic compounds are modulated by the nature of the substituents [25-27]. From an electronic point

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of view, they can be either electron-donor or electronacceptor through resonance or inductive effect; furthermore, a sterically demanding substituent can distort the planarity of the aromatic systems.

Another important characteristic of arenes is that most of them, besides having a permanent dipole moments, possess non-vanishing higher-order electrostatic moments [28], that allows them to participate in a variety of electrostatic interactions. The potential (sign and value) of an electrostatic interaction depends on the distance and the relative orientation of the dipole/multipole(s).

In this paper, we report the crystal structure of (-) pentafluorophenyl(*Z*,*S*,*S*)-2-(6-oxo-1,3,4,6,11,11a-hexahydro-2*H*-pyrido[1,2-*b*] isoquinolin-11-yliden) acetate (1); this molecule possesses both a benzenic type aromatic ring and a pentafluorinated phenyl ring. The molecular structure of this molecule is quite suitable to assess the relative importance of diverse interactions on the crystal packing. Due to the relevance of aromatic building blocks in the field of crystal engineering [29], the analysis of the crystal structure of compound 1 is useful to understand the different factors influencing the crystal packing of aromatic compounds [30,31].

# 2. Experimental

2.1. Synthesis of (-)-pentafluorophenyl(Z,S,S)-2-(6-oxo-1,3,4,6,11,11a-hexahydro-2H-pyrido [1,2-b]isoquinolin-11-yliden)acetate (1)

C<sub>6</sub>F<sub>5</sub>OH (1.04 g, 5.7 mmol), dicyclohexylcarbodiimide (DCC, 625 mg, 3 mmol), and 4-dimethylaminopyridine (DMAP, 50 mg, 0.40 mmol) were sequentially added to a solution of acid **2** [32] (500 mg, 1.9 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (60 ml). The mixture was stirred at room temperature for 7 h. Then, the solution was washed with 5% aqueous HCl, water, saturated aqueous NaHCO<sub>3</sub>, brine and dried (MgSO<sub>4</sub>). The solvent was removed under vacuum and the crude residue was chromatographed (hexane–EtOAc, 4:1) to give pure pentafluorophenyl ester **1** (730 mg, 91%). White solid. Mp 147–149 °C. [ $\alpha$ ]<sub>D</sub> = -324 (CHCl<sub>3</sub>, c = 0.6). <sup>1</sup>H NMR (300 MHz, 303 K, CDCl<sub>3</sub>)  $\delta$  8.33 (m, 1H), 7.82 (m, 1H), 7.63 (m, 2H), 6.66 (s, 1H), 5.43 (broad d, J = 11.0, 1H), 4.89 (broad d, J = 13.1, 1H), 2.79 (distorted dt, J = 12.8, 2.9, 1H), 2.00–1.40 (m, 6H). IR (KBr)  $\nu$  3380, 2900, 1720, 1620, 1595, 1570, 1490, 1350, 1205, 1085, 970. MS (ES, positive ionization mode) m/z 446 (M<sup>+</sup> + Na). Anal. calcd. for C<sub>21</sub>H<sub>14</sub>F<sub>5</sub>NO<sub>3</sub>: C, 59.57; H, 3.33; N, 3.31. Found: C, 59.38; H, 3.12; N, 3.31.

2.2. Crystal structure of (-) pentafluorophenyl(Z,S,S)-2-(6-oxo-1,3,4,6,11,11ahexahydro-2H-pyrido[1,2-b]isoquinolin-11yliden)acetate (1)

Single crystals of 1 were obtained by crystallization from hexane-ethyl acetate. A suitable crystal (block, white, dimensions  $0.50 \times 0.45 \times 0.40 \text{ mm}^3$ ) was used for the structure determination. X-ray data were collected using a Bruker SMART CCD area detector single-crystal diffractometer with graphite monochromated Mo K $\alpha$  radiation ( $\lambda = 0.71073$  Å) by the phi-omega scan method. A total of 1271 frames of intensity data were collected. The integration process yields a total of 10,382 reflections of which 4576 were independent [R(int) = 0.0402]. Absortion corrections were applied using the SADABS program [33] (maximum and minimum transmission coefficients, 1.000 and 0.456). The structure was solved using the Bruker SHELXTL-PC software [34] by direct methods on  $F^2$ . Hydrogen atoms were included in calculated positions and refined in the riding mode. The convergence was reached at a final R1 = 0.0441(for  $I > 2\sigma(I)$ ), wR2 = 0.1304 (for all data), 272 parameters, with allowance for the thermal anisotropy for all non-hydrogen atoms. The weighting scheme employed was  $w = [\sigma^2 (F_0^2 + 0.0712P)^2]$  and P = $(|F_0|^2 + 2|F_c|^2)/3$  and the goodness of fit on  $F^2$  was 1.039 for all the observed reflections. The details of the data collection and refinement are summarized in Table 1. Table 2 shows the atomic coordinates and isotropic displacement parameters for non-hydrogen atoms. The structure drawings were prepared using PLATON<sup>1</sup> and ORTEP-3<sup>2</sup> programs.

<sup>&</sup>lt;sup>1</sup> Web address: http://www.cryst.chem.uu.nl/platon/

<sup>&</sup>lt;sup>2</sup> Web address: http://www.chem.gla.ac.uk/~louis/software/ ortep3/

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 Table 1

 Crystal data and structure refinement for 1

Fw Space group Crystal system	423.33 <i>P</i> 2(1)2(1)2(1)
Space group Crystal system	P2(1)2(1)2(1)
Crystal system	
	Orthorhombic
Z	4
a (Å)	9.8700(2)
b (Å)	10.9262(3)
<i>c</i> (Å)	17.1709(4)
$\alpha = \beta = \gamma (\text{deg})$	90
$V(Å^3)$	1851.74(8)
$\rho$ (calc) (g/cm <sup>3</sup> )	1.518
$\mu_{\text{calc}} (\text{mm}^{-1})$	0.135
Radiation (Mo Ka) (Å)	0.70173
<i>T</i> (K)	298(2)
$\Theta$ range for data collection (deg)	2.21-28.27
Final R1 indices $[I > 2\sigma(I)]^{a}$	R1 = 0.0441
Final $wR2$ indices (all data) <sup>a</sup>	wR2 = 0.1304
Largest diff. peak and hole $(e \text{\AA}^{-3})$	0.216 and -0.239

### 3. Results and discussion

In connection with our continuous interest on aromatic compounds [35–38] and on peptide–arene hybrids<sup>3</sup> [39], we have prepared the tricyclic  $\alpha,\beta$ -unsaturated- $\gamma$ -amino acid **2** (Scheme 1) [32,40], that has been used as precursor for the synthesis of the peptide-heterocyclic hybrids of type **3**. An intermediate for the synthesis of **3** is the pentafluorophenyl ester **1** (Scheme 1), that bears a benzenic type ring and a pentafluorophenyl ring in its structure.

It is known that benzene and hexafluorobenzene form a 1:1 complex that have a higher melting point that either of their components [41]. Although previous work rationalized this behaviour to different grounds, it is currently accepted that the origin of this interaction is through a quadrupole–quadrupole interaction between the two aromatic compounds [42].

The *zz* components (perpendicular to the ring plane) of the quadrupole moment tensors of the pentafluorinated phenyl ring and of the benzenic-type ring of 1 are large in magnitude and with opposite signs [43–45]; this fact makes a face-to-face

Table 2 Atomic coordinates (× 10<sup>4</sup>) and equivalent isotropic displacement parameters ( $\mathring{A}^2 \times 10^3$ ) for **1**. *U*(eq) is defined as one third of the trace of the orthogonalized *U*<sub>ij</sub> tensor

		5		
	x	у	Z	U(eq)
F(1)	7780(2)	- 5366(2)	10.837(1)	128(1)
F(2)	8089(3)	-6245(2)	12.311(2)	179(1)
F(3)	6583(4)	-5329(2)	13.476(1)	176(1)
F(4)	4726(3)	3607(2)	13,187(1)	150(1)
F(5)	4399(2)	-2749(2)	11,740(1)	115(1)
O(1)	8273(2)	1230(2)	7669(1)	72(1)
O(2)	7218(2)	-2058(2)	10,689(1)	89(1)
O(3)	5832(2)	-3656(2)	10,510(1)	82(1)
N(1)	7968(2)	397(1)	8862(1)	49(1)
C(1)	8854(2)	1304(2)	9232(1)	56(1)
C(2)	8012(2)	2266(2)	9643(1)	60(1)
C(3)	7022(2)	1674(2)	10,214(1)	63(1)
C(4)	6191(2)	661(2)	9838(1)	56(1)
C(5)	7132(2)	-263(2)	9433(1)	48(1)
C(6)	6336(2)	-1301(2)	9071(1)	51(1)
C(7)	5879(2)	-1124(2)	8262(1)	51(1)
C(8)	6551(2)	-278(2)	7793(1)	50(1)
C(9)	7668(2)	513(2)	8099(1)	52(1)
C(10)	6189(2)	-134(2)	7013(1)	61(1)
C(11)	5133(3)	-815(2)	6707(1)	69(1)
C(12)	4441(2)	-1636(2)	7177(1)	67(1)
C(13)	4798(2)	-1796(2)	7942(1)	59(1)
C(14)	6034(3)	-2314(2)	9472(1)	66(1)
C(15)	6465(3)	-2586(2)	10,270(1)	66(1)
C(16)	6086(2)	-4030(2)	11,261(1)	56(1)
C(17)	7027(2)	-4936(2)	11,419(2)	72(1)
C(18)	7168(3)	-5373(2)	12,177(2)	93(1)
C(19)	6392(4)	-4905(3)	12,755(2)	97(1)
C(20)	5490(3)	-4033(3)	12,604(2)	83(1)
C(21)	5335(2)	-3594(2)	11,871(2)	68(1)

arrangement of both rings very favourable by quadrupolar interactions. Two face-to-face aromatic rings can be totally aligned or they can have their mass centres displaced each other (offset). The magnitude of the offset depends on some steric and electrostatic factors. It has been shown, from both theoretical [46] and experimental [47] points of view, that the higher the quadrupolar interaction is, lower is the offset.

On the basis of these premises, we reasoned that the crystal structure of compound 1 can give information on the relative importance of different intermolecular interactions in the crystal packing of organic compounds. Some putative interactions in the crystal packing of 1 include arene-arene interactions

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<sup>&</sup>lt;sup>3</sup> The hybrids peptide–arene are compounds having one or several peptidic chain(s) linked to a conformationally rigid scaffold (either aromatic carbocyclic or heterocyclic).

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(and the relative orientation of the aromatic rings), and fluorine-mediated interactions (fluorine-hydrogen, fluorine-arene, and so on) [48,49].

The molecular solid state structure of **1** is shown in Fig. 1. A selection of geometrical parameters is indicated in Table 3.

The solid state conformation of the fused tricyclic system of **1** is quite similar to the one found in solution (<sup>1</sup>H NMR evidence in CDCl<sub>3</sub>) and analogous to other derivatives of pyrido[1,2-*b*]isoquinoline [32, 50]. The conformation of the saturated ring of **1** is a nearly perfect chair with a slight pyramidalization at the nitrogen atom. An interesting structural feature of the conformation of the molecule **1** is that the pentafluorophenyl ring is nearly perpendicular to the mean plane of the pyrido[1,2-*b*]isoquinoline ring system (dihedral angle between the two rings is 84°); this relative orientation is suitable to maximize the number of intermolecular interactions in the crystal

packing. It is worth mentioning that an alternative arrangement (with the pentafluorophenyl and the tricyclic system in a coplanar conformation) would place a fluorine atom and the oxygen carbonyl in the same plane (and quite close), probably resulting in a destabilizing interaction from an electrostatic point of view.

The most interesting structural feature of the pentafluorophenyl ester **1** is its crystal packing (Fig. 2). The molecules of **1** are arranged in a herringbone (zig-zag) supramolecular structure, with an antiparallel array of two contiguous molecules (Fig. 3). The main intermolecular interaction is arene–arene between the pentafluorophenyl ring and the benzenic type ring of the two adjacent molecules. The distance between the geometrical centres of both rings is 4.3 Å, and they are almost perfectly aligned: the offset between rings is less than 0.2 Å, and the angle between both rings is ca. 6°.



Fig. 1. Molecular structure of (-)-pentafluorophenyl(Z,S,S)-2-(6-oxo-1,3,4,6,11,11a-hexahydro-2H-pyrido[1,2-b]isoquinolin-11-yliden)ace-tate (1). Thermal ellipsoids are represented at 50% probability.

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Table 3
Selected torsion angles (deg) for compound 1

Torsion		Torsion			
C(9)-N(1)-C(1)-C(2)	96.9(2)	C(1)-N(1)-C(9)-O(1)	12.0(3)		
C(5)-N(1)-C(1)-C(2)	-62.0(2)	C(5)-N(1)-C(9)-O(1)	168.06(18)		
N(1)-C(1)-C(2)-C(3)	54.3(2)	C(1)-N(1)-C(9)-C(8)	- 168.01(16)		
C(1)-C(2)-C(3)-C(4)	-51.1(3)	C(5)-N(1)-C(9)-C(8)	-11.9(3)		
C(2)-C(3)-C(4)-C(5)	52.4(2)	C(10)-C(8)-C(9)-O(1)	-2.6(3)		
C(9)-N(1)-C(5)-C(6)	29.2(3)	C(7)-C(8)-C(9)-O(1)	177.98(19)		
C(1)-N(1)-C(5)-C(6)	-173.24(17)	C(10)-C(8)-C(9)-N(1)	177.42(18)		
C(9)-N(1)-C(5)-C(4)	-94.7(2)	C(7)-C(8)-C(9)-N(1)	-2.0(3)		
C(1)-N(1)-C(5)-C(4)	62.87(19)	C(9)-C(8)-C(10)-C(11)	- 177.93(19)		
C(3)-C(4)-C(5)-N(1)	-56.8(2)	C(6)-C(7)-C(13)-C(12)	-177.4(2)		
C(3)-C(4)-C(5)-C(6)	178.49(17)	C(7)-C(6)-C(14)-C(15)	178.6(2)		
N(1)-C(5)-C(6)-C(14)	148.8(2)	C(5)-C(6)-C(14)-C(15)	-3.1(4)		
C(4)-C(5)-C(6)-C(14)	-88.9(2)	C(16)-O(3)-C(15)-O(2)	2.6(4)		
N(1)-C(5)-C(6)-C(7)	-32.8(2)	C(16)-O(3)-C(15)-C(14)	-177.9(2)		
C(4)-C(5)-C(6)-C(7)	89.5(2)	C(6)-C(14)-C(15)-O(2)	-6.9(5)		
C(14)-C(6)-C(7)-C(8)	- 159.6(2)	C(6)-C(14)-C(15)-O(3)	173.7(2)		
C(5)-C(6)-C(7)-C(8)	22.0(3)	C(15)-O(3)-C(16)-C(21)	86.1(3)		
C(14)-C(6)-C(7)-C(13)	19.4(3)	C(15)-O(3)-C(16)-C(17)	-99.6(3)		
C(5)-C(6)-C(7)-C(13)	-159.06(18)	O(3)-C(16)-C(17)-F(1)	6.2(3)		
C(6)-C(7)-C(8)-C(10)	176.69(18)	O(3)-C(16)-C(17)-C(18)	-174.0(2)		
C(13)-C(7)-C(8)-C(9)	177.15(17)	O(3)-C(16)-C(21)-F(5)	-3.7(3)		
C(6)-C(7)-C(8)-C(9)	- 3.9(3)	O(3)-C(16)-C(21)-C(20)	174.0(2)		

All the bond lengths and angles have standard values. The two aromatic rings are planar, having dihedral angles of either 0 or  $\pm 180^{\circ}$  being the deviation from planarity lower than  $2^{\circ}$ .

It is worth mentioning that the arene-arene intermolecular interaction is the only one detected in the crystal packing of **1**. Based on the facts commented previously, the origin of this phenomenon must be attributed to a favourable quadrupolar interaction between both rings.

The quadrupole moment is a tensorial magnitude that, at a molecular level, can be used as a measurement of the distribution of charge relative to a particular axis [51]. We [38,52] and others [10, 11,47,53–55] have shown that this property can be used to rationalize the molecular and supramolecular structure of organic compounds both in solution and in the solid state.

On the other hand, a current, highly appealing, topic of research is crystal engineering: the design, preparation, and structural characterization of molecules having defined crystal packing [29,56,57]. The interest on this topic is twofold: to deep in the knowledge of the factors that influence crystal packing, and to synthesize materials with useful technological [58] and pharmacological [59] proper-

ties. On the basis of the known characteristics of arenes, the molecules containing aromatic fragments have been frequently used as building blocks to design organic crystals. Although, the crystal structure of some fluorinated arenes have been described [60-63], this is the first report on the crystal structure of the pentafluorophenyl esters of an amino acid derivative (a  $\gamma$ -amino acid).<sup>4</sup>

Since pentafluorophenyl esters are readily available synthetic intermediates, they can be useful building blocks for the generation of defined crystal structures, as well as a tool to document the relative importance of the different intermolecular forces in the crystal.

Incidentally, we have prepared compound 1 as an intermediate for the synthesis of peptide-heterocyclic hybrids of type 3 [39]; that, in turn, were designed with the goal in mind to interact with biological targets through the aromatic ring. The results reported

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<sup>&</sup>lt;sup>4</sup> The first report on the crystal structure of a pentafluorophenyl ester (from 3-chloro-3-phenylpropanoate) has been recently disclosed on electronic format [63].



Fig. 2. View along the *c*-axis of the crystal packing of (-)-pentafluorophenyl(*Z*,*S*,*S*)-2-(6-oxo-1,3,4,6,11,11a-hexahydro-2*H*-pyrido[1,2-*b*]isoquinolin-11-yliden)acetate (1).



Fig. 3. Antiparallel dimer formed by stacking of two molecules of 1.

in this paper reinforces the capacity of the aromatic ring of the tricyclic scaffold to participate in intermolecular interactions.

# 4. Conclusions

Summarizing, we have reported the crystal structure of compound **1**, having an aromatic tricyclic  $\alpha$ , $\beta$ unsaturated- $\gamma$ -amino acid and a pentafluorophenyl fragment. The crystal packing of **1** has a face-to-face arrangement of both types of aromatic rings, that it is stabilized by a favourably quadrupolar interaction between the aromatic rings. Although other intermolecular interactions are possible in this compound (i.e. fluorine atom with other fragment), they have not been observed; what indicates the relatively high importance of quadrupolar interactions in the crystal packing of organic molecules.

### Acknowledgments

Financial support from the Spanish Ministry of Science and Technology (Project BQU2001-2270) and Madrid Autonomous Community (Project 08.5/0056/2000-2) is gratefully acknowledged.

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