

The work was supported in part by a grant from the Israeli Ministry of Science and Technology.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1132). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). **C54**, 1367–1369

## 1:1 Complex of Octadecanoic Acid and 3-Pyridinecarboxamide

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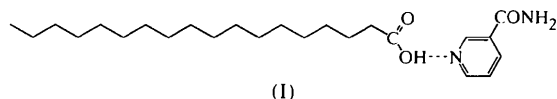
(Received 9 June 1997; accepted 11 December 1997)

### Abstract

An equimolar mixture of stearic acid (octadecanoic acid) and nicotinamide (3-pyridinecarboxamide) in acetone yields crystals of a 1:1 complex, C<sub>18</sub>H<sub>36</sub>O<sub>2</sub>·C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O. In this structure, the 1:1 complexes form dimers through hydrogen bonds between the amide groups, thus forming a lamellar structure. The alkyl chain of stearic acid has an all-*trans* conformation.

### Comment

The title compound, (I), has the bitter taste of nicotinamide and the pharmaceutical advantage of improved hygroscopicity (Ayukawa *et al.*, 1986). Nicotinamide is released from (I) at a certain temperature and pH level depending on the number of C atoms contained in the fatty acid. The applicability of a pH- and thermo-responsive drug delivery system has been reported (Yokoyama *et al.*, 1993; Yokoyama, Sunohara *et al.*, 1994; Yokoyama, Miyamura & Fujie, 1994). However, the crystal structures of these complexes have not been determined. Therefore, we report here the structure of the title compound, (I).



The numbering scheme for the equimolar complex of stearic acid and nicotinamide is given in Fig. 1. The equimolar complex forms a hydrogen-bonded dimer, thus forming a lamellar structure. As is apparent from Table 2, which gives geometric details of the hydrogen bonds, and also from Fig. 2, which shows a projection of the title compound along the *a* axis, three types of hydrogen bonding occur in this structure. One hydrogen bond forms between the N1 atom of the pyridine ring of the nicotinamide (acceptor) and the O2 atom of the stearic acid (donor), with an N1···O2 distance of 2.693 (1) Å. Another hydrogen bond forms between the O3 atom of the amide group of the nicotinamide (acceptor) and the N2 atom of the amide group of the other nicotinamide (donor), with an O3···N2<sup>i</sup> distance of 2.939 (1) Å, thus forming the dimer. The third hydrogen bond is formed between two amide groups, between atoms O3 (acceptor) and N2 (donor), with an O3···N2<sup>ii</sup> distance of 2.904 (1) Å, being parallel to the lamellar structures (the symmetry codes are as in Table 2).

As is apparent from Table 1, which gives bond lengths and torsion angles, the C1—O1 bond length of 1.199 (2) Å and the C1—O2 length of 1.321 (2) Å are characteristic of C=O and C—O bonds.

The amide group in the title compound is twisted out of the plane of the pyridine ring. The torsion angles N2—C24—C20—C21 and O3—C24—C20—C19 are 26.0 (2) and 25.3 (2)°, respectively; these angles are considerably different from the torsion angles of 179 and –168° calculated by Wright & King (1954). Compared with the crystal structure of nicotinamide alone, the amide group of the title compound is rotated in a counterclockwise direction by about 150° around C20—C24. That is to say, in the case of nicotinamide alone, two N atoms of the amide group and the pyridine ring are present on the same side, but in the title compound, the two N atoms are present on opposite sides.

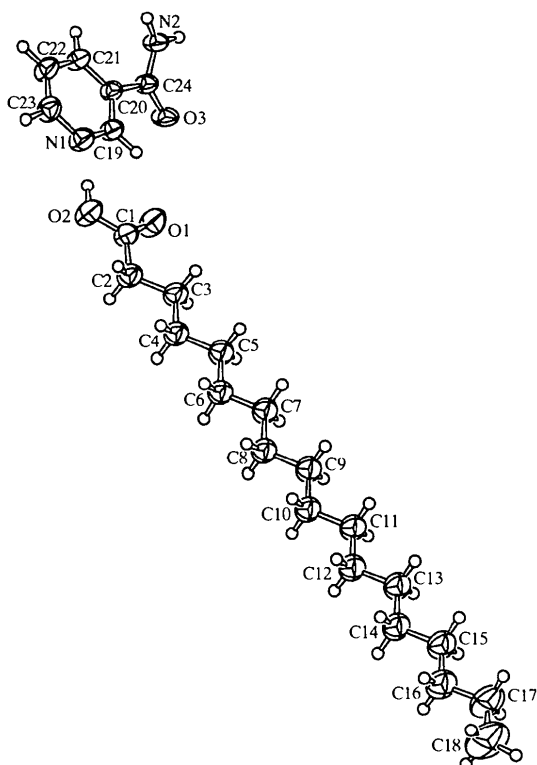


Fig. 1. Drawing of title compound showing the numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

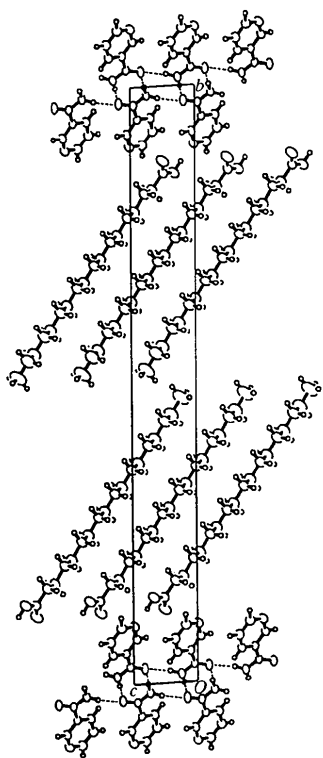


Fig. 2. A view of the title compound projected along the *a* axis. Intermolecular hydrogen bonds are represented by dashed lines.

The crystal structure of stearic acid has polymorphs; the *B*, *C* and *E* forms have been described in detail (Goto & Asada, 1978; Kaneko *et al.*, 1994*a,b*; Malta *et al.*, 1971; Kaneko & Kobayashi, 1990). The *E* form is metastable and is transformed into the *B* form, which is stable at room temperature. The *E* and *B* forms of stearic acid are transformed into the *C* form at about 315 K. The alkyl chains of the *E* and *C* forms have all-*trans* conformations, while the C2—C3 bond in the *B* form of stearic acid takes a *gauche* conformation. In the title complex, the alkyl chain of the stearic acid has an all-*trans* conformation, as in the *C* and *E* forms of stearic acid.

## Experimental

Crystals of the title compound were grown from an equimolar mixture of stearic acid and nicotinamide in acetone at 308 K.

### Crystal data

C<sub>18</sub>H<sub>36</sub>O<sub>2</sub>·C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O

*M<sub>r</sub>* = 406.607

Triclinic

*P* $\bar{1}$

*a* = 5.4313 (5) Å

*b* = 45.180 (3) Å

*c* = 5.0355 (5) Å

$\alpha$  = 92.924 (6)°

$\beta$  = 92.317 (8)°

$\gamma$  = 92.088 (6)°

*V* = 1232.1 (2) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.096 Mg m<sup>-3</sup>

*D<sub>m</sub>* not measured

Cu K $\alpha$  radiation

$\lambda$  = 1.5418 Å

Cell parameters from 25 reflections

$\theta$  = 27.8–28.5°

$\mu$  = 0.559 mm<sup>-1</sup>

*T* = 293.2 K

Needle

0.50 × 0.04 × 0.02 mm

Colorless

### Data collection

Rigaku AFC-7R diffractometer

$\omega$ -2 $\theta$  scans

Absorption correction: none

5966 measured reflections

4193 independent reflections

3231 reflections with

*I* > 1.5 $\sigma$ (*I*)

*R*<sub>int</sub> = 0.018

$\theta$ <sub>max</sub> = 65°

*h* = -6 → 6

*k* = -51 → 51

*l* = 0 → 5

3 standard reflections

every 150 reflections

intensity decay: none

### Refinement

Refinement on *F*<sup>2</sup>

*R* = 0.042

*wR* = 0.053

*S* = 2.428

4193 reflections

431 parameters

All H atoms refined

*w* = 1/[ $\sigma^2(F_o)$

+ 0.00002|*F<sub>o</sub>*|<sup>2</sup>]

( $\Delta/\sigma$ )<sub>max</sub> = 0.004

$\Delta\rho$ <sub>max</sub> = 0.24 e Å<sup>-3</sup>

$\Delta\rho$ <sub>min</sub> = -0.24 e Å<sup>-3</sup>

Extinction correction:

Zachariasen (1967) type

2, Gaussian isotropic

Extinction coefficient:

0.179 (5)

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C1	1.199 (2)	C9—C10	1.513 (2)
O2—C1	1.321 (2)	C10—C11	1.518 (2)
O3—C24	1.237 (1)	C11—C12	1.513 (2)
N1—C19	1.333 (2)	C12—C13	1.513 (2)
N1—C23	1.336 (2)	C13—C14	1.515 (2)
N2—C24	1.331 (2)	C14—C15	1.511 (2)
C1—C2	1.498 (2)	C15—C16	1.510 (2)
C2—C3	1.508 (2)	C16—C17	1.511 (3)
C3—C4	1.519 (2)	C17—C18	1.494 (3)
C4—C5	1.512 (2)	C19—C20	1.384 (2)
C5—C6	1.515 (2)	C20—C21	1.388 (2)
C6—C7	1.513 (2)	C20—C24	1.489 (2)
C7—C8	1.514 (2)	C21—C22	1.377 (2)
C8—C9	1.506 (2)	C22—C23	1.376 (2)
C19—N1—C23	118.0 (1)	C12—C13—C14	114.1 (1)
O1—C1—O2	122.4 (1)	C13—C14—C15	114.0 (2)
O1—C1—C2	125.7 (1)	C14—C15—C16	114.7 (2)
O2—C1—C2	111.8 (1)	C15—C16—C17	114.1 (2)
C1—C2—C3	115.7 (1)	C16—C17—C18	114.4 (2)
C2—C3—C4	111.6 (1)	N1—C19—C20	123.3 (1)
C3—C4—C5	114.9 (1)	C19—C20—C21	117.9 (1)
C4—C5—C6	113.4 (1)	C19—C20—C24	118.99 (10)
C5—C6—C7	114.6 (1)	C21—C20—C24	123.1 (1)
C6—C7—C8	114.6 (1)	C20—C21—C22	119.1 (1)
C7—C8—C9	114.5 (1)	C21—C22—C23	119.0 (1)
C8—C9—C10	114.5 (1)	N1—C23—C22	122.7 (1)
C9—C10—C11	114.4 (1)	O3—C24—N2	122.0 (1)
C10—C11—C12	114.3 (1)	O3—C24—C20	120.67 (10)
C11—C12—C13	114.2 (1)	N2—C24—C20	117.32 (9)

Table 2. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

D—H...A	D—H	H...A	D...A	D—H...A
O2—H1...N1	1.02 (1)	1.67 (1)	2.693 (1)	174 (1)
N2 <sup>i</sup> —H41 <sup>i</sup> ...O3	0.96 (1)	1.98 (1)	2.939 (1)	172 (1)
N2 <sup>ii</sup> —H42 <sup>ii</sup> ...O3	0.94 (1)	2.04 (2)	2.904 (1)	153 (1)

Symmetry codes: (i)  $-1 - x, -y, -z$ ; (ii)  $x, y, z - 1$ .

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *MULTAN88* (Debaerdemaeker *et al.*, 1988). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

We wish to thank Dr S. Shiro of the Rigaku Corporation for his helpful assistance in the diffractometry measurements. We also thank Mr M. Usuda for his continued assistance in this investigation.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1032). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). **C54**, 1369–1372

## 6,6'-Dimethylbiphenyl-2,2'-dicarboxylic Acid (6,6'-Dimethyldiphenic Acid)

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(Received 2 March 1998; accepted 6 April 1998)

## Abstract

The title compound,  $\text{C}_{16}\text{H}_{14}\text{O}_4$ , crystallized in the centrosymmetric space group  $P2_1/c$  with a single molecule in the asymmetric unit. The two carboxyl H atoms are ordered and are involved in hydrogen bonds whose  $\text{O}_d \cdots \text{O}_a$  distances are 2.660 (2) and 2.641 (2)  $\text{\AA}$ . Each of the hydrogen bonds forms a cyclic hydrogen-bonded dimer about a center of symmetry. Thus, each molecule is linked to two others to form a chain. The structure comprises two sets of such chains linked, not by conventional hydrogen bonds, but by one significantly attractive  $\text{C—H} \cdots \text{O}$  interaction. The intramolecular dihedral angle between the ring planes (twist angle) is 84.9 (1)°. The dihedral angles between the carboxyl-group planes and the planes of the rings to which they are attached are 36.7 (1) and 21.4 (1)°. A number of comparisons are made with the structure of the 'parent' compound, biphenyl-2,2'-dicarboxylic acid.

## Comment

This report on 6,6'-dimethylbiphenyl-2,2'-dicarboxylic acid, (I), is one of a series on hydrogen bonding in biphenylcarboxylic acids which includes biphenyl-