Crystal Structure of *N,N'*-Bis(*trans*-cinnamaldehyde)ethylenediimine

Aliakbar Dehno KHALAJI*† and Matthias WEIL**

*Department of Science, Gorgan University of Agricultural Sciences and Natural Resources, Gorgan, Iran

**Institute for Chemical Technologies and Analytics, Division of Structural Chemistry, Vienna University of Technology, Getreidemarkt 9/164-SC, A-1060 Vienna, Austria

The title compound, *N,N'*-bis(*trans*-cinnamaldehyde)ethylenediimine (ca₂en), C₂₀H₂₀N₂, was synthesized and structurally characterized by single-crystal X-ray diffraction. The compound crystallizes with two formula units in the monoclinic space group *P*2₁/*c* with lattice parameters (295 K) of *a* = 13.542(3), *b* = 7.6459(17), *c* = 8.1023(18) Å and β = 96.043(4)°. The molecule lies across a crystallographic inversion centre and has a *trans* configuration (*E, E*).

(Received May 9, 2007; Accepted May 10, 2007; Published on web October 25, 2007)

Bidentate Schiff bases are important ligands in the development of transition-metal complexes, such as copper(I)¹⁻³ and silver(I) complexes.^{4,5} The crystal structures of some diimine Schiff bases have already been described.⁶⁻⁹ In the present work, we report on the crystal structure of another diimine Schiff base, viz. *N,N'*-bis(*trans*-cinnamaldehyde)ethylenediimine (ca₂en) (Fig. 1).

The title compound (ca₂en) was prepared by the reaction of *trans*-cinnamaldehyde with ethylene diamine (molar ratio 2:1) in a methanol solution at 298 K. The white precipitate was filtered off and dried under a vacuum. Colorless single crystals of ca₂en were obtained by slow evaporation of an acetone-chloroform solution (1:1 v/v) of this material at 273 K.

A colorless single crystal with a plate-like habit and dimensions of 0.65 × 0.30 × 0.03 mm³ was chosen for a single-crystal X-ray diffraction study. Data were collected on a SMART-APEX 1 CCD diffractometer (graphite monochromator, Mo *K*_α radiation, 0.71073 Å) at 295 K. Details of the data-collection and refinement parameters are listed in Table 1. The structure was solved by direct methods. The positions of the H atoms were placed at geometrically idealized positions and constrained to ride on their parent atoms with *U*_{iso}(H) = 1.2*U*_{eq}(C).

The atomic coordinates for non-hydrogen atoms are listed in Table 2 and selected interatomic distances and angles are gathered in Table 3.

In the asymmetric unit of the title compound there is one-half of a C₆H₅CHCHCHNCH₂CH₂NCHCHCHC₆H₅ molecule. The C(10)–C(10) (–*x*+1, –*y*, –*z*–1) bond lies on a crystallographic inversion center, and the two C₆H₅CHCHCHN groups are in

trans orientation. An ORTEP plot of the molecular structure with the atomic numbering scheme is depicted in Fig. 2.

The C(1)–N(1) bond length of 1.2580(18) Å and the C(10)–N(1) bond length of 1.4573(17) Å conform to the values for a double and single bond, respectively. The C(1)–N(1) bond is slightly shorter than the corresponding bond in *N,N'*-bis(3-nitrobenzylidene) butane-1,4-diamine [1.267(3) Å],⁸ but is longer than that in *N,N'*-bis(2-nitrobenzylidene) ethane-1,2-diamine [1.250(2) Å].⁹ The C(10)–N(1)–C(1) and N(1)–C(1)–C(2) bond angles are 116.91(13)° and 123.25(14)°,

Table 1 Crystal and experimental data

Empirical formula: C ₂₀ H ₂₀ N ₂
Formula weight = 288.386
Crystal system: monoclinic
Space group: <i>P</i> 2 ₁ / <i>c</i>
Unit cell dimensions:
<i>a</i> = 13.542(3) Å
<i>b</i> = 7.6459(17) Å
<i>c</i> = 8.1023(18) Å
β = 96.043(4)°
<i>V</i> = 834.2(3) Å ³
<i>D</i> _x = 1.148 g/cm ³
No. of reflections used = 2081
2 θ _{max} = 56.72°
<i>R</i> (1398 reflections) = 0.0545
<i>wR</i> ₂ (2081 reflections) = 0.1579
($\Delta\rho$) _{max} = 0.007
($\Delta\rho$) _{max} = 0.161 e Å ^{–3}
($\Delta\rho$) _{max} = –0.116 e Å ^{–3}
Measurement: Bruker SMART 1K CCD diffractometer
Data collection: SMART (Bruker, 2002)
Structure solution (direct methods): SHELXS-97 (Sheldrick, 1997)
Structure refinement (full matrix least-squares on <i>F</i> ²): SHELXL-97 (Sheldrick, 1997)

CCDC 632486 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.uk/data_request/cif.

Fig. 1 Chemical structure of the title compound.

† To whom correspondence should be addressed.
E-mail: alidkhalaji@yahoo.com

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters

Atom	x	y	z	$U_{eq}(\text{\AA}^2)$
N(1)	0.46193(10)	0.03869(16)	-0.28961(14)	0.0662(4)
C(1)	0.37592(11)	0.07258(17)	-0.25331(17)	0.0571(4)
C(2)	0.34309(11)	0.03461(18)	-0.09288(17)	0.0570(4)
C(3)	0.25139(11)	0.06755(18)	-0.05899(17)	0.0595(4)
C(4)	0.20820(10)	0.03058(19)	0.09549(16)	0.0578(4)
C(5)	0.25954(12)	-0.0575(2)	0.22693(19)	0.0684(4)
C(6)	0.21567(15)	-0.0934(2)	0.3689(2)	0.0801(5)
C(7)	0.11957(16)	-0.0425(3)	0.3817(2)	0.0917(6)
C(8)	0.06789(14)	0.0467(3)	0.2538(2)	0.0988(7)
C(9)	0.11157(12)	0.0827(2)	0.1119(2)	0.0797(5)
C(10)	0.48324(12)	0.08050(19)	-0.45773(17)	0.0648(4)

Table 3 Selected bond distances (Å) and angles (°)

C(2)-C(3)	1.324(2)	C(3)-C(2)-C(1)	122.12(14)
C(2)-C(1)	1.4467(18)	C(1)-N(1)-C(10)	116.91(13)
N(1)-C(1)	1.2580(18)	N(1)-C(1)-C(2)	123.25(14)
N(1)-C(10)	1.4573(17)	C(2)-C(3)-C(4)	127.48(15)
C(4)-C(3)	1.4638(18)	N(1)-C(10)-C(1)	110.21(14)

Fig. 2 Molecular structure of *N,N'*-bis(*trans*-cinnamaldehyde) ethylenediimine with the atom numbering scheme. The given displacement ellipsoids are drawn at the 30% probability level. All non-labeled C and N atoms are generated by the symmetry operator $-x+1, -y, -z-1$.

respectively. These bond lengths and angles are in good agreement with the corresponding lengths and angles in related

Fig. 3 Crystal packing of the title compound, viewed along the *c*-axis. H atoms have been omitted for clarity.

Schiff base compounds.⁶⁻⁹ The crystal packing of the title compound is shown in Fig. 3.

References

1. A. D. Khalaji and R. Welter, *Inorg. Chim. Acta*, **2006**, 359, 4403.
2. A. D. Khalaji, M. Amirasr, and R. Welter, *Anal. Sci.*, **2006**, 22, x51.
3. M. Amirasr, A. D. Khalaji, and L. R. Falvello, *Inorg. Chim. Acta*, **2006**, 359, 713.
4. M. Amirasr, A. D. Khalaji, L. R. Falvello, and T. Soler, *Polyhedron*, **2006**, 25, 1967.
5. A. D. Khalaji, K. Aoki, and M. Amirasr, *J. Coord. Chem.*, **2007**, 60, 201.
6. S.-P. Yang, L.-J. Han, D.-Q. Wang, and H.-T. Xia, *Acta Crystallogr.*, **2007**, E63, o141.
7. S.-P. Yang, L.-J. Han, D.-Q. Wang, and T.-Z. Ding, *Acta Crystallogr.*, **2007**, E63, o361.
8. Y.-G. Li, H.-L. Zhu, X.-Z. Chen, and Y. Song, *Acta Crystallogr.*, **2005**, E61, o4156.
9. M. H. Habibi, K. Barati, M. Zendehtdel, R. W. Harrington, and W. Clegg, *Anal. Sci.*, **2007**, 23, x61.