

***N*-Methyl-*N*-*cis*-styrylcinnamamide monohydrate,
a compound of *Clausena lansium* (Lour.) Skeels****Xue-Song Huang,* Shi-Yi Ou and
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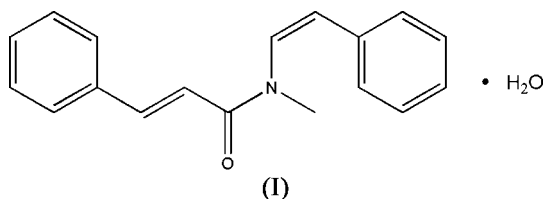
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Received 13 March 2006
Accepted 15 April 2006**Key indicators**Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.042
 wR factor = 0.123
Data-to-parameter ratio = 17.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The anhydrous form of the title compound, $\text{C}_{18}\text{H}_{17}\text{NO}\cdot\text{H}_2\text{O}$, was isolated from the dried seed of *Clausena lansium* (Lour.) Skeels. The liquid anhydrous form absorbs water from humid air to form crystals of the title compound. In the crystal structure, the conformations of the styryl and cinnamamide groups are *E* and *Z*, respectively. The uncoordinated water molecules are hydrogen bonded to the organic molecules.

Comment

As part of our efforts to search for antitumor and antiviral compounds from natural sources, an investigation of the chemical components of *Clausena lansium* has been undertaken. *N*-Methyl-*N*-*cis*-styrylcinnamamide was previously isolated from the seed of *C. lansium* (Lin, 1989) and synthesized in the laboratory by Stefanuti *et al.* (2000). It is a pale-yellow liquid at room temperature and its structure was elucidated by spectroscopic methods (Lin, 1989). Recently, we isolated this compound from the seed of *C. lansium* using a modified procedure (see below) and obtained single crystals of *N*-methyl-*N*-*cis*-styrylcinnamamide monohydrate, (I), from a CHCl_3 solution in humid air.



The molecular structure of (I) is shown in Fig. 1. The conformations in the styryl and cinnamamide groups are *E* and *Z*, respectively. This is in agreement with the results of spectroscopic research (Lin, 1989).

The crystal structure is stabilized by hydrogen bonds (Fig. 2 and Table 2).

The corresponding anhydrous compound of (I), *N*-methyl-*N*-*cis*-styrylcinnamamide, is a pale yellow liquid at room temperature. This liquid absorbs water from humid air to form the title hydrate, (I). The melting point of (I) should be higher than the corresponding anhydrous compound, thus (I) crystallizes from the anhydrous liquid at room temperature. The crystals of (I) easily transform to a pale-yellow liquid if put in a silica gel desiccator at room temperature. This confirms the lower melting point for the anhydrous compound of (I).

Experimental

Pulverized dried seeds were extracted with 80% ethanol solution three times under reflux. The extract was concentrated *in vacuo*, then

suspended in distilled water and partitioned successively with petroleum ether, CHCl_3 and EtOAc. The CHCl_3 fraction was subjected to column chromatography over silica gel and eluted with a gradient hexane–EtOAc system (from 0:100 to 100:0) to afford 36 fractions. Fraction 14 (about 120 ml) was kept at room temperature in humid air for 3 d; a small amount of viscous liquid of a pale-yellow color separated. Single crystals of (I) grew in the viscous liquid after one week. The crystals of (I) turn into a pale-yellow liquid if put in a silica gel desiccator.

Crystal data

$\text{C}_{18}\text{H}_{17}\text{NO}\cdot\text{H}_2\text{O}$ $D_x = 1.197 \text{ Mg m}^{-3}$
 $M_r = 281.34$ Mo $K\alpha$ radiation
 Tetragonal, $I4_1/a$ $\mu = 0.08 \text{ mm}^{-1}$
 $a = 30.7601(8) \text{ \AA}$ $T = 293(2) \text{ K}$
 $c = 6.6023(4) \text{ \AA}$ Prism, pale yellow
 $V = 6247.0(4) \text{ \AA}^3$ $0.48 \times 0.31 \times 0.30 \text{ mm}$
 $Z = 16$

Data collection

Bruker SMART 1000 CCD 3406 independent reflections
 diffractometer 2118 reflections with $I > 2\sigma(I)$
 φ and ω scans $R_{\text{int}} = 0.036$
 Absorption correction: none $\theta_{\text{max}} = 27.1^\circ$
 17817 measured reflections

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 3.2119P]$
 $R[F^2 > 2\sigma(F^2)] = 0.042$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.123$ $(\Delta/\sigma)_{\text{max}} = 0.001$
 $S = 1.00$ $\Delta\rho_{\text{max}} = 0.12 \text{ e \AA}^{-3}$
 3406 reflections $\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
 199 parameters
 H atoms treated by a mixture of independent and constrained refinement

Table 1

Selected bond lengths (\AA).

C7–C8	1.316 (2)	C10–C11	1.323 (3)
C9–O1	1.230 (2)	C10–N1	1.412 (2)
C9–N1	1.355 (2)	C18–N1	1.462 (2)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
$\text{O2W}\text{---}H2B\cdots\text{O1}^i$	0.93 (3)	1.89 (3)	2.818 (2)	174 (2)
$\text{O2W}\text{---}H2A\cdots\text{O2W}^{ii}$	0.85 (3)	1.93 (3)	2.773 (2)	173 (2)

Symmetry codes: (i) $x, y, z + 1$; (ii) $y + \frac{1}{4}, -x + \frac{5}{4}, z + \frac{1}{4}$.

H atoms of water molecules were located in difference Fourier maps and refined isotropically. Methyl H atoms were positioned geometrically with $\text{C}\text{---}H = 0.96 \text{ \AA}$ and their torsion angles refined to fit the electron density [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$]. Other H atoms were

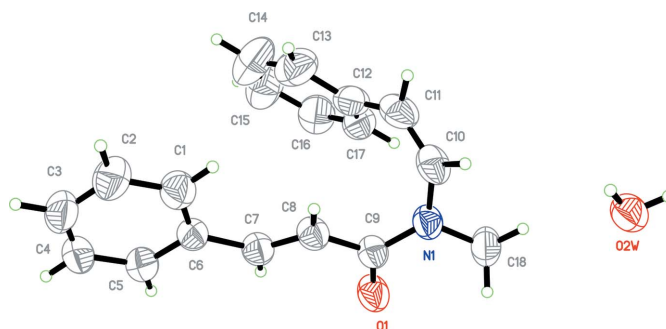


Figure 1

The structure of (I), with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

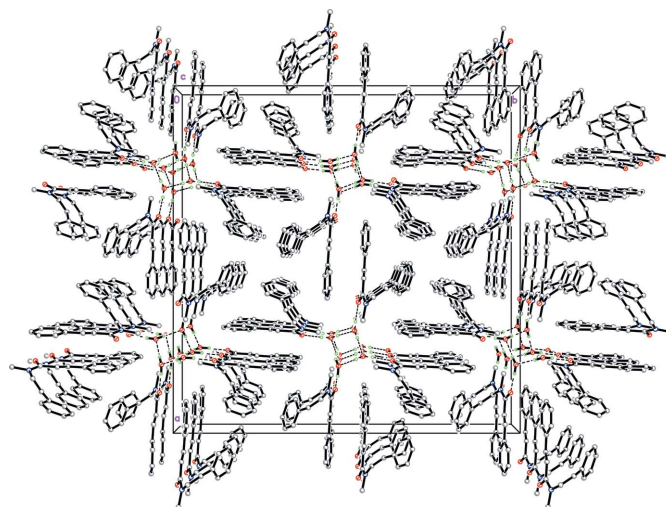


Figure 2

The unit-cell packing; dashed lines indicate the hydrogen-bonding network. H atoms not involved in hydrogen bonding have been omitted.

positioned geometrically with $\text{C}\text{---}H = 0.93 \text{ \AA}$ and refined as riding [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$].

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE-Plus* (Bruker, 2003); data reduction: *SAINTE-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2005); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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