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**(1E,3Z,5E)-2,5-Diaza-1,6-bis(dimethyl-amino)-1,3,5-hexatriene-3,4-dicarbonitrile**

HASAN KÜÇÜKBAY,<sup>a</sup> ENGIN ÇETINKAYA,<sup>a</sup> DİNÇER ÜLKÜ<sup>b\*</sup>  
AND M. NAWAZ TAHİR<sup>b</sup>

<sup>a</sup>Department of Chemistry, İnönü University, Malatya 44069, Turkey, and <sup>b</sup>Department of Engineering Physics, Hacettepe University, Beytepe 06532, Ankara, Turkey.  
E-mail: dulku@eti.cc.hun.edu.tr

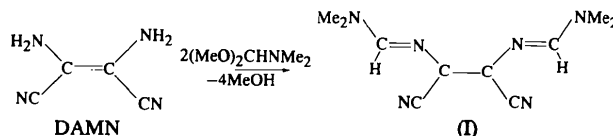
(Received 5 December 1995; accepted 1 April 1996)

**Abstract**

The title structure consists of discrete C<sub>10</sub>H<sub>14</sub>N<sub>6</sub> molecules which have a *cis* configuration and an extended conjugation. The molecules are slightly distorted from planarity.

**Comment**

*N,N*-Dimethylformamide dimethyl acetal reacts as a formylating agent with primary amines providing amidines which may be used in the synthesis of more complex molecules (Abdullah & Brinkmeyer, 1979; Williams & Brown, 1995), for example, *N,N*-dimethylformamide dimethyl acetal is known to react with *o*-phenylenediamine to give benzimidazole (Stanovnik & Tisler, 1974). In contrast, the analogous reaction with diaminomaleonitrile (DAMN) under mild conditions affords bis(amidine) (I), instead of the expected 4,5-dicyanoimidazole heterocycle, in almost quantitative yield. Since the stereochemistry of (I) could not be established definitely on the basis of spectroscopic data, an X-ray analysis was conducted.



The title molecule has a *cis* configuration and an extended conjugation (Fig. 1). Intramolecular bond lengths and angles have usual values. There are no

unusual close intermolecular contacts and the asymmetric unit is not quite planar. The dihedral angles between the planes defined by *A* (C5, N3, C3, N2), *B* (C10, N6, C8, N5) and *C* (N1, C1, C2, C7, C6, N4) are *A/B* 6 (1), *B/C* 8 (1) and *A/C* 3 (2)°.

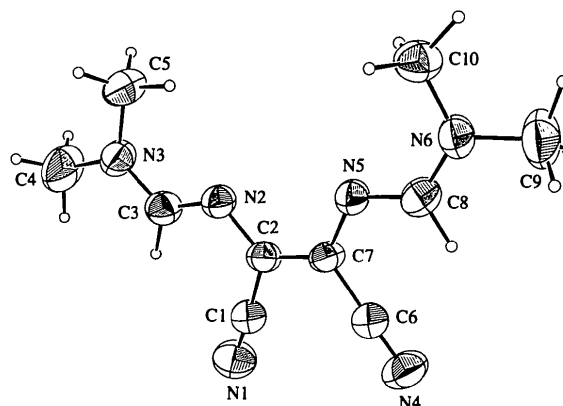


Fig. 1. An ORTEP (Johnson, 1965) drawing of (I) with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small circles of arbitrary radii.

**Experimental**

A mixture of diaminomaleonitrile (10 g, 92.59 mmol) and *N,N*-dimethylformamide dimethyl acetal (28 ml, 210.08 mmol) in toluene (20 ml) was heated for 1 h in a water bath and for an additional 30 min at 403 K in order to remove the methanol formed. All volatiles were then driven off *in vacuo*. The residual crude brown product was crystallized from dimethyl sulfoxide (35 ml) to afford crystals of the title compound.

*Crystal data*

C<sub>10</sub>H<sub>14</sub>N<sub>6</sub>  
*M<sub>r</sub>* = 218.263  
Monoclinic  
*P*2<sub>1</sub>/*a*  
*a* = 7.671 (1) Å  
*b* = 20.414 (2) Å  
*c* = 8.326 (2) Å  
*β* = 112.39 (2)°  
*V* = 1205.6 (5) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.203 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

*Data collection*

Enraf–Nonius CAD-4 diffractometer  
*ω*/*2θ* scans  
Absorption correction: empirical *via ψ* scans (*MolEN*; Fair, 1990)  
*T<sub>min</sub>* = 0.967, *T<sub>max</sub>* = 0.998  
2711 measured reflections  
2343 independent reflections

Mo *Kα* radiation  
*λ* = 0.71073 Å  
Cell parameters from 25 reflections  
*θ* = 10.68–18.18°  
*μ* = 0.075 mm<sup>-1</sup>  
*T* = 295 K  
Prism  
0.35 × 0.25 × 0.15 mm  
Brown

1098 observed reflections  
[*I* > 3σ(*I*)]  
*R<sub>int</sub>* = 0.015  
*θ<sub>max</sub>* = 26.32°  
*h* = 0 → 9  
*k* = 0 → 25  
*l* = -10 → 9  
3 standard reflections  
frequency: 120 min  
intensity decay: 4.76%

## Refinement

Refinement on *F**R* = 0.045*wR* = 0.043*S* = 0.76

1098 reflections

145 parameters

Unit weights applied

 $(\Delta/\sigma)_{\max} = 0.00014$ 

$$\Delta\rho_{\max} = 0.12 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Atomic scattering factors  
from *International Tables*  
for *X-ray Crystallography*  
(1974, Vol. IV)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: SK1005). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub>
N1	0.4286 (4)	0.2827 (2)	−0.1504 (4)	5.66 (9)
N2	0.8457 (4)	0.3047 (1)	0.2060 (3)	3.55 (6)
N3	0.9199 (4)	0.2034 (1)	0.3383 (4)	4.27 (7)
N4	0.5004 (4)	0.4662 (2)	−0.2117 (4)	5.51 (9)
N5	0.8922 (4)	0.4414 (1)	0.1704 (3)	3.46 (6)
N6	1.0184 (4)	0.5439 (1)	0.2638 (4)	4.26 (8)
C1	0.5564 (5)	0.3099 (2)	−0.0570 (4)	3.90 (9)
C2	0.7210 (4)	0.3404 (2)	0.0716 (4)	3.39 (8)
C3	0.8128 (4)	0.2425 (2)	0.2130 (4)	3.88 (8)
C4	0.8711 (6)	0.1348 (2)	0.3419 (6)	7.3 (1)
C5	1.0810 (5)	0.2278 (2)	0.4829 (5)	4.9 (1)
C6	0.6040 (5)	0.4387 (2)	−0.0962 (4)	3.90 (8)
C7	0.7438 (4)	0.4062 (2)	0.0533 (4)	3.41 (8)
C8	0.8909 (5)	0.5044 (2)	0.1546 (4)	4.09 (9)
C9	1.0004 (6)	0.6147 (2)	0.2419 (6)	6.6 (1)
C10	1.1707 (5)	0.5195 (2)	0.4160 (5)	4.8 (1)

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## Coumurrayin

RAJNIKANT,<sup>a</sup> VIVEK K. GUPTA,<sup>a</sup> ATTAR SINGH,<sup>a</sup> MADAN LAL<sup>a</sup> AND BABU VARGHESE<sup>b</sup>

<sup>a</sup>*X-ray Crystallography Laboratory, Department of Physics, University of Jammu, Jammu Tawi-180 004, India, and*  
<sup>b</sup>*Regional Sophisticated Instrumentation Centre, Indian Institute of Technology, Madras-600 036, India*

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Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N1—C1	1.137 (4)	N5—C8	1.291 (5)
N2—C2	1.373 (4)	N6—C8	1.326 (5)
N2—C3	1.299 (5)	N6—C9	1.457 (5)
N3—C3	1.323 (4)	N6—C10	1.446 (5)
N3—C4	1.452 (5)	C1—C2	1.449 (5)
N3—C5	1.447 (4)	C2—C7	1.370 (5)
N4—C6	1.135 (4)	C6—C7	1.457 (5)
N5—C7	1.385 (4)		
C2—N2—C3	118.0 (3)	N2—C2—C1	120.9 (4)
C3—N3—C4	120.8 (3)	N2—C2—C7	122.2 (3)
C3—N3—C5	121.7 (3)	C1—C2—C7	116.9 (3)
C4—N3—C5	117.4 (3)	N2—C3—N3	123.6 (3)
C7—N5—C8	118.2 (3)	N4—C6—C7	177.1 (5)
C8—N6—C9	120.6 (4)	N5—C7—C2	122.4 (3)
C8—N6—C10	122.0 (3)	N5—C7—C6	120.3 (4)
C9—N6—C10	117.3 (3)	C2—C7—C6	117.2 (4)
N1—C1—C2	175.6 (4)	N5—C8—N6	124.1 (4)

All H atoms were taken from difference maps and assigned *U*<sub>iso</sub> values equal to 1.3*U*<sub>eq</sub> of the parent atoms. A riding model was adopted.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN SIR*. Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *MolEN ORTEP* (Johnson, 1965). Software used to prepare material for publication: *MolEN*.

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## Abstract

In the title compound, 5,7-dimethoxy-8-(3-methyl-2-butenyl)-2*H*-1-benzopyran-2-one, C<sub>16</sub>H<sub>18</sub>O<sub>4</sub>, the coumarin ring system is planar. The methoxy groups at C5 and C7 are coplanar with the coumarin moiety. The side group located at C8 is planar and at an angle of 108.29 (7)° with respect to the coumarin nucleus. The crystal structure is stabilized by van der Waals interactions.

## Comment

Coumarin derivatives are biologically important (Michel & Durant, 1976; Schmalte, Jarchow, Hausen & Schulz, 1982). The structure of coumurrayin, (I), a coumarin isolated from the roots of *Seseli Sibiricum* Benth. (Kumar, Gupta, Banerjee & Atal, 1978), is presented in this paper as a part of our programme on the crystal structure analysis of some naturally occurring coumarins (Rajnikant, Goswami, Yadava & Padmanabhan, 1991, 1993; Rajnikant, Goswami, Yadava, Padmanabhan, Gupta & Banerjee, 1993; Gupta *et al.*, 1993; Magotra, Gupta, Rajnikant, Goswami & Thappa, 1995).