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Key indicators

Single-crystal X-ray study
T = 100 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.061
wR factor = 0.165
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

3-(Methylaminomethylene)pentane-2,4-dione

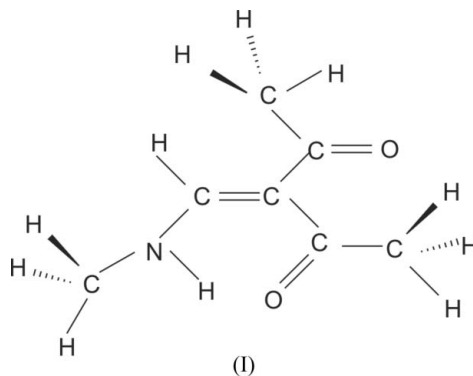
The conformations of NH and CH₃ of the title compound, C₇H₁₁NO₂, are mainly stabilized by intramolecular N—H···O and C—H···O hydrogen bonds of 1.983, 2.579 and 2.485 Å, as well as intermolecular N—H···O and C—H···O hydrogen bonds of 2.274, 2.520, 2.484, 2.713 and 2.683 Å.

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Comment

3-(Methylaminomethylene)pentane-2,4-dione, (I), belongs to the so-called push–pull olefins. Push–pull ethylenes represent a specific group of highly reactive organic compounds characterised by the presence of different substituents on each side of the C=C double bond (Cook, 1969). Their general formula can be expressed in the form R₁X—CR₂=CR₃R₄. R₁, R₂ (for X = O and X = NH or NR₁) can be hydrogen, alkyl or heteroaryl groups, and R₃, R₄ are strong electron-acceptor groups such as —CN, —COR or —COOR. Enamines (X = NH, NR) are frequently used for many organic syntheses in the field of pharmaceuticals, polymers and dyes (Dyke, 1973). Properties such as the polar character, electronic interactions between substituents and the double bond are responsible for their non-linear optical properties and their use as new electro-optic materials (Kolev *et al.*, 2003). The conformations of NH and CH₃ of the title compound, C₇H₁₁NO₂, are mainly stabilized by intramolecular N—H···O and C—H···O hydrogen bonds, as well as intermolecular N—H···O and C—H···O hydrogen bonds (Table 1).



Experimental

To 3-(methoxymethylene)pentane-2,4-dione (1.42 g, 10 mmol) or 3-(ethoxymethylene)pentane-2,4-dione (1.56 g, 10 mmol) in methanol (10 ml) (ethanol for ethoxy derivative) was added dropwise an ethanol methylamine solution (12 mmol; amount according to concentration and density) over a period of 30 min while stirring the reaction mixture with a magnetic stirrer. The mixture was warmed slightly and stirred overnight at room temperature. The reaction

mixture was then heated under reflux (*ca* 20 min, adding a reflux condenser to the reaction flask). After disappearance of the starting material (thin-layer chromatography; Silufol 254, Kavalier, Czechoslovakia; eluent: chloroform–methanol 10:1, detection: UV light 254 nm), the reaction mixture was evaporated on a vacuum evaporator and chromatographed on silica gel (eluent: dichloromethane–methanol 10:1).

Crystal data

$C_7H_{11}NO_2$	$Z = 4$
$M_r = 141.17$	$D_x = 1.252 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 3.9640 (3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 20.3808 (9) \text{ \AA}$	$T = 100 \text{ K}$
$c = 9.4566 (6) \text{ \AA}$	Block, colourless
$\beta = 101.453 (6)^\circ$	$0.51 \times 0.12 \times 0.11 \text{ mm}$
$V = 748.78 (8) \text{ \AA}^3$	

Data collection

Oxford Diffraction GEMINI R diffractometer	15570 measured reflections
ω and φ scans	1359 independent reflections
Absorption correction: analytical (Clark & Reid, 1995)	1206 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.652$, $T_{\max} = 0.719$	$R_{\text{int}} = 0.033$
	$\theta_{\max} = 25.4^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1094P)^2 + 0.1705P]$
$R[F^2 > 2\sigma(F^2)] = 0.061$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.165$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.16$	$\Delta\rho_{\max} = 0.59 \text{ e \AA}^{-3}$
1359 reflections	$\Delta\rho_{\min} = -0.29 \text{ e \AA}^{-3}$
95 parameters	
H-atom parameters constrained	

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N-H2\cdots O1$	0.86	1.98	2.622 (1)	130
$C1-H1A\cdots O2$	0.96	2.58	2.771 (1)	91
$C1-H1B\cdots O2$	0.96	2.49	2.771 (1)	97
$N-H2\cdots O1^i$	0.86	2.27	2.946 (1)	135
$C7-H7C\cdots O1^{ii}$	0.96	2.52	3.455 (3)	165
$C6-H6\cdots O2^{iii}$	0.93	2.68	3.593 (2)	166
$C5-H5A\cdots O2^{iii}$	0.96	2.71	3.353 (1)	125
$C5-H5B\cdots O2^{iv}$	0.96	2.48	3.419 (3)	165

Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $-x - 1, -y + 1, -z + 1$; (iii) $x - 1, -y + \frac{1}{2}, z - \frac{1}{2}$; (iv) $x, -y + \frac{1}{2}, z - \frac{1}{2}$.

Methyl, olefinic and amino H atoms were positioned geometrically and allowed to ride on their corresponding parent atoms at distances of 0.96, 0.93 and 0.86 \AA , respectively, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2001); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2005); program(s) used to solve structure: *SHELXS97*

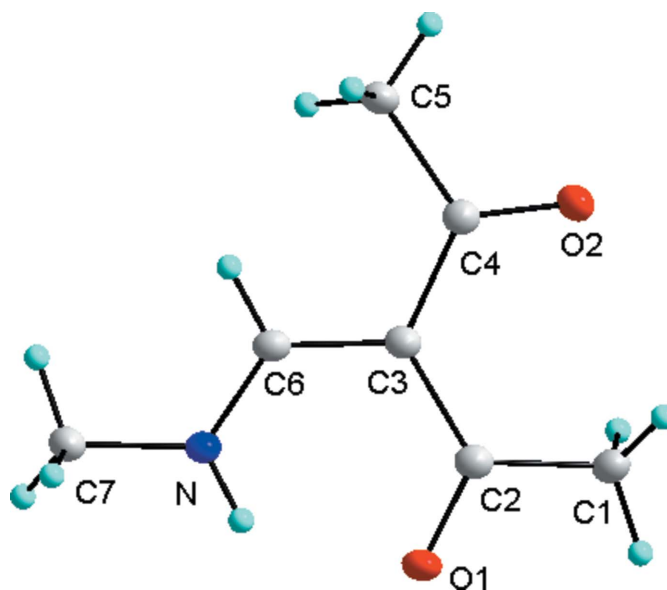


Figure 1

The numbering scheme of (I). Displacement ellipsoids are drawn at the 40% probability level.

(Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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