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Structure of a Substituted 2-Furylethylene Derivative

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Abstract. Methyl 2-cyano-3-(5-dimethylamino-2-furyl)-acrylate, $C_{11}H_{12}N_2O_3$, $M_r = 220{\cdot}2$, monoclinic, $P2_1/c$, $a = 10{\cdot}012\ (2)$, $b = 13{\cdot}820\ (3)$, $c = 8{\cdot}293\ (1)$ Å, $\beta = 106{\cdot}39\ (1)°$, $V = 1100{\cdot}8$ Å^3, $Z = 4$, $D_m = 1{\cdot}31$, $D_x = 1{\cdot}33$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}82$ mm^{-1}, $F(000) = 464$, $T = 293$ K. Final $R = 0{\cdot}065$ for 802 observed reflections. It is proved that in the reaction of 5-dimethylamino-2-furaldehyde with the methyl ester of cyanoacetic acid only the *E* isomer is produced. The molecules are nearly planar with dihedral angles between furan, methyl ester and dimethylamine planes ranging from 11·2 (2) to 23·6 (5)°. They are aligned to form stacks perpendicular to the *c* axis.

Experimental. Orange prismatic crystal with dimensions 0·43 × 0·09 × 0·08 mm; D_m by flotation in *n*-octane/CCl_4; monoclinic space group $P2_1/c$ (No. 14); lattice parameters and Bravais translation lattice found by program *UB* (Sivý, Sivý & Koreň, 1987). Intensities collected with Syntex $P2_1$ diffractometer, graphite monochromator, $\theta/2\theta$ scan, $2\theta_{max} = 110°$; time per reflection *ca* 60 s, two standard reflections, variation 2·2%; 25 reflections with $11{\cdot}2 < 2\theta < 26{\cdot}6°$ used for refinement of lattice parameters; min. and max. transmission factors are the same and equal 0·8475 (absorption correction not applied); index range $0 \le h \le 8$, $0 \le k \le 14$, $-10 \le l \le 10$; 1621 reflections measured, 1393 unique, $R_{int} = 0{\cdot}03$ (238 reflections used), 802 reflections observed with $I > 1{\cdot}5\sigma(I)$, 591 unobserved. Because of strong secondary extinction the following reflections were excluded: 200, 102, 1,13,2, 294, 494, 594, 694, 794, and $\bar{3}66$. Data reduction carried out with program *XP*21 (Pavelčík, 1987). A furan ring, entered as a randomly oriented and randomly positioned molecular group to program *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), gave for $E > 1{\cdot}5$ (174 reflections used) the full structure except for the C(16) atom, which was found from a difference Fourier map.

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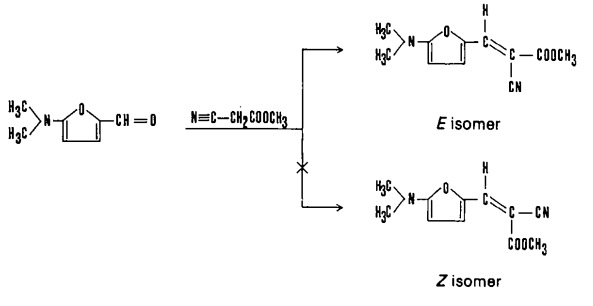
H atoms were generated by program *SHELX*76 (Sheldrick, 1976) and their coordinates and thermal parameters were fixed. Full-matrix least-squares refinement on F (145 parameters) by program *SHELX*76. Scattering factors and f', f'' from *International Tables for X-ray Crystallography* (1974). Max. positive and max. negative electron density in final difference Fourier synthesis 0·32 and −0·33 e Å^{-3}; final $R = 0{\cdot}065$, $wR = 0{\cdot}069$; $w = 1{\cdot}3565/[\sigma^2(F_o) + 0{\cdot}001458F_o^2]$; $(\Delta/\sigma)_{max} = 0{\cdot}001$ in final refinement cycle. Calculations performed using an M4030-1 computer, Slovak Technical University, Bratislava, Czechoslovakia.

The numbering of atoms is shown in Fig. 1. Fractional atomic coordinates for non-H atoms are in Table 1;* bond distances and angles are in Table 2. All interlayer interatomic constants in the present structure are larger than the sum of the respective van der Waals radii, *i.e.* O···O > 3·7, O···C > 3·1, O···N > 3·5, C···C > 3·5, C···N > 3·3, and N···N > 3·4 Å.

Related literature. Condensation reactions of 5-*X*-2-furaldehydes with the active methylene group of the *X*—CH_2—*Y* type, where *X* and *Y* are acceptor groups, can provide two isomeric products (Ilavský & Kováč, 1983) (see scheme below). The compound studied has an *E, s-trans* configuration.



* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and details of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51454 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

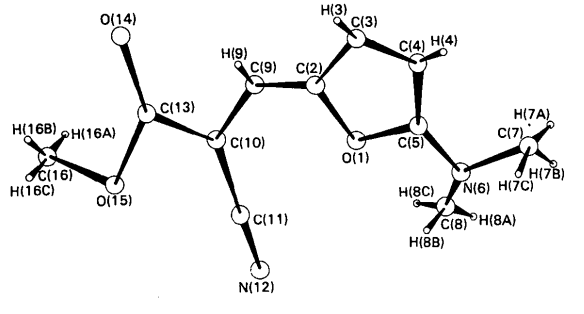


Fig. 1. Numbering of the molecule of $C_{11}H_{12}N_2O_3$.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$

	x	y	z	B_{eq}(Å^2)
O(1)	0·7714 (4)	0·2638 (3)	0·1276 (5)	4·1 (1)
C(2)	0·8680 (6)	0·2039 (4)	0·0799 (8)	4·2 (2)
C(3)	0·9648 (6)	0·2630 (5)	0·0390 (8)	4·8 (2)
C(4)	0·9313 (6)	0·3577 (5)	0·0617 (8)	4·8 (2)
C(5)	0·8125 (6)	0·3554 (4)	0·1152 (7)	4·1 (2)
N(6)	0·7355 (5)	0·4244 (4)	0·1568 (6)	4·6 (2)
C(7)	0·7678 (6)	0·5250 (5)	0·1328 (8)	5·4 (2)
C(8)	0·6024 (6)	0·4006 (5)	0·1860 (9)	5·3 (2)
C(9)	0·8527 (5)	0·1054 (4)	0·0811 (7)	4·3 (2)
C(10)	0·7590 (6)	0·0482 (4)	0·1283 (7)	4·0 (2)
C(11)	0·6513 (7)	0·0858 (4)	0·1928 (8)	4·5 (2)
N(12)	0·5649 (5)	0·1158 (4)	0·2431 (7)	5·8 (2)
C(13)	0·7638 (6)	−0·0560 (4)	0·1062 (7)	4·3 (2)
O(14)	0·8406 (5)	−0·0969 (3)	0·0372 (6)	6·0 (2)
O(15)	0·6734 (4)	−0·1049 (3)	0·1681 (5)	5·1 (1)
C(16)	0·6642 (7)	−0·2081 (4)	0·1422 (9)	6·1 (3)

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(1)–C(2)	1·412 (8)	N(6)–C(8)	1·458 (9)
–C(5)	1·344 (7)	C(10)–C(9)	1·366 (9)
C(2)–C(3)	1·381 (9)	–C(11)	1·430 (10)
–C(9)	1·370 (8)	–C(13)	1·454 (8)
C(3)–C(4)	1·377 (10)	C(11)–N(12)	1·139 (10)
C(4)–C(5)	1·382 (10)	C(13)–O(14)	1·219 (8)
N(6)–C(5)	1·331 (8)	–O(15)	1·342 (8)
–C(7)	1·454 (9)	O(15)–C(16)	1·441 (7)

C(2)–O(1)–C(5)	105·4 (5)	C(7)–N(6)–C(8)	119·2 (5)
O(1)–C(2)–C(3)	107·8 (5)	C(2)–C(9)–C(10)	131·6 (6)
–C(9)	119·6 (6)	C(9)–C(10)–C(11)	123·3 (5)
C(3)–C(2)–C(9)	132·6 (6)	–C(13)	119·2 (6)
C(2)–C(3)–C(4)	108·3 (6)	C(11)–C(10)–C(13)	117·5 (6)
C(3)–C(4)–C(5)	105·7 (6)	C(10)–C(11)–N(12)	179·5 (6)
O(1)–C(5)–C(4)	110·9 (5)	C(10)–C(13)–O(14)	124·5 (6)
–N(6)	115·3 (6)	–O(15)	113·6 (6)
C(4)–C(5)–N(6)	132·9 (6)	O(14)–C(13)–O(15)	121·9 (5)
C(5)–N(6)–C(7)	118·8 (6)	C(13)–O(15)–C(16)	117·6 (5)
–C(8)	120·3 (5)		

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