

Fig. 1. Perspective drawing of the results of the X-ray study on the dimer. Only one of the two molecules in the asymmetric unit is shown.

both the *cis* (Brossi *et al.*, 1988) and *trans* (Gerpe, Yeh, Brossi & Flippen-Anderson, 1988) (C15 relative to O16) forms of the deoxyarteether monomer have been reported. In the present compound the conformation of

the deoxyarteether moieties is the same as that found for the *trans* isomer. Ring *A* has a normal chair conformation while ring *B* has a slightly distorted chair conformation (absolute values for ring torsion angles vary from 48.5 to 59.1° for ring *A* and from 41.3 to 76.9° for ring *B*). Ring *D* has assumed a somewhat distorted conformation (absolute values for ring torsion angles vary from 15.0 to 73.5°) such that no unusual bond lengths or angles appear in the molecule. The five-membered ring has a normal envelope conformation with O1 being the out-of-plane atom. In the dimer, however, the methyl group on C11 and the oxygen atom on C12 are *gauche* with respect to one another (C15–C11–C12–O16 torsion angles range from –75.2 to –82.9°).

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A Tautomeric Form of Tetraethylammonium 1-Acrylamidononahydro-*closo*-decaborate(1–)

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Abstract. C₈H₂₀N⁺.C₃H₁₄B₁₀NO[–], *M_r* = 318.5, monoclinic, *P*2₁/*c*, *a* = 9.934 (4), *b* = 15.207 (4), *c* = 13.489 (5) Å, β = 94.16 (3)°, *V* = 2032.5 (11) Å³, *Z*

= 4, *D_x* = 1.041 g cm^{–3}, λ(Mo *K*α) = 0.71073 Å, μ(Mo *K*α) = 0.53 cm^{–1}, *F*(000) = 688, *T* = 295 K. Final *R* = 0.051 for 1929 observed reflections. The

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structure shows that the title compound consists of a $(C_2H_5)_4N^+$ cationic fragment and a *closo*- $B_{10}H_9^-$ anionic unit, in which a tautomeric form of the acrylamido moiety replaces a terminal hydrogen on one of the basal borons of the polyhedral cage. The average $B_{apical}-B_{basal}$ and $B_{basal}-B_{basal}$ bond distances in the upper half of the cage and the corresponding distances in the lower half of the cage are 1.693 (5), 1.820 (5), 1.686 (5) and 1.820 (5) Å, respectively. The remaining eight average B-B distances across the equatorial belt of the anion are 1.810 (5) Å. The *exo*-polyhedral B-N, N-C, C-O, C-C(O) and C-C bond distances and the average B-N-C, N-C-O, N-C-C, O-C-C and C-C-C bond angles are 1.523 (4), 1.289 (4), 1.304 (4), 1.448 (4) and 1.308 (4) Å, and 121.7 (3)°, respectively, thus confirming the planar geometry of the $HN=C(OH)-CH=CH_2$ moiety. This O-protonated amide moiety is greatly stabilized by its resonance structure. The geometry of the tetraethylammonium cation is approximately tetrahedral with an average C-N-C bond angle of 111.3 (3)°.

Introduction. The most convenient preparation of the $B_{10}H_{10}^{2-}$ ion was first reported in the latter part of the 1950's (Hawthorne & Pittochelli, 1959). Since then several salts of this dianion have been synthesized (Muetterties & Knoth, 1968; Muetterties, 1975; Greenwood & Thomas, 1973). Simple substitution of $B_{10}H_{10}^{2-}$ has been described for a wide range of substituents including -OH, -OR, -SH, -SR, -C(O)R, -OC(O)R, -R, -CN, -N₃, -C(O)NH₂, -NCO, -C(O)OR, -NH₂ and -NNAr. The diazonium derivative, $B_{10}H_8(N_2)_2$, serves as a useful intermediate for the preparation of a wide range of derivatives of the type $L_2B_{10}H_8$ since the nitrogen is easily displaced by a variety of weak and strong nucleophiles including NH₃, amines, N₃⁻, nitriles, OH⁻, H₂S and CO (Muetterties & Knoth, 1968; Muetterties, 1975; Jolly, 1971; Wiberg & Amberger, 1971; Greenwood & Morris, 1974; Greenwood & Thomas, 1973). An X-ray crystallographic study of $Cu_2B_{10}H_{10}$ has established the structure of $B_{10}H_{10}^{2-}$ as a bicapped Archimedean square antiprism of D_{4d} symmetry (Dobrott & Lipscomb, 1962). However, the crystal structures of the derivatives of *closo*- $B_{10}H_{10}^{2-}$ ion containing *exo*-polyhedral amide, acrylamide, isocyanate or diazo moieties have not been reported to date. In order to study the effects of substituents on the polyhedral cage geometry and to confirm the existence of tautomerism in the *exo*-polyhedral acrylamido moiety, we have conducted an X-ray investigation of the title compound and report herein the results.

Experimental. The title compound $[(C_2H_5)_4N][B_{10}H_9-NH=C(OH)CH=CH_2]$ (I), a tautomeric form of $[(C_2H_5)_4N][B_{10}H_9NH_2C(=O)CH=CH_2]$ (1a), was prepared as a yellow solid in a reaction involving

$[(C_2H_5)_4N][B_{10}H_9N\equiv C-CH=CH_2]$ and an excess of water at room temperature (Zhang, Zhu & Zhu, 1987, unpublished results). This solid was crystallized and then recrystallized from acetone to collect (I) in high purity and in 69% yield.

Crystals of (I) are yellow cubes, unit cell parameters by least-squares fit of 15 reflections in the range $10 \leq 2\theta \leq 25^\circ$, crystal dimensions 0.40 × 0.30 × 0.22 mm, space group $P2_1/c$ from systematic absences ($0k0$, k odd; $h0l$, l odd); automatic Nicolet $R3m/V$ diffractometer, graphite-monochromated $Mo K\alpha$ radiation, $\theta/2\theta$ scan mode (scan rate 6.0–30.0 min⁻¹, depending upon the intensity), 3896 measured reflections, 3576 independent reflections in the range $3 \leq 2\theta \leq 50^\circ$, $R_{int} = 0.012$, hkl range $h -11 \rightarrow 11$, $k 0 \rightarrow 18$, $l 0 \rightarrow 16$; 1929 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change (1.2%) in intensity during the data collection; Lorentz-polarization correction, no extinction corrections. Absorption correction based on ψ -scans; the maximum and minimum transmission factors were 0.999 and 0.895, respectively. Structure solved by direct-methods program used in Nicolet Software Package *SHELXTL-Plus* (Sheldrick, 1988), refinement by full-matrix least squares using *SHELXTL-Plus*, anisotropic; boron cage H atoms were located in difference Fourier maps, H atoms included with isotropic temperature factors; primary and secondary H atoms on carbons in the cationic fragment were constrained tetrahedrally; $w = 1/(\sigma^2F + 0.0070F^2)$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0.051$, $wR = 0.078$, $S = 0.86$, $(\Delta/\sigma)_{max} = 0.046$, $\Delta\rho_{(max, min)} = 0.12, -0.23 \text{ e \AA}^{-3}$ in final difference Fourier map. Atomic scattering factors for C, H, B and N were those stored in *SHELXTL-Plus*.

Discussion. The final atomic parameters of the non-H atoms are given in Table 1.* The bond lengths, bond angles and selected torsion angles with their standard deviations are given in Table 2. The identification of the atoms and the configuration of the molecule are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1. The structure shows that the title compound (I) consists of a $(C_2H_5)_4N^+$ cationic fragment and a *closo*- $B_{10}H_9^-$ anionic unit, in which a tautomeric form of the acrylamido moiety replaces a terminal hydrogen on one of the basal borons of the polyhedral cage. The average $B_{apical}-B_{basal}$ and $B_{basal}-B_{basal}$ bond distances in the upper half of the cage and the corresponding distances in the lower half of the cage are 1.693 (5), 1.820 (5), 1.686 (5) and

* Lists of structure factors, anisotropic temperature factors, selected torsion angles and H parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51424 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O	1382 (3)	9912 (2)	1052 (2)	97 (1)
N(1)	1934 (2)	8636 (2)	1829 (2)	46 (1)
C(1)	1529 (3)	9440 (2)	1861 (2)	50 (1)
C(2)	1210 (3)	9877 (2)	2768 (2)	53 (1)
C(3)	635 (3)	10651 (2)	2773 (3)	62 (1)
B(1)	2157 (3)	8120 (2)	888 (2)	46 (1)
B(2)	690 (3)	7569 (3)	312 (2)	55 (1)
B(3)	1609 (4)	8367 (3)	-395 (2)	59 (1)
B(4)	3226 (4)	8506 (2)	63 (3)	58 (1)
B(5)	3555 (3)	7477 (2)	487 (2)	50 (1)
B(6)	2073 (3)	6941 (2)	937 (2)	50 (1)
B(7)	1042 (4)	6530 (3)	-5 (3)	61 (1)
B(8)	1282 (3)	7282 (3)	-883 (2)	58 (1)
B(9)	2669 (3)	6658 (2)	-269 (2)	54 (1)
B(10)	2987 (3)	7717 (2)	-813 (2)	57 (1)
N(2)	3333 (2)	3958 (2)	2140 (2)	61 (1)
C(4)	2607 (3)	4280 (3)	1187 (3)	81 (1)
C(5)	3294 (6)	4074 (3)	253 (3)	107 (2)
C(6)	4694 (3)	4388 (2)	2341 (3)	75 (1)
C(7)	4676 (5)	5377 (3)	2442 (5)	115 (2)
C(8)	2420 (4)	4157 (3)	2969 (3)	86 (2)
C(9)	2941 (5)	3881 (4)	3996 (3)	104 (2)
C(10)	3635 (3)	2984 (2)	2086 (3)	79 (1)
C(11)	2399 (5)	2409 (3)	1889 (6)	127 (3)

Table 2. Bond lengths (\AA), bond angles ($^\circ$) and selected torsion angles ($^\circ$)

O—C(1)	1.304 (4)	N(1)—C(1)	1.289 (4)
N(1)—B(1)	1.523 (4)	N(2)—C(4)	1.509 (4)
N(2)—C(6)	1.511 (4)	N(2)—C(8)	1.520 (5)
N(2)—C(10)	1.513 (4)	C(1)—C(2)	1.448 (4)
C(2)—C(3)	1.308 (4)	C(4)—C(5)	1.508 (6)
C(6)—C(7)	1.510 (6)	C(8)—C(9)	1.502 (6)
C(10)—C(11)	1.516 (6)	B(1)—B(2)	1.809 (4)
B(1)—B(3)	1.814 (4)	B(1)—B(4)	1.697 (5)
B(1)—B(5)	1.812 (5)	B(1)—B(6)	1.796 (5)
B(2)—B(3)	1.828 (5)	B(2)—B(6)	1.826 (5)
B(2)—B(7)	1.680 (5)	B(2)—B(8)	1.809 (5)
B(3)—B(4)	1.693 (5)	B(3)—B(8)	1.799 (5)
B(3)—B(10)	1.812 (5)	B(4)—B(5)	1.687 (5)
B(4)—B(10)	1.690 (5)	B(5)—B(6)	1.826 (5)
B(5)—B(9)	1.801 (5)	B(5)—B(10)	1.838 (5)
B(6)—B(7)	1.695 (5)	B(6)—B(9)	1.823 (5)
B(7)—B(8)	1.675 (5)	B(7)—B(9)	1.692 (5)
B(8)—B(9)	1.820 (5)	B(8)—B(10)	1.816 (5)
B(9)—B(10)	1.806 (5)		
C(1)—O—H(10)	107 (3)	C(1)—N(1)—B(1)	125.7 (2)
C(1)—N(1)—H(1N)	113 (2)	B(1)—N(1)—H(1N)	121 (2)
C(4)—N(2)—C(6)	112.2 (3)	C(4)—N(2)—C(8)	106.8 (2)
C(6)—N(2)—C(8)	110.8 (3)	C(4)—N(2)—C(10)	111.0 (3)
C(6)—N(2)—C(10)	104.9 (2)	C(8)—N(2)—C(10)	111.2 (3)
O—C(1)—N(1)	120.5 (3)	O—C(1)—C(2)	115.9 (3)
N(1)—C(1)—C(2)	123.6 (2)	C(1)—C(2)—C(3)	122.7 (3)
C(1)—C(2)—H(2)	113 (2)	C(3)—C(2)—H(2)	124 (2)
C(2)—C(3)—H(31)	125 (2)	C(2)—C(3)—H(32)	126 (2)
H(31)—C(3)—H(32)	109 (3)	N(2)—C(4)—C(5)	115.3 (3)
N(2)—C(6)—C(7)	115.7 (3)	N(2)—C(8)—C(9)	116.0 (3)
N(2)—C(10)—C(11)	114.4 (3)	N(1)—B(1)—B(2)	115.9 (2)
N(1)—B(1)—B(3)	129.6 (2)	B(2)—B(1)—B(3)	60.6 (2)
N(1)—B(1)—B(4)	120.2 (2)	B(2)—B(1)—B(4)	113.7 (2)
B(3)—B(1)—B(4)	57.5 (2)	N(1)—B(1)—B(5)	133.4 (2)
B(2)—B(1)—B(5)	103.4 (2)	B(3)—B(1)—B(5)	90.5 (2)
B(4)—B(1)—B(5)	57.4 (2)	N(1)—B(1)—B(6)	118.4 (2)
B(2)—B(1)—B(6)	60.9 (2)	B(3)—B(1)—B(6)	103.2 (2)
B(4)—B(1)—B(6)	113.7 (2)	B(5)—B(1)—B(6)	60.8 (2)
B(1)—B(2)—B(3)	59.8 (2)	B(1)—B(2)—B(6)	59.2 (2)
B(3)—B(2)—B(6)	101.5 (2)	B(1)—B(2)—B(7)	111.8 (2)

Table 2 (cont.)

B(3)—B(2)—B(7)	111.8 (2)	B(6)—B(2)—B(7)	57.6 (2)
B(1)—B(2)—B(8)	101.0 (2)	B(3)—B(2)—B(8)	59.3 (2)
B(6)—B(2)—B(8)	90.2 (2)	B(7)—B(2)—B(8)	57.2 (2)
B(1)—B(3)—B(2)	59.6 (2)	B(1)—B(3)—B(4)	57.8 (2)
B(2)—B(3)—B(4)	113.0 (2)	B(1)—B(3)—B(8)	101.2 (2)
B(2)—B(3)—B(8)	59.8 (2)	B(4)—B(3)—B(8)	113.0 (3)
B(1)—B(3)—B(10)	90.2 (2)	B(2)—B(3)—B(10)	102.6 (2)
B(4)—B(3)—B(10)	57.6 (2)	B(8)—B(3)—B(10)	60.4 (2)
B(1)—B(4)—B(3)	64.7 (2)	B(1)—B(4)—B(5)	64.8 (2)
B(3)—B(4)—B(5)	99.3 (2)	B(1)—B(4)—B(10)	98.6 (2)
B(3)—B(4)—B(10)	64.8 (2)	B(5)—B(4)—B(10)	65.9 (2)
B(1)—B(5)—B(4)	57.9 (2)	B(1)—B(5)—B(6)	59.2 (2)
B(4)—B(5)—B(6)	112.7 (2)	B(1)—B(5)—B(9)	100.9 (2)
B(4)—B(5)—B(9)	112.1 (2)	B(6)—B(5)—B(9)	60.3 (2)
B(1)—B(5)—B(10)	89.4 (2)	B(4)—B(5)—B(10)	57.1 (2)
B(6)—B(5)—B(10)	101.9 (2)	B(9)—B(5)—B(10)	59.5 (2)
B(1)—B(6)—B(2)	59.9 (2)	B(1)—B(6)—B(5)	60.0 (2)
B(2)—B(6)—B(5)	102.2 (2)	B(1)—B(6)—B(7)	111.7 (2)
B(2)—B(6)—B(7)	56.8 (2)	B(5)—B(6)—B(7)	112.0 (2)
B(1)—B(6)—B(9)	100.6 (2)	B(2)—B(6)—B(9)	89.6 (2)
B(5)—B(6)—B(9)	59.2 (2)	B(7)—B(6)—B(9)	57.3 (2)
B(2)—B(7)—B(6)	65.5 (2)	B(2)—B(7)—B(8)	65.2 (2)
B(6)—B(7)—B(8)	99.6 (3)	B(2)—B(7)—B(9)	99.4 (3)
B(6)—B(7)—B(9)	65.1 (2)	B(8)—B(7)—B(9)	65.5 (2)
B(2)—B(8)—B(3)	60.9 (2)	B(2)—B(8)—B(7)	57.5 (2)
B(2)—B(8)—B(9)	113.5 (2)	B(2)—B(8)—B(9)	90.2 (2)
B(3)—B(8)—B(9)	101.5 (2)	B(7)—B(8)—B(9)	57.7 (2)
B(2)—B(8)—B(10)	103.2 (2)	B(3)—B(8)—B(10)	60.2 (2)
B(7)—B(8)—B(10)	113.1 (2)	B(9)—B(8)—B(10)	59.6 (2)
B(5)—B(9)—B(6)	60.5 (2)	B(5)—B(9)—B(7)	113.4 (2)
B(6)—B(9)—B(7)	57.5 (2)	B(5)—B(9)—B(8)	102.7 (2)
B(6)—B(9)—B(8)	90.0 (2)	B(7)—B(9)—B(8)	56.8 (2)
B(5)—B(9)—B(10)	61.3 (2)	B(6)—B(9)—B(10)	103.3 (2)
B(7)—B(9)—B(10)	112.8 (3)	B(8)—B(9)—B(10)	60.1 (2)
B(3)—B(10)—B(4)	57.7 (2)	B(3)—B(10)—B(5)	89.8 (2)
B(4)—B(10)—B(5)	56.9 (2)	B(3)—B(10)—B(8)	59.5 (2)
B(4)—B(10)—B(8)	112.3 (3)	B(5)—B(10)—B(8)	101.5 (2)
B(3)—B(10)—B(9)	101.6 (2)	B(4)—B(10)—B(9)	111.7 (2)
B(5)—B(10)—B(9)	59.2 (2)	B(8)—B(10)—B(9)	60.3 (2)
H(10)—O—C(1)—N(1)	1 (3)	H(10)—O—C(1)—C(2)	-179 (3)
H(1N)—N(1)—C(1)—O	-177 (2)	H(1N)—N(1)—C(1)—C(2)	3 (2)
H(1N)—N(1)—B(1)—B(2)	83 (2)	H(1N)—N(1)—B(1)—B(3)	156 (2)
H(1N)—N(1)—B(1)—B(4)	-133 (2)	H(1N)—N(1)—B(1)—B(5)	-61 (2)
H(1N)—N(1)—B(1)—B(6)	14 (2)	O—C(1)—C(2)—H(2)	-173 (2)
N(1)—C(1)—C(2)—H(2)	7 (2)	C(1)—C(2)—C(3)—H(32)	-6 (3)
H(2)—C(2)—C(3)—H(31)	4 (3)	H(2)—C(2)—C(3)—H(32)	176 (3)
B(1)—N(1)—C(1)—O	-6.1 (4)	B(1)—N(1)—C(1)—C(2)	173.9 (3)
C(1)—N(1)—B(1)—B(2)	-86.6 (3)	C(1)—N(1)—B(1)—B(3)	-14.4 (5)
C(1)—N(1)—B(1)—B(4)	56.6 (4)	C(1)—N(1)—B(1)—B(5)	128.7 (3)
C(1)—N(1)—B(1)—B(6)	-156.0 (3)	O—C(1)—C(2)—C(3)	9.1 (5)
N(1)—C(1)—C(2)—C(3)	-170.9 (3)	N(1)—B(1)—B(2)—B(3)	122.6 (3)
N(1)—B(1)—B(2)—B(6)	-109.5 (3)	N(1)—B(1)—B(2)—B(7)	-134.1 (3)
N(1)—B(1)—B(2)—B(8)	167.0 (2)	B(3)—B(1)—B(2)—B(6)	127.9 (2)
B(3)—B(1)—B(2)—B(7)	103.3 (3)	B(3)—B(1)—B(2)—B(8)	44.3 (2)
B(4)—B(1)—B(2)—B(3)	-23.0 (2)	B(4)—B(1)—B(2)—B(6)	104.9 (3)
B(4)—B(1)—B(2)—B(7)	80.3 (3)	B(4)—B(1)—B(2)—B(8)	21.4 (3)
B(5)—B(1)—B(2)—B(3)	-82.9 (2)	B(5)—B(1)—B(2)—B(6)	45.0 (2)
B(5)—B(1)—B(2)—B(7)	20.4 (3)	B(5)—B(1)—B(2)—B(8)	-38.6 (3)
B(6)—B(1)—B(2)—B(3)	-127.9 (2)	B(6)—B(1)—B(2)—B(7)	-24.6 (2)
B(6)—B(1)—B(2)—B(8)	-83.6 (2)	B(9)—B(7)—B(8)—B(3)	-89.0 (3)
N(1)—B(1)—B(3)—B(2)	-100.7 (3)	N(1)—B(1)—B(3)—B(4)	104.4 (3)
N(1)—B(1)—B(3)—B(8)	-145.4 (3)	N(1)—B(1)—B(3)—B(10)	154.8 (3)
N(1)—B(1)—B(6)—B(2)	105.6 (3)	N(1)—B(1)—B(6)—B(5)	-126.4 (3)
N(1)—B(1)—B(6)—B(7)	129.9 (3)	N(1)—B(1)—B(6)—B(9)	-171.1 (2)

1.820(5) Å, respectively; the average B—B distances across the equatorial belt of the anion are 1.810(5) Å. However, these distances are all slightly shorter than those in the structure of Cu₂B₁₀H₁₀ (Dobrott & Lipscomb, 1962) and are comparable to those of [C₅H₅FeC₅H₄CH₂N(CH₃)₂]₂B₁₀H₁₀ (Zhang, Cai, Chen, Pan, Lu, Zhang & Zhu, 1982). The exopolyhedral B—N bond distance of 1.523(4) Å in (I) is slightly shorter than the sum of the single-bond covalent

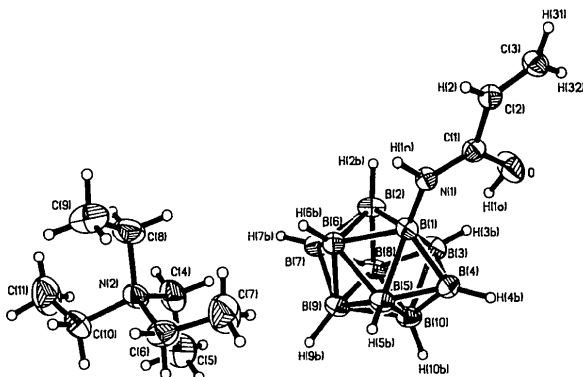


Fig. 1. ORTEP drawing of the molecule. Thermal ellipsoids scaled to enclose 30% probability. H atoms are represented as spheres of arbitrary radii.

radii of boron and nitrogen (1.58 Å), but is significantly longer than B=N (1.34 Å) and delocalized B–N (1.44 Å) distances (Niedenzu & Dawson, 1965). A high degree of double-bond character of the N–C bond [1.288 (4) Å] and planarity of the entire amide moiety [121.7 (3)°] are consistent with the microwave measurements on formamide, and have been invoked to explain a relatively high rotational barrier about the C–N bond to make the structure of amides a relatively rigid one. Furthermore, O-protonated amide, as in (I), is greatly stabilized by resonance (Streitwieser & Heathcock, 1985).

The slightly distorted tetrahedral geometry of the (C₂H₅)₄N⁺ ion with an average C–N–C bond angle of 111.3 (3)° is unexceptional and deserves no special comment.

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Platelet Activating Factor Antagonist Design: Structure of Methyl *trans*-5-(3,4-Dimethoxyphenyl)-2,3,4,5-tetrahydro-2-oxo-4-furancarboxylate

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Abstract. C₁₄H₁₆O₆, *M_r* = 280.28, monoclinic, *P*2₁/*c*, *a* = 6.070 (2), *b* = 9.526 (5), *c* = 22.418 (5) Å, β = 94.32 (2)°, *V* = 1293 Å³, *Z* = 4, *D_x* = 1.44 g cm⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 0.71 cm⁻¹, *F*(000) = 592,

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T = 293 K, final *R* = 0.043 for 1400 observed [*F_o* ≥ 5σ(*F_o*)] reflections. The observed structure confirms a *trans* stereorelationship for the two substituents and an envelope conformation for the lactone ring. There is no crystallographically imposed symmetry. An analysis of the closest contacts in the cell lattice reveals two types of intermolecular interactions for this compound.