Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($Å^2$) *for* (5)

$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
C1	0.0811 (2)	−0.3988 (8)	0.264 (1)	0.052 (1)
C2	0.0609 (2)	−0.5401 (8)	0.349 (1)	0.062 (2)
C3	0.0288 (2)	−0.6143 (9)	0.225 (1)	0.070 (2)
C4	0.0183 (2)	−0.5470 (9)	0.020 (1)	0.068 (2)
C5	0.0384 (2)	−0.4059 (8)	−0.073 (1)	0.054 (1)
C6	0.0701 (1)	−0.3299 (7)	0.050 (1)	0.042 (1)
C7	0.0909 (1)	−0.1767 (7)	−0.0436 (9)	0.042 (1)
O8	0.0748 (1)	−0.1151 (5)	−0.2399 (7)	0.059 (1)
C9	0.0970 (2)	0.0413 (8)	−0.294 (1)	0.061 (2)
C10	0.1318 (2)	0.0448 (7)	−0.113 (1)	0.050 (1)
N11	0.1215 (1)	−0.1000 (6)	0.0418 (8)	0.048 (1)
C12	0.1347 (2)	0.2139 (8)	0.020 (1)	0.058 (2)
S13	0.1595 (1)	0.3806 (2)	−0.1489 (4)	0.0641 (6)
O14	0.1461 (1)	0.3538 (7)	−0.3908 (9)	0.086 (2)
C15	0.2112 (1)	0.3036 (6)	−0.1279 (9)	0.043 (1)
C16	0.2332 (2)	0.3419 (7)	0.066 (1)	0.047 (1)
C17	0.2742 (2)	0.2896 (7)	0.083 (1)	0.046 (1)
C18	0.2923 (1)	0.2032 (7)	−0.1007 (9)	0.042 (1)
C19	0.2699 (2)	0.1647 (7)	−0.297 (1)	0.049 (1)
C20	0.2290 (2)	0.2165 (7)	−0.307 (1)	0.051 (1)
O21	0.3323 (1)	0.1572 (7)	−0.0749 (7)	0.060 (1)

The crystals were not of high quality, especially those of compound (2) as evidenced by the high R_{int} value. This is not unexpected in crystals of structures with long hydrophobic chains of atoms. Data for compounds (2) and (3) were collected using $\theta/2\theta$ scans. The scan width was $(1.0 + 0.14\tan\theta)°$, with a θ-scan rate of 0.8° $\min^{-1}$ and background counts for 5 s on each side of every scan. Data for compound (5) was collected using a $\theta/2\theta$ scan mode, with a scan width of 1.8°, a scan rate of 1.0° $\min^{-1}$ and background counts for 20 s on each side of every scan. Scattering factors, dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C, Tables 6.1.1.4, 4.2.6.8 and 4.2.4.2, respectively). The absolute configuration at S13 has been assigned to agree with the known chirality at C10. Confirmation of the assignments of the C10 configuration of the investigated crystals was established as described by Flack (1983) and Flack & Schwarzenbach (1988). The relatively high *wR* values are due to crystal quality and to the fact that *wR* values are calculated on F^2 using all data.

Data collection: Siemens *P*3 software for (2) and (3); Picker software for (5). Cell refinement: Siemens *P*3 software for (2) and (3); Picker software for (5). Data reduction: Siemens *P*3 software for (2) and (3); Picker software for (5). For all compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ZORTEP* (Zsolnai, 1995); software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SX1014). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Methyl (1*R*,2*R*)- and (1*S*,2*S*)-1-Cyano-2-[(*S*)-2,2-dimethyl-1,3-dioxolan-4-yl]cyclopropane-1-carboxylate

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Abstract

In the title diastereomeric compounds, $C_{11}H_{15}NO_4$, the methyl ester group adopts a bisecting conformation with the carbonyl O atom eclipsing the cyclopropane ring. In the crystals of both compounds, the molecules are piled up along one of the crystallographic axes.

Comment

Cyclopropane amino acids are particularly interesting because they constitute a unique form of 'conformationally constrained' amino acid which has been found in nature, generally in the unbound form or, in some cases, as a constituent of small peptides. When introduced into a peptide chain, the peculiar nature of this kind of amino acid residue is expected to cause profound changes in the proximal peptide conformation, which may affect the ability of the peptide to fit an enzyme active site and/or its intended bioreceptor.

Our interest in the asymmetric synthesis of cyclopropane amino acids (Cativiela, Díaz-de-Villegas & Jiménez, 1994, 1995*a*,*b*,*c*, 1996) has prompted us to use a chiral cyano ester as a synthetic precur-

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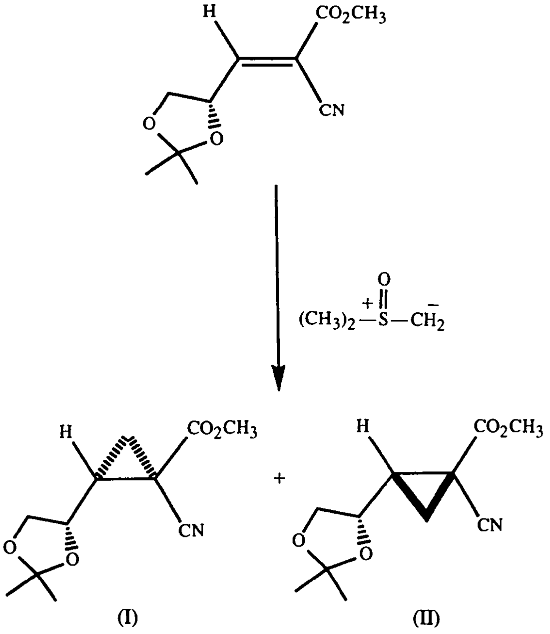
sor of these compounds and we have tested the reaction of methylides with methyl (*E*)-2-cyano-3-[(*S*)-2,2-dimethyl-1,3-dioxolan-4-yl]acrylate derived from D-glyceraldehyde. When methyl (*E*)-2-cyano-3-[(*S*)-2,2-dimethyl-1,3-dioxolan-4-yl]acrylate was treated with Corey ylide, *i.e.* dimethyloxosulfonium methylide, at 223 K until completion, a mixture of two cyclopropane cyano esters, (I) and (II), was obtained in the ratio 8:2, and X-ray analyses of these compounds were carried out in order to determine unambiguously the absolute configuration at the two new chiral C atoms formed in the methylene-insertion reaction.



The molecular structures of (I) and (II) are shown in Figs. 1 and 2, respectively. The X-ray analyses established the stereochemistries at the two new chiral C1 and C2 atoms as *R* and *R* for (I), and *S* and *S* for (II). The absolute configuration is deduced from the known stereochemistry of the chiral centre at C7, which originated from (*R*)-isopropylideneglyceraldehyde. These results indicate that the addition of ylide had occurred preferably, with total stereoselectivity, to the 1*Si*–2*Re* diastereotopic face of the double bond.

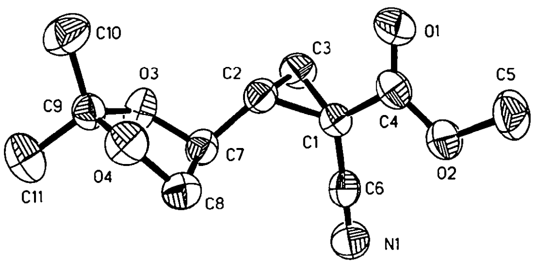


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

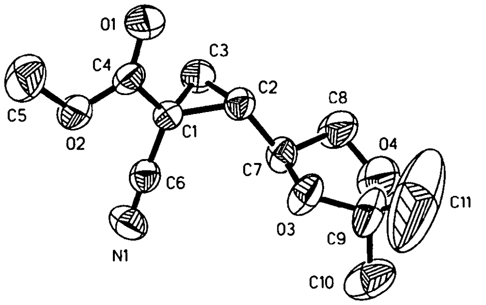


Fig. 2. The molecular structure of (II) shpwing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

In compounds (I) and (II), the C6—C1—C2—C7 torsion angle is slightly twisted by 6.7 (3) and −1.4 (7)°, respectively, from the theoretical value (0°) for a *cis*-disubstituted cyclopropane. The methyl ester groups, which have the C5—O2—C4—C1 sequence in a *trans* disposition [−179.5 (2) for (I) and −179.0 (4)° for (II)], adopt a bisecting conformation with the carbonyl O atom eclipsing the cyclopropane ring [torsion angles *M*1—C1—C4—O1 are −6.1 (4) for (I) and −1.7 (8)° for (II), where *M*1 is the midpoint of the C2—C3 bond]. This conformation allows optimum conjugation between the C═O moiety of the methyl ester group and the cyclopropane ring.

For compound (I), the dioxolane ring adopts a major half-chair conformation [puckering parameters (Cremer & Pople, 1975; Nardelli, 1983) are Q = 0.346 (2) Å and φ = 49.5 (4)°], while for compound (II), the dioxolane ring exhibits an envelope conformation [puckering parameters are Q = 0.335 (7) Å and φ = −145.9 (10)°], with the C8 atom deviating by 0.527 (8)° from the plane through the other four atoms. The spatial arrangement of the dioxolane ring relative to the cyclopropane ring is defined by torsion angles C3—C2—C7—C8 [160.0 (2)°] and C1—C2—C7—O3 [−158.0 (2)°] for (I) and by torsion angles C1—C2—C7—C8 [153.5 (5)°] and C3—C2—C7—O3 [−159.9 (4)°] for (II), so in both cases, the C7—C8 and C7—O3 bonds of the dioxolane ring adopt an antiperiplanar conformation with respect to the C1—C2 and C3—C2 bonds of the cyclopropane ring.

In the crystals of both cyclopropane derivatives, the molecules are piled up along one of the crystallographic axes [*b* for (I) and *a* for (II)]. The methyl C11 atom in (II) is probably disordered since it has a physically unreasonable U_{11} value of 0.76 (5) Å^2.

Experimental

The synthesis of the title cyclopropane cyano esters was carried out under an atmosphere of argon by reaction of methyl (*E*)-2-cyano-3-[(*S*)-2,2-dimethyl-1,3-dioxolan-4-yl]acrylate and freshly prepared NaH-derived dimethyloxosulfonium methylide (molar ratio 1:1.2) in dry dichloromethane (8 ml) for

15 min at room temperature. The products, (I) and (II) in a ratio of 4:1, were isolated from the reaction mixture by column chromatography [eluent: hexane–ethyl acetate (4:1)]. Crystals were obtained by slow evaporation from a hexane–ether (1:1) solution.

Compound (I)

Crystal data

$C_{11}H_{15}NO_4$
$M_r = 225.24$
Orthorhombic
$P2_12_12_1$
$a = 22.370\,(3)$ Å
$b = 5.6920\,(10)$ Å
$c = 9.390\,(2)$ Å
$V = 1195.6\,(4)$ Å^3
$Z = 4$
$D_x = 1.251$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 28 reflections
$\theta = 5.01–12.47°$
$\mu = 0.096$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.72 × 0.38 × 0.26 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1776 measured reflections
1591 independent reflections
1398 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0262$

$\theta_{\rm max} = 24.99°$
$h = -1 \rightarrow 26$
$k = -1 \rightarrow 6$
$l = -1 \rightarrow 11$
3 standard reflections monitored every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0424$
$wR(F^2) = 0.1156$
$S = 1.036$
1590 reflections
146 parameters
H atoms: only overall U = 0.085 (3) Å^2 refined
$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.1649P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.041$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (I)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$
O1	0.94164 (8)	−0.2494 (4)	−0.0883 (2)	0.0622 (6)
O2	0.98038 (8)	0.0666 (4)	0.0243 (2)	0.0534 (5)
O3	0.75558 (7)	−0.3428 (3)	0.2465 (2)	0.0545 (5)
O4	0.72605 (8)	−0.0386 (3)	0.1051 (2)	0.0571 (5)
N1	0.94093 (10)	0.0868 (5)	0.3718 (2)	0.0599 (6)
C1	0.91567 (10)	−0.1752 (4)	0.1535 (2)	0.0405 (5)
C2	0.85095 (10)	−0.2652 (5)	0.1399 (3)	0.0449 (6)
C3	0.89852 (12)	−0.4306 (5)	0.1833 (3)	0.0532 (7)
C4	0.94746 (10)	−0.1273 (5)	0.0154 (3)	0.0459 (6)
C5	1.01271 (13)	0.1302 (7)	−0.1046 (3)	0.0678 (9)
C6	0.93044 (10)	−0.0281 (5)	0.2750 (3)	0.0445 (6)
C7	0.80368 (10)	−0.1777 (5)	0.2422 (3)	0.0458 (6)
C8	0.77333 (12)	0.0488 (5)	0.1930 (3)	0.0566 (7)
C9	0.70467 (10)	−0.2473 (5)	0.1729 (3)	0.0460 (6)
C10	0.68533 (15)	−0.4153 (7)	0.0587 (3)	0.0703 (9)
C11	0.65540 (15)	−0.1940 (8)	0.2801 (4)	0.0875 (11)

Table 2. *Geometric parameters (Å, °) for (I)*

O1—C4	1.203 (3)	C1—C4	1.503 (3)
O2—C4	1.329 (4)	C1—C3	1.529 (4)
O2—C5	1.456 (3)	C1—C2	1.541 (3)
O3—C7	1.429 (3)	C2—C3	1.478 (4)
O3—C9	1.439 (3)	C2—C7	1.513 (3)
O4—C9	1.430 (3)	C7—C8	1.529 (4)
O4—C8	1.431 (3)	C9—C10	1.501 (4)
N1—C6	1.144 (3)	C9—C11	1.523 (4)
C1—C6	1.453 (3)		
C4—O2—C5	115.4 (2)	O1—C4—C1	122.8 (3)
C7—O3—C9	109.5 (2)	O2—C4—C1	111.0 (2)
C9—O4—C8	106.2 (2)	N1—C6—C1	178.6 (3)
C6—C1—C4	117.7 (2)	O3—C7—C2	109.1 (2)
C6—C1—C3	117.4 (2)	O3—C7—C8	103.2 (2)
C4—C1—C3	116.7 (2)	C2—C7—C8	113.3 (2)
C6—C1—C2	118.0 (2)	O4—C8—C7	102.1 (2)
C4—C1—C2	115.7 (2)	O4—C9—O3	105.3 (2)
C3—C1—C2	57.6 (2)	O4—C9—C10	107.9 (2)
C3—C2—C7	122.5 (2)	O3—C9—C10	109.3 (2)
C3—C2—C1	60.8 (2)	O4—C9—C11	111.8 (3)
C7—C2—C1	119.6 (2)	O3—C9—C11	109.3 (2)
C2—C3—C1	61.6 (2)	C10—C9—C11	113.0 (3)
O1—C4—O2	126.2 (2)		
C6—C1—C2—C7	6.7 (3)	C1—C2—C7—O3	−158.0 (2)
C5—O2—C4—C1	−179.5 (2)	C3—C2—C7—C8	160.0 (2)

Compound (II)

Crystal data

$C_{11}H_{15}NO_4$
$M_r = 225.24$
Orthorhombic
$P2_12_12_1$
$a = 5.9110\,(10)$ Å
$b = 9.445\,(2)$ Å
$c = 22.051\,(4)$ Å
$V = 1231.1\,(4)$ Å^3
$Z = 4$
$D_x = 1.215$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 26 reflections
$\theta = 4.66–12.30°$
$\mu = 0.093$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.70 × 0.30 × 0.22 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1800 measured reflections
1627 independent reflections
1072 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0303$

$\theta_{\rm max} = 24.99°$
$h = -1 \rightarrow 7$
$k = -1 \rightarrow 11$
$l = -1 \rightarrow 26$
3 standard reflections monitored every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0675$
$wR(F^2) = 0.1897$
$S = 1.021$
1627 reflections
146 parameters
H atoms: only overall U = 0.123 (7) Å^2 refined
$w = 1/[\sigma^2(F_o^2) + (0.0892P)^2 + 0.8493P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.022$
$\Delta\rho_{\rm max} = 0.40$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.38$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (II)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.7117 (7)	1.0866 (4)	0.4420 (2)	0.0676 (12)
O2	1.0278 (6)	0.9766 (3)	0.4734 (2)	0.0552 (10)
O3	0.8244 (7)	0.7776 (4)	0.26805 (15)	0.0691 (12)
O4	0.5320 (10)	0.6655 (8)	0.2186 (2)	0.129 (2)
N1	1.0423 (9)	0.6325 (5)	0.4296 (2)	0.0671 (14)
C1	0.7681 (8)	0.8440 (5)	0.4168 (2)	0.0424 (11)
C2	0.6269 (9)	0.8505 (6)	0.3586 (2)	0.0500 (13)
C3	0.5161 (9)	0.8096 (6)	0.4163 (2)	0.0592 (14)
C4	0.8305 (9)	0.9824 (5)	0.4453 (2)	0.0466 (12)
C5	1.1037 (12)	1.1053 (6)	0.5037 (3)	0.079 (2)
C6	0.9234 (9)	0.7275 (6)	0.4245 (2)	0.0485 (12)
C7	0.6540 (9)	0.7401 (5)	0.3102 (2)	0.0520 (14)
C8	0.4418 (12)	0.7214 (9)	0.2722 (3)	0.091 (2)
C9	0.7581 (16)	0.7300 (8)	0.2093 (3)	0.086 (2)
C10	0.8931 (14)	0.6037 (8)	0.1906 (4)	0.099 (3)
C11	0.7640 (4)	0.8389 (10)	0.1674 (4)	0.317 (15)

Table 4. *Geometric parameters (Å, °) for (II)*

O1—C4	1.211 (6)	C1—C4	1.496 (7)
O2—C4	1.322 (6)	C1—C3	1.525 (7)
O2—C5	1.457 (6)	C1—C2	1.534 (7)
O3—C7	1.416 (6)	C2—C3	1.483 (7)
O3—C9	1.426 (7)	C2—C7	1.500 (7)
O4—C8	1.401 (7)	C7—C8	1.518 (8)
O4—C9	1.483 (10)	C9—C11	1.382 (11)
N1—C6	1.146 (6)	C9—C10	1.494 (10)
C1—C6	1.443 (7)		
C4—O2—C5	116.9 (4)	O1—C4—C1	122.8 (5)
C7—O3—C9	108.8 (5)	O2—C4—C1	112.2 (4)
C8—O4—C9	107.8 (5)	N1—C6—C1	177.9 (5)
C6—C1—C4	117.4 (4)	O3—C7—C2	111.6 (4)
C6—C1—C3	117.4 (4)	O3—C7—C8	104.8 (4)
C4—C1—C3	115.5 (4)	C2—C7—C8	112.6 (5)
C6—C1—C2	118.4 (4)	O4—C8—C7	101.3 (5)
C4—C1—C2	116.8 (4)	C11—C9—O3	111.4 (7)
C3—C1—C2	58.0 (3)	C11—C9—O4	114.9 (11)
C3—C2—C7	118.5 (5)	O3—C9—O4	104.6 (5)
C3—C2—C1	60.7 (3)	C11—C9—C10	113.4 (9)
C7—C2—C1	120.6 (4)	O3—C9—C10	110.8 (6)
C2—C3—C1	61.3 (3)	O4—C9—C10	101.0 (6)
O1—C4—O2	125.0 (5)		
C6—C1—C2—C7	−1.4 (7)	C3—C2—C7—O3	−159.9 (4)
C5—O2—C4—C1	−179.0 (4)	C1—C2—C7—C8	153.5 (5)

For both compounds, data collection: *XSCANS* (Siemens, 1993); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1989); software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1282). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Strained Tricyclic Product of a Dipolar 1,3-Cycloaddition Reaction of Diazepine

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Abstract

In the title compound, diethyl 1,1′-bis(*p*-chlorophenyl)-6,7a-dimethyl-4,7a,1′,4′,5′,6′-hexahydropyrrolo[4,5-*a*]-[1,2,4]triazole-5-spiro-6′-[1,2,4]triazine-3,3′-dicarboxylate, $C_{27}H_{28}Cl_2N_6O_4$, the tricyclic system is composed of two five-membered rings sharing one side and one six-membered ring having one C atom in common with one of the five-membered rings. Strain in this molecule is reflected by significant variations in the bond distances and bond angles.

Comment

We report here the dipolar 1,3-cycloaddition reaction of diazepine, (1), [which can exist in the tautomeric forms (1*A*) and (1*B*)] with the *N*-aryl-*C*-ethoxycarbonyl