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## Structures of Diels–Alder Reaction Bis-Adducts of 4-Phenyl- or 4-Methyl-1,2,4-triazoline-3,5-dione with 1,6-Methano[10]annulene

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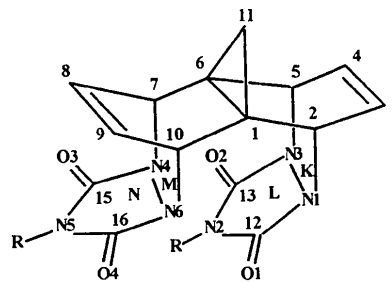
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**Abstract.** 2,9-Dimethyl-1,2,3,5,5a,6,8,9,10,12,12a,13-dodecahydro-5,13;6,12-bisetheno-5a,12a-methano[1,2,4]triazolo[1',2':1,2]pyridazino[4,5-d][1,2,4]triazolo[1,2-a]pyridazine-1,3,8,10-tetraone (1),  $C_{17}H_{16}N_6O_4$ ,  $M_r = 368.35$ , triclinic,  $P\bar{1}$ ,  $a = 8.756$  (4),  $b = 12.373$  (6),  $c = 8.426$  (5) Å,  $\alpha = 102.99$  (3),  $\beta = 115.79$  (3),  $\gamma = 98.06$  (3)°,  $V = 770.86$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.588$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.110$  cm<sup>-1</sup>,  $F(000) = 384$ ,  $T = 298$  K,  $R = 0.047$ , for 2670 reflections. 2,9-Diphenyl-1,2,3,5,5a,6,8,9,10,12,12a,13-dodecahydro-5,13;6,12-bisetheno-5a,12a-methano[1,2,4]triazolo[1',2':1,2]pyridazino[4,5-d][1,2,4]triazolo[1,2-a]pyridazine-1,3,8,10-tetraone (2),  $C_{27}H_{20}N_6O_4$ ,  $M_r = 492.50$ , monoclinic,  $P2_1/c$ ,  $a = 14.440$  (7),  $b = 9.416$  (5),  $c = 16.227$  (9) Å,  $\beta = 98.42$  (3)°,  $V = 2182.55$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.499$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.098$  cm<sup>-1</sup>,  $F(000) = 1024$ ,  $T = 298$  K,  $R = 0.072$  for 2344 reflections. 2,9-Diphenyl-1,2,3,5,5a,6,8,9,10,12,12a,13-dodecahydro-5,13;6,12-bisetheno-5a,12a-methano[1,2,4]triazolo[1',2':1,2]pyridazino[4,5-d][1,2,4]triazolo[1,2-a]pyridazine-1,3,8,10-tetraone 0.5-ethanol solvate (3),  $C_{27}H_{20}N_6O_4 \cdot \frac{1}{2}C_2H_6O$ ,  $M_r = 515.53$ , triclinic,  $P\bar{1}$ ,  $a = 12.673$  (6),  $b = 11.431$  (6),  $c = 9.439$  (5) Å,  $\alpha = 68.49$  (3),  $\beta = 111.39$  (3),  $\gamma = 97.72$  (3)°,  $V = 1184.52$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.446$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.097$  cm<sup>-1</sup>,  $F(000) = 538$ ,  $T = 298$  K,  $R = 0.065$  for 2834 reflections. Crystal structures of two Diels–Alder bis-adducts of 4-phenyl- and 4-methyl-1,2,4-triazoline-3,5-dione with 1,6-methano[10]annulene show that the molecules adopt the *exo-endo* configuration. The phenyl derivative crystallizes with or without a solvent molecule.

**Introduction.** Many Diels–Alder reactions between propellanes containing one or two cyclohexadiene rings and bridged [10]annulenes with triazolinediones show a preference for the *endo* mono-adducts (Kalo, Vogel & Ginsburg, 1977; Kalo, Bloomfield & Ginsburg, 1978; Kalo & Ginsburg, 1978; Ashkenazi, Kalo, Ruttimann & Ginsburg, 1978; Ashkenazi, Vogel & Ginsburg, 1977, 1978; Ashkenazi, Peled, Vogel & Ginsburg, 1979; Gleiter & Ginsburg, 1979). Only one mono-adduct of both the *exo* and the *endo* were detected in 11-cyano-1,6-methano[10]annulene, while its bis-adduct afforded the *endo-endo* derivative (Ashkenazi, Kaftory, Arad, Apeloig & Ginsburg, 1981) whose crystal structure has been published (Kaftory, 1983; Kaftory & Agmon, 1984).

1,6-Methano[10]annulene itself affords the *endo-exo* bis-adduct (1). We describe here the crystal structure of three compounds: (1) the *endo-exo* bis-adduct of 1,6-methano[10]annulene with the methyl derivative of the triazolinedione; (2) the solvent free *endo-exo* bis-adduct with the phenyl derivative of triazolinedione and (3) the same as (2) with a molecule of ethanol.



(1)  $R = \text{Me}$   
(2), (3)  $R = \text{Ph}$

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Table 1. Crystallographic and experimental details

	(1)	(2)	(3)
Crystal shape	Prism	Plate	Needle
Crystal size (mm)	0.4 × 0.3 × 0.3	0.5 × 0.3 × 0.1	0.5 × 0.2 × 0.2
Scan width Δω (°)	1.4	1.0	1.2
Scan time (s)	28	20	24
Background time (s)	20	20	24
θ <sub>min</sub> -θ <sub>max</sub> (°)	2.5-25	2.5-23	2.5-24
hkl range h	-9 → 9	-15 → 15	-13 → 13
k	-14 → 14	-0 → 10	-11 → 12
l	0 → 10	0 → 17	0 → 10
No. of reflections measured	2707	3034	3713
No. of unique reflections	2670	2906	3589
No. of significant reflections*	2670	2344	2834
Weighting coefficients† k	0.2637	0.3441	1.4629
g	0.0116	0.0316	0.0006
No. of refined parameters	442	308	416
(Δσ) <sub>max</sub>	0.7	0.8	0.9
(Δρ) <sub>max</sub> (e Å <sup>-3</sup> )	0.2	0.3	0.3
(Δρ) <sub>min</sub> (e Å <sup>-3</sup> )	-0.2	-0.3	-0.3
wR	0.065	0.098	0.063
R	0.047	0.072	0.065

\* Criterion: (1)  $F_o > 0.0$ ; (2)  $F_o > 1.5\sigma(F_o)$ ; (3)  $F_o > 1.5\sigma(F_o)$ .

†  $w = k/[\sigma^2(F_o) + gF_o^2]$ .

Table 2. Positional parameters ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^4$ ) for (1)

	x	y	z	$U_{eq}$
O(1)	-1382 (2)	4653 (1)	1996 (2)	318 (4)
O(2)	-2570 (2)	993 (1)	-1834 (2)	331 (3)
O(3)	2056 (2)	1501 (1)	101 (2)	353 (4)
O(4)	3158 (2)	5045 (1)	4159 (2)	295 (3)
N(1)	-2346 (2)	2823 (1)	2061 (2)	236 (3)
N(2)	-2031 (2)	2947 (1)	-327 (2)	250 (4)
N(3)	-2725 (2)	1697 (1)	874 (2)	243 (3)
N(4)	1047 (2)	2123 (1)	2181 (2)	234 (3)
N(5)	2808 (2)	3417 (1)	1858 (2)	258 (3)
N(6)	1402 (2)	3234 (1)	3441 (2)	224 (3)
C(1)	-202 (2)	2420 (1)	4770 (2)	217 (4)
C(2)	-1850 (2)	2872 (2)	4007 (2)	237 (4)
C(3)	-3417 (3)	2012 (2)	3755 (3)	300 (4)
C(4)	-3734 (3)	953 (2)	2674 (3)	308 (4)
C(5)	-2479 (3)	838 (2)	1882 (3)	253 (4)
C(6)	-554 (2)	1223 (2)	3470 (3)	234 (4)
C(7)	959 (3)	1180 (2)	3023 (3)	247 (4)
C(8)	2710 (3)	1541 (2)	4792 (3)	304 (4)
C(9)	3031 (3)	2565 (2)	5951 (3)	297 (4)
C(10)	1590 (2)	3168 (2)	5264 (2)	230 (4)
C(11)	-234 (3)	1385 (2)	5433 (3)	264 (4)
C(12)	-1834 (2)	3606 (2)	1360 (3)	240 (4)
C(13)	-2422 (2)	1778 (2)	-554 (2)	236 (4)
C(14)	-1535 (4)	3422 (2)	-1512 (3)	363 (6)
C(15)	2006 (2)	2250 (2)	1266 (3)	251 (4)
C(16)	2563 (2)	4024 (2)	3292 (2)	223 (4)
C(17)	3849 (3)	3936 (2)	1133 (4)	362 (5)

Table 3. Positional parameters ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^4$ ) for (2)

	x	y	z	$U_{eq}$
O(1)	7645 (2)	-1457 (4)	2876 (2)	380 (9)
O(2)	5439 (2)	1039 (4)	4083 (2)	441 (8)
O(3)	7054 (3)	4048 (4)	4735 (2)	513 (10)
O(4)	9357 (2)	1610 (4)	3671 (2)	420 (9)
N(1)	6395 (2)	-52 (4)	2360 (2)	267 (8)
N(2)	6599 (2)	-484 (4)	3702 (2)	282 (9)
N(3)	5704 (2)	701 (4)	2725 (2)	292 (8)
N(4)	7065 (2)	2799 (4)	3507 (2)	301 (8)
N(5)	8340 (3)	2713 (4)	4463 (2)	311 (8)
N(6)	7793 (2)	2030 (4)	3168 (2)	248 (8)
C(1)	6917 (3)	2169 (5)	1784 (3)	290 (10)
C(2)	6605 (3)	624 (5)	1579 (2)	280 (10)
C(3)	5665 (4)	618 (5)	1033 (3)	379 (12)
C(4)	5000 (4)	1284 (6)	1371 (3)	429 (12)
C(5)	5325 (3)	1944 (5)	2213 (3)	363 (11)
C(6)	6149 (3)	2974 (5)	2132 (3)	289 (11)
C(7)	6587 (3)	3852 (5)	2893 (3)	343 (12)
C(8)	7365 (4)	4782 (5)	2673 (3)	403 (13)
C(9)	8020 (4)	4076 (5)	2355 (3)	368 (13)
C(10)	7864 (3)	2503 (5)	2289 (3)	270 (10)
C(11)	6383 (4)	3341 (5)	1277 (3)	381 (12)
C(12)	6974 (3)	-729 (4)	2965 (2)	278 (8)
C(13)	5862 (3)	514 (5)	3574 (3)	299 (10)
C(14)	6972 (3)	-1076 (4)	4496 (2)	252 (8)
C(15)	7440 (3)	3296 (5)	4290 (3)	342 (12)
C(16)	8594 (3)	2080 (4)	3754 (3)	280 (8)
C(17)	8967 (3)	2940 (5)	5223 (3)	341 (10)
C(18)	6382 (3)	-1465 (4)	5048 (3)	279 (8)
C(19)	6773 (3)	-2062 (5)	5814 (3)	334 (10)
C(20)	7734 (3)	-2254 (5)	6004 (3)	366 (12)
C(21)	8301 (3)	-1854 (5)	5441 (3)	393 (12)
C(22)	7942 (3)	-1239 (5)	4691 (3)	333 (11)
C(23)	9104 (4)	4300 (5)	5546 (3)	421 (13)
C(24)	9711 (4)	4482 (6)	6271 (3)	501 (14)
C(25)	10185 (4)	3342 (6)	6675 (3)	498 (15)
C(26)	10040 (3)	2002 (6)	6345 (3)	462 (14)
C(27)	9438 (3)	1783 (5)	5621 (3)	348 (11)

**Experimental.** The bis-adducts were obtained according to procedures described in the references cited in the *Introduction*. The crystals were obtained by recrystallization from ethanol. Crystallographic data, details of intensity measurements and structure refinement are given in Table 1. Data were collected using a Philips PW 1100 diffractometer with graphite-monochromated Mo  $K\alpha$  radiation and  $\theta/2\theta$  scans. Lattice parameters were calculated using 25 reflections for each compound. Three reflections (for each compound) were monitored and showed a variation in intensities of ca 5%. Absorption and extinction corrections were not applied. The structures were solved using *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977) and refined by full-matrix least squares (based on  $F$ ) by *SHELX76* (Sheldrick, 1976) with anisotropic atomic displacement parameters for O, N and C atoms, isotropic for H. The atomic scattering factors were taken from Cromer & Mann (1968), and from Stewart, Davidson & Simpson (1965). All H-atom positions were located from difference Fourier maps.

**Discussion.** Final positional parameters for (1), (2) and (3) are given in Tables 1, 2 and 3,\* respectively (atomic notation is given in the scheme above). Molecular stereoviews for the molecules (1) and (2) are shown in Figs. 1 and 2, respectively. Bond lengths and angles are given in Table 5.

The major differences in bond lengths and angles are observed in the two triazolinedione moieties within each of the molecules (the *endo* and the *exo*).

\* Lists of structure factors, atomic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55801 (51 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1019]

All the differences are within the expected values obtained by mapping the simultaneous double  $N$ -inversion (Kaftory & Agmon, 1984). On the *endo* side the angle between planes  $M$ ,  $N$  is smaller

Table 4. Positional parameters ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^4$ ) for (3)

$U_{eq} = (1/3) \text{ trace } U.$				
	x	y	z	$U_{eq}$
O(1)	5068 (2)	4274 (2)	7649 (3)	332 (8)
O(2)	8673 (2)	4420 (3)	11262 (3)	422 (9)
O(3)	9394 (2)	1711 (3)	10359 (3)	431 (8)
O(4)	5803 (2)	1767 (2)	6714 (3)	424 (8)
N(1)	6618 (2)	5580 (3)	7486 (3)	277 (8)
N(2)	6755 (2)	4131 (3)	9852 (3)	276 (8)
N(3)	7722 (2)	5611 (3)	8597 (3)	289 (8)
N(4)	8234 (2)	3319 (3)	8283 (3)	264 (8)
N(5)	7531 (2)	1375 (3)	8861 (4)	305 (8)
N(6)	7116 (2)	3337 (3)	7129 (3)	263 (8)
C(1)	7369 (3)	5461 (3)	5499 (4)	294 (8)
C(2)	6550 (3)	6168 (3)	5771 (4)	314 (8)
C(3)	7010 (4)	7487 (4)	5676 (5)	385 (11)
C(4)	8041 (4)	7501 (4)	6706 (5)	400 (11)
C(5)	8557 (3)	6209 (3)	7780 (4)	324 (8)
C(6)	8555 (3)	5459 (3)	6722 (4)	298 (8)
C(7)	9037 (3)	4121 (4)	7498 (5)	318 (8)
C(8)	8994 (3)	3603 (4)	6220 (5)	390 (12)
C(9)	7973 (4)	3609 (4)	5153 (5)	390 (11)
C(10)	7067 (3)	4125 (4)	5446 (4)	311 (8)
C(11)	8321 (3)	6136 (4)	4952 (5)	367 (8)
C(12)	6022 (3)	4615 (3)	8234 (4)	274 (8)
C(13)	7836 (3)	4679 (3)	10051 (4)	300 (8)
C(14)	6476 (3)	3115 (3)	11106 (4)	280 (8)
C(15)	8500 (3)	2081 (3)	9272 (4)	307 (8)
C(16)	6715 (3)	2111 (3)	7457 (4)	302 (8)
C(17)	7415 (3)	58 (3)	9634 (4)	329 (8)
C(18)	7191 (3)	2095 (4)	12029 (5)	372 (10)
C(19)	6925 (4)	1132 (4)	13231 (5)	462 (12)
C(20)	5939 (4)	1168 (4)	13520 (5)	484 (13)
C(21)	5227 (4)	2182 (4)	12596 (6)	469 (12)
C(22)	5502 (3)	3167 (4)	11389 (5)	371 (10)
C(23)	8134 (4)	-780 (4)	9710 (5)	425 (12)
C(24)	7982 (4)	-2061 (4)	10450 (6)	545 (14)
C(25)	7123 (5)	-2480 (4)	11080 (6)	550 (15)
C(26)	6432 (4)	-1633 (4)	11009 (5)	518 (12)
C(27)	6558 (3)	-350 (4)	10292 (5)	431 (12)
C(E)	112 (7)	474 (6)	5406 (10)	982 (25)
O(E)	-410 (8)	239 (9)	6579 (14)	1090 (33)

[141.8 (2), 133.8 (4) and 136.9 (4) $^\circ$  in (1), (2) and (3), respectively] than on the *exo* side (*K*, *L*) [156.1 (2), 154.6 (4) and 158.2 (4) $^\circ$ ]. As a result, the N—N bond lengths on the *endo* side are longer [1.432 (2), 1.450 (4) and 1.435 (3)  $\text{\AA}$  in (1), (2) and (3), respectively] than on the *exo* side [1.416 (2), 1.422 (4) and 1.415 (3)  $\text{\AA}$ ].

The most striking difference is the non-planarity at N(2) of compound (1) [when it is bonded to a methyl group, the sum of the valence angles at N(2) is 358.9 (4) $^\circ$ ]. Kaftory & Agmon (1984), using geometrical data from similar compounds, pointed out that on flattening at N(1)—N(3) there is an increase in conjugation with the neighbouring carbonyls [reflected by the shortening of N(1)—C(12) and N(3)—C(13) compared with N(4)—C(15) and N(6)—C(16)] at the expense of a decrease in the conjugation of N(2), which can be regained for compounds like (2) and (3) which have a bonded phenyl ring [in these cases N(2) is planar] and not a methyl.

Compound (3) crystallizes with a molecule of ethanol which is found to be disordered about an inversion centre, forming a weak hydrogen bond (2.883  $\text{\AA}$ ) between the ethanol O atom O(E) and atom O(3) of the bis-adduct.

Table 5. Selected bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) for compounds (1), (2) and (3)

	(1)	(2)	(3)
O(1)—C(12)	1.217 (2)	1.213 (5)	1.210 (4)
O(2)—C(13)	1.223 (2)	1.203 (5)	1.213 (3)
O(3)—C(15)	1.210 (2)	1.204 (6)	1.215 (3)
O(4)—C(16)	1.213 (2)	1.213 (5)	1.213 (3)
N(1)—N(3)	1.416 (2)	1.422 (4)	1.415 (3)
N(1)—C(2)	1.486 (2)	1.490 (5)	1.482 (4)
N(1)—C(12)	1.361 (2)	1.352 (4)	1.370 (4)
N(2)—C(12)	1.394 (2)	1.403 (5)	1.409 (3)
N(2)—C(13)	1.387 (2)	1.412 (5)	1.405 (4)
N(2)—C(14)	1.450 (3)	1.434 (4)	1.436 (4)
N(3)—C(5)	1.486 (2)	1.493 (5)	1.481 (4)
N(3)—C(13)	1.361 (3)	1.374 (5)	1.365 (3)
N(4)—N(6)	1.432 (2)	1.450 (4)	1.435 (3)
N(4)—C(7)	1.505 (2)	1.500 (5)	1.492 (4)
N(4)—C(15)	1.380 (3)	1.388 (5)	1.384 (4)
N(5)—C(15)	1.383 (2)	1.400 (6)	1.388 (4)
N(5)—C(16)	1.390 (2)	1.393 (6)	1.396 (3)
N(5)—C(17)	1.454 (3)	1.435 (5)	1.426 (4)
N(6)—C(10)	1.495 (2)	1.511 (5)	1.494 (4)
N(6)—C(16)	1.375 (2)	1.387 (5)	1.386 (4)
C(1)—C(2)	1.545 (2)	1.544 (6)	1.540 (6)
C(1)—C(6)	1.529 (2)	1.519 (6)	1.525 (4)
C(1)—C(10)	1.530 (2)	1.520 (5)	1.538 (5)
C(1)—C(11)	1.509 (2)	1.519 (6)	1.493 (5)
C(2)—C(3)	1.515 (3)	1.508 (6)	1.525 (5)
C(3)—C(4)	1.329 (2)	1.330 (8)	1.317 (5)
C(4)—C(5)	1.521 (3)	1.511 (6)	1.520 (4)
C(5)—C(6)	1.544 (2)	1.556 (6)	1.536 (6)
C(6)—C(7)	1.532 (3)	1.542 (6)	1.534 (5)
C(6)—C(11)	1.512 (3)	1.515 (7)	1.493 (5)
C(7)—C(8)	1.514 (2)	1.508 (7)	1.509 (7)
C(8)—C(9)	1.320 (2)	1.321 (7)	1.317 (5)
C(9)—C(10)	1.519 (3)	1.499 (6)	1.509 (7)
N(3)—N(1)—C(2)	113.1 (1)	112.6 (3)	113.1 (2)
N(3)—N(1)—C(12)	109.1 (1)	109.2 (2)	109.0 (3)
C(2)—N(1)—C(12)	131.3 (2)	129.6 (3)	132.0 (3)
C(12)—N(2)—C(13)	111.6 (1)	111.1 (3)	111.4 (3)
C(12)—N(2)—C(14)	124.0 (2)	124.1 (3)	124.5 (3)
C(13)—N(2)—C(14)	123.3 (1)	124.5 (3)	123.8 (3)
N(1)—N(3)—C(5)	112.2 (1)	112.4 (2)	112.6 (3)
N(1)—N(3)—C(13)	108.8 (1)	109.3 (3)	109.8 (3)
C(5)—N(3)—C(13)	131.0 (1)	130.7 (3)	131.5 (3)
N(6)—N(4)—C(7)	112.1 (1)	111.5 (2)	111.8 (3)
N(6)—N(4)—C(15)	107.4 (1)	108.0 (3)	107.8 (3)
C(7)—N(4)—C(15)	124.3 (1)	117.7 (3)	120.6 (3)
C(15)—N(5)—C(16)	111.5 (1)	110.6 (3)	110.0 (3)
C(15)—N(5)—C(17)	124.2 (2)	124.3 (3)	125.6 (4)
C(16)—N(5)—C(17)	124.2 (2)	124.3 (4)	124.0 (3)
N(4)—N(6)—C(10)	112.0 (1)	111.3 (3)	111.6 (2)
N(4)—N(6)—C(16)	109.0 (1)	107.5 (3)	107.8 (3)
C(10)—N(6)—C(16)	123.2 (2)	118.2 (3)	120.2 (3)
C(2)—C(1)—C(6)	109.9 (2)	110.1 (3)	109.7 (2)
C(2)—C(1)—C(10)	119.6 (1)	121.5 (3)	119.4 (3)
C(2)—C(1)—C(11)	121.1 (1)	117.3 (4)	120.7 (3)
C(6)—C(1)—C(10)	109.3 (1)	110.1 (3)	109.4 (3)
C(6)—C(1)—C(11)	59.7 (1)	59.8 (3)	59.3 (3)
C(10)—C(1)—C(11)	118.0 (2)	119.2 (4)	118.7 (2)
N(1)—C(2)—C(1)	108.1 (1)	108.0 (3)	107.9 (3)
N(1)—C(2)—C(3)	102.4 (2)	102.6 (3)	102.7 (3)
C(1)—C(2)—C(3)	109.2 (1)	109.9 (3)	109.0 (3)
C(2)—C(3)—C(4)	114.2 (1)	113.5 (4)	113.6 (5)
C(3)—C(4)—C(5)	113.7 (2)	114.8 (5)	114.8 (4)
N(3)—C(5)—C(4)	102.5 (1)	102.7 (3)	102.5 (3)
N(3)—C(5)—C(6)	108.0 (1)	108.3 (3)	107.4 (2)
C(4)—C(5)—C(6)	110.3 (2)	108.7 (3)	109.7 (3)
C(1)—C(6)—C(5)	109.1 (1)	109.2 (3)	109.9 (3)
C(1)—C(6)—C(7)	109.9 (2)	109.2 (3)	109.2 (3)
C(1)—C(6)—C(11)	59.5 (1)	60.1 (3)	59.3 (2)
C(5)—C(6)—C(7)	120.2 (2)	119.7 (3)	120.6 (4)
C(5)—C(6)—C(11)	118.2 (1)	119.7 (4)	118.8 (3)
C(7)—C(6)—C(11)	120.3 (2)	119.4 (3)	119.2 (3)
N(4)—C(7)—C(6)	105.2 (1)	105.8 (3)	106.1 (2)
N(4)—C(7)—C(8)	104.5 (2)	104.7 (3)	104.7 (3)
C(6)—C(7)—C(8)	110.7 (2)	110.8 (3)	110.9 (4)
C(7)—C(8)—C(9)	113.9 (2)	113.6 (4)	113.8 (3)
C(8)—C(9)—C(10)	114.3 (2)	114.6 (4)	114.0 (3)
N(6)—C(10)—C(1)	104.5 (2)	105.6 (3)	104.9 (3)
N(6)—C(10)—C(9)	104.6 (1)	104.7 (3)	105.5 (4)
C(1)—C(10)—C(9)	111.6 (1)	110.9 (4)	110.9 (3)
C(1)—C(11)—C(6)	60.8 (1)	60.1 (3)	61.4 (3)
O(1)—C(12)—N(1)	128.6 (2)	126.9 (3)	127.8 (4)
O(1)—C(12)—N(2)	126.6 (1)	127.5 (3)	127.5 (3)
N(1)—C(12)—N(2)	104.7 (1)	105.5 (3)	104.7 (3)

Table 5 (cont.)

	(1)	(2)	(3)
O(2)—C(13)—N(2)	126.9 (1)	128.3 (4)	127.8 (4)
O(2)—C(13)—N(3)	127.9 (1)	127.5 (4)	127.5 (3)
N(2)—C(13)—N(3)	105.1 (1)	104.1 (3)	104.6 (3)
O(3)—C(15)—N(4)	126.9 (2)	126.4 (4)	125.8 (3)
O(3)—C(15)—N(5)	126.8 (1)	127.5 (4)	127.1 (4)
N(4)—C(15)—N(5)	106.2 (1)	106.1 (3)	106.9 (3)
O(4)—C(16)—N(5)	127.7 (1)	127.0 (4)	127.1 (3)
O(4)—C(16)—N(6)	126.9 (1)	126.2 (4)	126.2 (4)
N(5)—C(16)—N(6)	105.3 (1)	106.7 (3)	106.6 (3)
N(2)—C(14)—C(18)		120.2 (3)	120.1 (3)
N(2)—C(14)—C(22)		118.6 (3)	119.7 (4)
N(5)—C(17)—C(23)		120.0 (4)	120.4 (3)
N(5)—C(17)—C(27)		119.1 (4)	118.3 (3)

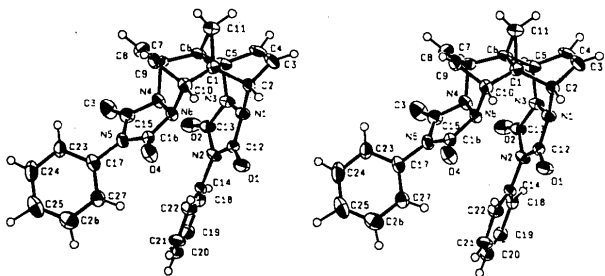


Fig. 1. Stereoscopic view of (1).

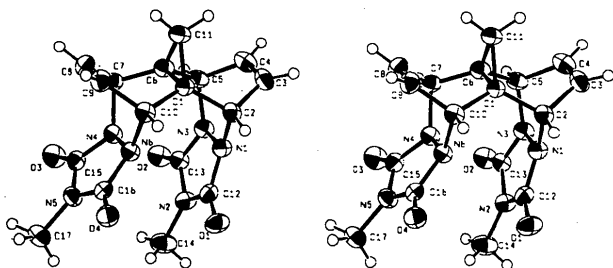


Fig. 2. Stereoscopic view of (2).

Compounds (2) and (3) also differ in the rotation of the phenyl ring with respect to the plane of the triazolinedione fragment: 49.1 (4) and 38.2 (4)° at N(2) for (2) and (3), respectively, and 61.0 (4) and 51.7 (4)° at N(5).

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## 9-Chloro-2-ethoxy-6-nitroacridine

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**Abstract.** 9-Chloro-2-ethoxy-6-nitroacridine (CENA) chloroform solvate, C<sub>15</sub>H<sub>11</sub>ClN<sub>2</sub>O<sub>3</sub>·CHCl<sub>3</sub>, *M<sub>r</sub>* = 422.10, triclinic, *P* $\bar{1}$ , *a* = 7.349 (1), *b* = 10.613 (2), *c* = 11.721 (2) Å,  $\alpha$  = 84.42 (1),  $\beta$  = 79.42 (1),  $\gamma$  = 83.23 (1)°, *V* = 889.7 (4) Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.576 g cm<sup>-3</sup>,  $\lambda$ (Mo *K*α) = 0.71073 Å,  $\mu$  = 6.85 cm<sup>-1</sup>, *F*(000) = 428, *T* = 293 K, final *R* = 0.062 for 3027 observed reflections. The chloroform solvate

crystals are stable at standard conditions, desolvate around 335 K and melt at 464 K. The acridine rings are arranged in stacks with a ring–ring stacking distance of 3.6 Å.

**Introduction.** This report is part of our ongoing investigation of acridine derivatives substituted at the 9 position (Toma, Ray, Stowell & Byrn, 1990;