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

X—H···Y	X—H	H···Y	X···Y	X—H···Y
O(21)—H(21)···O(M)	0.97 (2)	1.78 (3)	2.740 (5)	168 (4)
O(22)—H(22)···O(21)^{i}	0.97 (2)	1.67 (4)	2.639 (4)	171 (4)
O(M)—H(M)···O(23)^{i}	0.97 (4)	2.12 (4)	2.772 (5)	123 (4)

Symmetry code: (i) $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$.

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References

BORTHWICK, P. W. (1980). *Acta Cryst.* B**36**, 628–632.

BRIDEL, M. & LAVIEILLE, R. (1931). *Bull. Soc. Chim. Biol.* **13**, 781–791.

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 71–98. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

DIAS RODRIGUES, A. M. G. & LECHAT, J. R. (1988). *Acta Cryst.* C**44**, 1963–1965.

DOLDER, F., LICHTI, H., MOSETTIG, E. & QUITT, P. (1960). *J. Am. Chem. Soc.* **82**, 246–247.

DUBOIS, G. E., DIETRICH, P. S., LEE, J. F., MCGARRAUGH, G. V. & STEPHENSON, R. A. (1981). *J. Med. Chem.* **24**, 1269–1271.

ELIEL, E. (1962). *Stereochemistry of Carbon Compounds.* New York: McGraw-Hill.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

LEISEROWITZ, L. V. (1976). *Acta Cryst.* B**32**, 775–802.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MIZUKAMI, H., SHIBA, L. & OHASHI, H. (1982). *Phytochemistry*, **21**, 1927–1930.

MOSETTIG, E., BEGLINGER, U., DOLDER, F., LICHTI, H., QUITT, P. & WATERS, J. A. (1963). *J. Am. Chem. Soc.* **85**, 2305–2309.

NOVAK, A. (1974). *Struct. Bonding (Berlin)*, **18**, 177–216.

RUDDAT, M., HEFTMANN, E. & LANG, A. (1965). *Arch. Biochem. Biophys.* **110**, 496–499.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1993). C**49**, 731–735

Structures of Diels–Alder Mono-Adducts of 4-Phenyl- or 4-Methyl-1,2,4-triazolidine-3,5-dione with 11-Substituted-1,6-methano[10]annulene

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Abstract. 1*H*,2*H*,5*H*,10*H*-5a,9a-Dihydro-2-phenyl-5,10-etheno-5a,9a-methano[1,2,4]triazolo[1,2-*b*]phthalazine-1,3-dione (1), $C_{19}H_{15}N_3O_2$, $M_r = 317.35$, monoclinic, $P2_1/c$, $a = 8.500$ (4), $b = 14.271$ (7), $c = 12.758$ (6) Å, $\beta = 91.97$ (2)°, $V = 1546.67$ Å^3, $Z = 4$, $D_x = 1.363$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 0.696$ cm^{-1}, $F(000) = 664$, $R = 0.053$ for 2329 reflections. Methyl 1*H*,5*H*,10*H*-2,3,5a,9a-tetrahydro-2-methyl-1,3-dioxo-5,10-etheno-5a,9a-methano[1,2,4]triazolo[1,2-*b*]phthalazine-14-acetate (2), $C_{16}H_{15}N_3O_4$, $M_r = 313.31$, monoclinic, $P2_1$, $a = 6.421$ (3), $b = 10.872$ (5), $c = 10.564$ (5) Å, $\beta = 91.05$ (2)°, $V = 737.34$ Å^3, $Z = 2$, $D_x = 1.412$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.097$ cm^{-1}, $F(000) = 328$, $R = 0.042$ for 1262 reflections. 1*H*,2*H*,5*H*,10*H*-5a,9a-Dihydro-14-methyl-2-phenyl-5,10-etheno-5a,9a-imino[1,2,4]triazolo[1,2-*b*]phthalazine-1,3-dione (3), $C_{19}H_{16}N_4O_2$, $M_r = 332.36$, triclinic, $P\bar{1}$, $a = 26.027$ (13), $b = 10.155$ (5), $c = 6.147$ (3) Å, $\alpha = 96.99$ (3), $\beta = 93.58$ (3), $\gamma = 92.64$ (3)°, $V = 1607.09$ Å^3, $Z = 4$, $D_x = 1.374$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.086$ cm^{-1}, $F(000) = 696$, $R = 0.088$ for 3526 reflections. Crystal structures of the Diels–Alder adducts of 4-phenyl- or 4-methyl-1,2,4-triazolidine-3,5-dione with 11-substituted bridged [10]annulenes show that the mono-adduct has the Alder *endo* configuration. The triazolidinedione ring in each case is *anti* to the three-membered ring and to its substituent.

Introduction. Many Diels–Alder reactions between propellanes containing one or two cyclohexadiene rings and bridged [10]annulenes with triazolidinediones have been conducted by Kalo, Vogel & Ginsburg (1977*a*,*b*), Kalo, Bloomfield & Ginsburg (1978), Kalo & Ginsburg (1978), Ashkenazi, Kalo, Ruttimann & Ginsburg (1978), Ashkenazi, Vogel &

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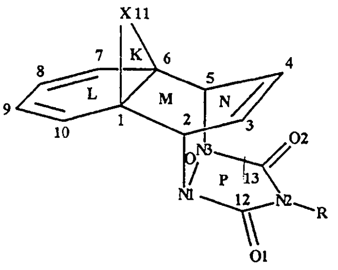
Table 1. *Crystallographic and experimental details*

	(1)	(2)	(3)
Crystal shape	Needle	Prism	Prism
Crystal size (mm)	0.4 × 0.3 × 0.2	0.25 × 0.25 × 0.3	0.4 × 0.4 × 0.3
$\Delta\omega$ (°)	1.0 + 0.1 tanθ	1.5	0.9
Scan time (s)	Variable	30	18
Background time (s)	20	20	18
$\theta_{\min}$–$\theta_{\max}$ (°)	2.5–65	2.5–25	2.5–23
hkl range: *h*	−9,9	−7,7	−28,28
k	0,16	0,12	0,11
l	0,14	0,12	0,6
Reflections measured	2671	1376	4470
Unique reflections	2540	1360	4330
Significant reflections	2329	1262	3526
Criterion for observation	$F_o > 1.5\sigma(F_o)$	$F_o > 1.5\sigma(F_o)$	$F_o > 0.0$
Weighting coefficients* *k*	1.0000	0.7945	1.7075
G	0.0155	0.0038	0.0011
Refined parameters	267	278	(290 × 2)
$(\Delta/\sigma)_{\max}$	0.6	1.1	0.9
$(\Delta\rho)_{\max}$ (e Å^{-3})	0.2	0.2	0.3
$(\Delta\rho)_{\min}$ (e Å^{-3})	−0.3	−0.2	−0.3
wR	0.072	0.049	0.088
R	0.053	0.042	0.088

* $w = k/[\sigma^2(F_o) + GF_o^2]$.

Ginsburg (1977, 1978), Ashkenazi, Peled, Vogel & Ginsburg (1979) and Gleiter & Ginsburg (1979). *A priori*, one would expect the formation of two isomeric mono-adducts, *exo* and *endo*. Only 11-cyano-1,6-methano[10]annulene afforded [see (4)] both isomeric *exo* and *endo* derivatives (Ashkenazi, Kaftory, Arad, Apeloig & Ginsburg, 1981) and their complete crystal structures have been described by Kaftory (1983) and Kaftory & Agmon (1984). In three other examples where *X* is CH_2, $CHCO_2Me$ or NMe, only the *endo* isomers were observed. We compare here the molecular structures of the three mono-adducts (1), (2) and (3).



	X	*R*
(1)	CH_2	Ph
(2)	$CH{-}CO_2Me$	Me
(3)	NMe	Ph
(4)	CHCN	Me

Table 2. *Positional parameters* (× 10^4) *and* U_{eq} *values* (Å^2 × 10^4) *for compound* (1)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
O(1)	74 (2)	1193 (1)	7559 (1)	582 (4)
O(2)	1776 (2)	1982 (1)	4315 (1)	515 (4)
N(1)	−833 (2)	2249 (1)	6311 (1)	382 (3)
N(2)	1156 (2)	1328 (1)	5918 (1)	390 (4)
N(3)	−313 (2)	2493 (1)	5283 (1)	361 (3)
C(1)	−2521 (2)	3579 (1)	6333 (2)	410 (4)
C(2)	−1169 (2)	3108 (1)	6951 (1)	417 (5)
C(3)	312 (3)	3681 (1)	6970 (2)	499 (5)
C(4)	787 (2)	3913 (1)	6017 (2)	469 (5)
C(5)	−244 (2)	3541 (1)	5136 (1)	405 (5)
C(6)	−1945 (2)	3850 (1)	5225 (1)	413 (4)
C(7)	−3040 (3)	3649 (1)	4333 (2)	530 (5)
C(8)	−4515 (3)	3364 (2)	4484 (2)	646 (7)
C(9)	−5034 (3)	3105 (2)	5507 (3)	673 (7)
C(10)	−4077 (3)	3139 (2)	6363 (2)	549 (6)
C(11)	−2373 (2)	4586 (1)	6012 (2)	474 (5)
C(12)	144 (2)	1563 (1)	6713 (1)	401 (4)
C(13)	992 (2)	1947 (1)	5083 (1)	366 (4)
C(14)	2335 (2)	608 (1)	6006 (2)	407 (4)
C(15)	3553 (3)	691 (2)	6728 (2)	576 (6)
C(16)	4696 (3)	12 (2)	6810 (2)	699 (7)
C(17)	4614 (3)	−755 (2)	6153 (2)	646 (6)
C(18)	3407 (3)	−842 (2)	5439 (2)	691 (7)
C(19)	2248 (3)	−155 (2)	5343 (2)	622 (6)

Table 3. *Positional parameters* (× 10^4) *and* U_{eq} *values* (Å^2 × 10^3) *for compound* (2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
O(1)	3746 (7)	5406 (5)	1957 (3)	77 (1)
O(2)	−2206 (5)	3123 (6)	1926 (3)	60 (1)
O(3)	1422 (5)	2310	−4593 (3)	62 (1)
O(4)	4679 (5)	2835 (6)	−4149 (3)	74 (1)
N(1)	1884 (5)	4441 (5)	337 (3)	44 (1)
N(2)	576 (6)	4448 (5)	2252 (3)	51 (1)
N(3)	−5 (5)	3727 (5)	318 (3)	40 (1)
C(1)	2689 (5)	3791 (6)	−1728 (3)	38 (1)
C(2)	3568 (6)	3823 (6)	−374 (4)	44 (1)
C(3)	3790 (7)	2536 (6)	186 (4)	47 (1)
C(4)	2042 (6)	1888 (6)	168 (4)	41 (1)
C(5)	224 (6)	2559 (6)	−414 (3)	37 (1)
C(6)	652 (5)	3022 (6)	−1762 (3)	36 (1)
C(7)	−1141 (7)	3533 (6)	−2471 (4)	50 (1)
C(8)	−987 (9)	4575 (7)	−3122 (4)	60 (1)
C(9)	908 (10)	5301 (6)	−3086 (4)	63 (2)
C(10)	2582 (9)	4972 (6)	−2409 (4)	52 (1)
C(11)	2596 (6)	2571 (6)	−2423 (4)	39 (1)
C(12)	2262 (8)	4820 (6)	1569 (4)	52 (1)
C(13)	−720 (7)	3692 (6)	1561 (4)	44 (1)
C(14)	228 (16)	4742 (8)	3580 (5)	78 (2)
C(15)	2763 (6)	2574 (6)	−3834 (4)	43 (1)
C(16)	5229 (13)	2754 (13)	−5472 (7)	100 (3)

Experimental. The three mono-adducts have been obtained by a Diels–Alder reaction of 4-substituted 1,2,4-triazolidine-3,5-diones with bridged [10]annulenes. Procedures are given in the references mentioned above. Crystallographic data, details of intensity measurements and structure refinement are given in Table 1. Densities were not measured. A Philips PW1100 diffractometer with graphite-monochromated Cu *Kα* [for (1)] and Mo *Kα* [for (2) and (3)] radiations and $\theta/2\theta$ scans was used for data collection. Lattice parameters calculated using 25 reflections for each compound. Three reflections (for each compound) monitored; variation in intensities 5%. Absorption and extinction corrections not applied. Structures solved either by *MULTAN*76 (Main, Lessinger, Woolfson, Germain & Declercq, 1977) or by *SHELX*76 (Sheldrick, 1976) and refined by full-matrix least squares (based on *F*) with anisotropic atomic displacement parameters for O, N and C atoms, isotropic for H atoms; scattering factors were taken from Cromer & Mann (1968) and from Stewart, Davidson & Simpson (1965). The two independent molecules in (3) were refined in separate blocks. The agreement factor of 0.088 is slightly high because all the reflections ($F_o > 0.0$) were used in the refinement. All H-atom positions were located from difference Fourier maps.

Table 4. *Positional parameters* (× 10^4) *and* U_{eq} *values* ($Å^2 \times 10^3$) *for compound* (3)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Molecule *A*				
O(1)	3148 (2)	3958 (4)	2511 (6)	48 (1)
O(2)	3981 (2)	2769 (4)	8695 (7)	52 (1)
N(1)	3772 (2)	5051 (4)	5070 (7)	33 (1)
N(2)	3551 (2)	2946 (4)	5289 (7)	34 (1)
N(3)	4020 (2)	4691 (4)	7018 (7)	35 (1)
N(4)	4028 (2)	8158 (4)	8268 (7)	39 (1)
C(1)	4041 (2)	7325 (5)	6159 (9)	40 (1)
C(2)	3559 (2)	6424 (5)	5423 (10)	38 (1)
C(3)	3203 (3)	6368 (5)	7264 (10)	41 (2)
C(4)	3427 (2)	6008 (6)	9054 (12)	46 (2)
C(5)	3991 (2)	5761 (5)	8949 (9)	41 (1)
C(6)	4295 (2)	6928 (5)	8216 (9)	38 (1)
C(7)	4865 (3)	6930 (6)	8360 (13)	60 (2)
C(8)	5134 (3)	7390 (7)	6845 (14)	68 (2)
C(9)	4893 (3)	7782 (7)	4893 (14)	70 (2)
C(10)	4378 (3)	7681 (6)	4463 (11)	57 (2)
C(11)	4306 (3)	9449 (7)	8613 (16)	56 (2)
C(12)	3449 (2)	3987 (5)	4124 (9)	36 (1)
C(13)	3863 (2)	3389 (5)	7232 (9)	38 (1)
C(14)	3335 (2)	1623 (5)	4735 (9)	36 (1)
C(15)	3062 (2)	993 (6)	6137 (12)	48 (2)
C(16)	2845 (2)	−282 (6)	5514 (12)	56 (2)
C(17)	2908 (3)	−900 (6)	3416 (12)	55 (2)
C(18)	3175 (3)	−268 (6)	1985 (12)	55 (2)
C(19)	3394 (2)	998 (6)	2618 (10)	47 (2)
Molecule *B*				
O(1)	1008 (2)	−1935 (4)	4276 (6)	51 (1)
O(2)	1844 (2)	−524 (4)	−1421 (6)	52 (1)
N(1)	977 (2)	−29 (4)	2592 (7)	39 (1)
N(2)	1433 (2)	−1658 (4)	1096 (7)	33 (1)
N(3)	1230 (2)	415 (4)	789 (7)	34 (1)
N(4)	982 (2)	3451 (4)	3845 (7)	39 (1)
C(1)	713 (2)	2142 (5)	3669 (9)	39 (1)
C(2)	1013 (2)	1063 (5)	4503 (9)	39 (1)
C(3)	1582 (2)	1455 (6)	4971 (10)	45 (2)
C(4)	1798 (3)	1869 (6)	3274 (11)	48 (2)
C(5)	1443 (2)	1826 (5)	1275 (9)	36 (1)
C(6)	966 (2)	2609 (5)	1687 (9)	39 (1)
C(7)	623 (3)	2877 (6)	−144 (11)	52 (2)
C(8)	109 (3)	2834 (7)	−41 (13)	66 (2)
C(9)	−129 (3)	2376 (7)	1823 (15)	71 (3)
C(10)	143 (3)	1996 (7)	3471 (14)	56 (2)
C(11)	695 (3)	4661 (6)	4042 (12)	46 (2)
C(12)	1131 (2)	−1280 (5)	2856 (8)	35 (1)
C(13)	1549 (2)	−586 (5)	11 (9)	39 (1)
C(14)	1643 (2)	−2929 (5)	660 (9)	35 (1)
C(15)	1604 (2)	−3566 (6)	−1445 (11)	44 (2)
C(16)	1819 (2)	−4771 (6)	−1911 (12)	51 (2)
C(17)	2067 (3)	−5333 (6)	−283 (13)	56 (2)
C(18)	2104 (2)	−4714 (6)	1877 (13)	53 (2)
C(19)	1886 (2)	−3489 (5)	2351 (10)	43 (1)

Discussion. Final positional parameters for (1)–(3) are given in Tables 2–4.* Molecular stereoviews for (1)–(3) are shown in Figs. 1–3. Bond lengths and angles are given in Tables 5 and 6.

Similarity of the molecular conformations of the three compounds can be seen by comparison of the angles between the various planes as denoted by *K* to *P* in the structural diagram (Table 7). The three compounds differ in the substituents *X* and *R* and in the steric effects imposed by these substituents, expressed in the angles between the various planes. The angle between *K* and *L* is smaller in (1) [106.2 (2)°] than in (2) [109.1 (5)° average] or (3) [110.9 (4)°], in accordance with the difference in repulsion between hydrogen [at C(11) in (1)], methyl [in (3)] and methyl acetate [in (2)] with the cyclohexadiene portion (plane *L*). The effect is therefore also imposed on the angles between *K* and *M* and *L* and *M* (to account for the constant sum of angles being 360°).

All bond lengths and angles are normal. The geometry within the triazolidinedione moiety is in accordance with that expected as discussed by Kaftory & Agmon (1984).

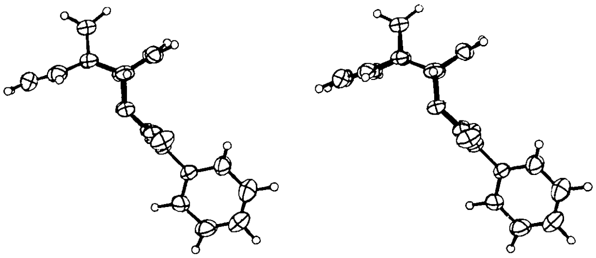


Fig. 1. Stereoscopic view of compound (1).

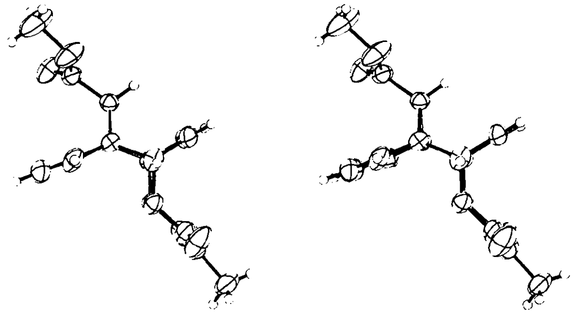


Fig. 2. Stereoscopic view of compound (2).

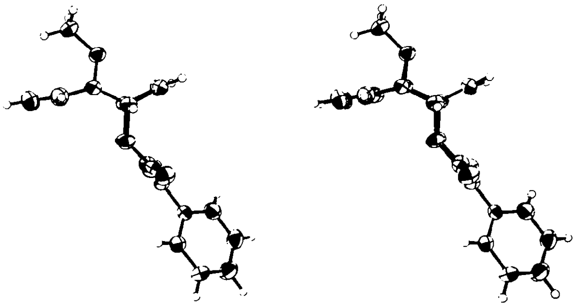


Fig. 3. Stereoscopic view of compound (3*A*).

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* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55812 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1020]

Table 5. *Selected bond lengths* (Å) *for compounds* (1), (2), (3*A*) *and* (3*B*)

Compound (1)	
O(1)—C(12)	1.205 (1)
O(2)—C(13)	1.205 (2)
N(1)—N(3)	1.441 (1)
N(1)—C(2)	1.506 (1)
N(1)—C(12)	1.372 (2)
N(2)—C(12)	1.393 (2)
N(2)—C(13)	1.387 (1)
N(2)—C(14)	1.437 (2)
N(3)—C(5)	1.508 (2)
N(3)—C(13)	1.386 (2)
C(1)—C(2)	1.527 (2)
C(1)—C(6)	1.561 (2)
C(1)—C(10)	1.465 (3)
C(1)—C(11)	1.501 (2)
C(2)—C(3)	1.501 (2)
C(3)—C(4)	1.336 (3)
C(4)—C(5)	1.499 (2)
C(5)—C(6)	1.519 (2)
C(6)—C(7)	1.474 (2)
C(6)—C(11)	1.507 (2)
C(7)—C(8)	1.339 (3)
C(8)—C(9)	1.441 (4)
C(9)—C(10)	1.340 (4)

Compound (2)	
O(1)—C(12)	1.211 (7)
O(2)—C(13)	1.206 (6)
O(3)—C(15)	1.200 (5)
O(4)—C(15)	1.312 (5)
O(4)—C(16)	1.450 (8)
N(1)—N(3)	1.440 (5)
N(1)—C(2)	1.488 (5)
N(1)—C(12)	1.382 (5)
N(2)—C(12)	1.372 (6)
N(2)—C(13)	1.371 (6)
N(2)—C(14)	1.460 (6)
N(3)—C(5)	1.495 (7)
N(3)—C(13)	1.399 (5)
C(1)—C(2)	1.529 (5)
C(1)—C(6)	1.552 (6)
C(1)—C(10)	1.473 (8)
C(1)—C(11)	1.516 (8)
C(2)—C(3)	1.525 (8)
C(3)—C(4)	1.325 (7)
C(4)—C(5)	1.499 (6)
C(5)—C(6)	1.541 (5)
C(6)—C(7)	1.471 (6)
C(6)—C(11)	1.522 (5)
C(7)—C(8)	1.330 (9)
C(8)—C(9)	1.450 (9)
C(9)—C(10)	1.329 (7)
C(11)—C(15)	1.496 (5)

Compound (3*A*)	
O(1)—C(12)	1.222 (6)
O(2)—C(13)	1.191 (7)
N(1)—N(3)	1.420 (5)
N(1)—C(2)	1.521 (6)
N(1)—C(12)	1.384 (6)
N(2)—C(12)	1.373 (6)
N(2)—C(13)	1.416 (6)
N(2)—C(14)	1.425 (6)
N(3)—C(5)	1.517 (6)
N(3)—C(13)	1.390 (6)
N(4)—C(1)	1.462 (6)
N(4)—C(6)	1.455 (6)
N(4)—C(11)	1.453 (8)
C(1)—C(2)	1.528 (7)
C(1)—C(6)	1.498 (7)
C(1)—C(10)	1.472 (9)
C(2)—C(3)	1.511 (8)
C(3)—C(4)	1.312 (9)
C(4)—C(5)	1.505 (8)
C(5)—C(6)	1.524 (7)
C(6)—C(7)	1.479 (8)
C(7)—C(8)	1.32 (1)
C(8)—C(9)	1.43 (1)
C(9)—C(10)	1.35 (1)

Compound (3*B*)	
O(1)—C(12)	1.210 (6)
O(2)—C(13)	1.210 (7)
N(1)—N(3)	1.432 (6)
N(1)—C(2)	1.509 (6)
N(1)—C(12)	1.374 (6)
N(2)—C(12)	1.403 (6)
N(2)—C(13)	1.375 (6)
N(2)—C(14)	1.426 (6)
N(3)—C(5)	1.501 (6)
N(3)—C(13)	1.399 (6)
N(4)—C(1)	1.463 (6)
N(4)—C(6)	1.485 (6)
N(4)—C(11)	1.464 (7)
C(1)—C(2)	1.497 (7)
C(1)—C(6)	1.535 (7)
C(1)—C(10)	1.478 (8)
C(2)—C(3)	1.516 (8)
C(3)—C(4)	1.320 (9)
C(4)—C(5)	1.486 (8)
C(5)—C(6)	1.523 (7)
C(6)—C(7)	1.451 (8)
C(7)—C(8)	1.34 (1)
C(8)—C(9)	1.45 (1)
C(9)—C(10)	1.31 (1)

Table 6. *Selected bond angles* (°) *for compounds* (1), (2), (3*A*) *and* (3*B*)

Compound (1)	
N(3)—N(1)—C(2)	111.5 (1)
N(3)—N(1)—C(12)	108.2 (1)
C(2)—N(1)—C(12)	120.1 (1)
C(12)—N(2)—C(13)	110.9 (1)
C(12)—N(2)—C(14)	124.2 (1)
C(13)—N(2)—C(14)	124.5 (1)
N(1)—N(3)—C(5)	111.6 (1)
N(1)—N(3)—C(13)	107.7 (1)
C(5)—N(3)—C(13)	120.0 (1)
C(2)—C(1)—C(6)	108.8 (1)
C(2)—C(1)—C(10)	117.5 (1)
C(2)—C(1)—C(11)	119.6 (1)
C(6)—C(1)—C(10)	116.2 (1)
C(6)—C(1)—C(11)	58.9 (1)
C(10)—C(1)—C(11)	120.1 (1)
N(1)—C(2)—C(1)	103.4 (1)
N(1)—C(2)—C(3)	106.1 (1)
C(1)—C(2)—C(3)	112.6 (1)
C(2)—C(3)—C(4)	113.6 (1)
C(3)—C(4)—C(5)	114.1 (1)
N(3)—C(5)—C(4)	106.4 (1)
N(3)—C(5)—C(6)	103.7 (1)
C(4)—C(5)—C(6)	112.0 (1)
C(1)—C(6)—C(5)	109.0 (1)
C(1)—C(6)—C(7)	116.2 (1)
C(1)—C(6)—C(11)	58.6 (1)
C(5)—C(6)—C(7)	117.6 (1)
C(5)—C(6)—C(11)	120.3 (1)
C(7)—C(6)—C(11)	119.4 (1)
C(6)—C(7)—C(8)	121.2 (2)
C(7)—C(8)—C(9)	121.7 (2)
C(8)—C(9)—C(10)	122.2 (2)
C(1)—C(10)—C(9)	121.2 (2)
C(1)—C(11)—C(6)	62.5 (1)
O(1)—C(12)—N(1)	126.9 (1)
O(1)—C(12)—N(2)	126.7 (1)
N(1)—C(12)—N(2)	106.3 (1)
O(2)—C(13)—N(2)	127.4 (1)
O(2)—C(13)—N(3)	126.5 (1)
N(2)—C(13)—N(3)	106.1 (1)
N(2)—C(14)—C(15)	120.0 (1)
N(2)—C(14)—C(19)	119.6 (1)

Compound (2)	
C(15)—O(4)—C(16)	118.5 (4)
N(3)—N(1)—C(2)	111.7 (4)
N(3)—N(1)—C(12)	107.9 (3)
C(2)—N(1)—C(12)	119.5 (3)
C(12)—N(2)—C(13)	111.9 (3)
C(12)—N(2)—C(14)	125.2 (5)
C(13)—N(2)—C(14)	122.7 (5)
N(1)—N(3)—C(5)	112.0 (3)
N(1)—N(3)—C(13)	107.0 (3)
C(5)—N(3)—C(13)	119.9 (4)
C(2)—C(1)—C(6)	109.3 (3)
C(2)—C(1)—C(10)	116.7 (4)
C(2)—C(1)—C(11)	118.9 (4)
C(6)—C(1)—C(10)	115.2 (3)
C(6)—C(1)—C(11)	59.5 (3)
C(10)—C(1)—C(11)	121.7 (3)
N(1)—C(2)—C(1)	102.9 (3)
N(1)—C(2)—C(3)	106.4 (3)
C(1)—C(2)—C(3)	111.8 (4)
C(2)—C(3)—C(4)	114.2 (4)
C(3)—C(4)—C(5)	113.6 (5)
N(3)—C(5)—C(4)	106.6 (3)
N(3)—C(5)—C(6)	102.8 (4)
C(4)—C(5)—C(6)	112.8 (3)
C(1)—C(6)—C(5)	108.6 (2)
C(1)—C(6)—C(7)	117.4 (4)
C(1)—C(6)—C(11)	59.1 (3)
C(5)—C(6)—C(7)	116.3 (3)
C(5)—C(6)—C(11)	118.7 (4)
C(7)—C(6)—C(11)	122.0 (3)
C(6)—C(7)—C(8)	121.3 (4)
C(7)—C(8)—C(9)	121.4 (5)
C(8)—C(9)—C(10)	122.5 (5)
C(1)—C(10)—C(9)	121.8 (5)
C(1)—C(11)—C(6)	61.4 (3)
C(1)—C(11)—C(15)	118.5 (4)
C(6)—C(11)—C(15)	122.0 (3)
O(1)—C(12)—N(1)	126.8 (4)
O(1)—C(12)—N(2)	126.9 (4)
N(1)—C(12)—N(2)	106.3 (4)
O(2)—C(13)—N(2)	127.8 (4)
O(2)—C(13)—N(3)	126.1 (4)
N(2)—C(13)—N(3)	106.1 (4)
O(3)—C(15)—O(4)	123.1 (3)
O(3)—C(15)—C(11)	127.0 (3)
O(4)—C(15)—C(11)	109.8 (3)

Compound (3*A*)	
N(3)—N(1)—C(2)	112.3 (3)
N(3)—N(1)—C(12)	108.0 (3)
C(2)—N(1)—C(12)	119.0 (4)
C(12)—N(2)—C(13)	110.7 (4)
C(12)—N(2)—C(14)	125.0 (4)
C(13)—N(2)—C(14)	124.1 (4)
N(1)—N(3)—C(5)	111.6 (3)
N(1)—N(3)—C(13)	108.7 (4)
C(5)—N(3)—C(13)	119.3 (4)
C(1)—N(4)—C(6)	61.8 (3)
C(1)—N(4)—C(11)	119.5 (5)
C(6)—N(4)—C(11)	121.7 (4)
N(4)—C(1)—C(2)	116.0 (4)
N(4)—C(1)—C(16)	58.8 (3)
N(4)—C(1)—C(10)	121.7 (4)
C(2)—C(1)—C(6)	110.3 (4)
C(2)—C(1)—C(10)	118.0 (5)
C(6)—C(1)—C(10)	117.2 (5)
N(1)—C(2)—C(1)	102.9 (4)
N(1)—C(2)—C(3)	105.1 (3)
C(1)—C(2)—C(3)	111.5 (5)
C(2)—C(3)—C(4)	113.6 (5)
C(3)—C(4)—C(5)	115.2 (5)
N(3)—C(5)—C(4)	105.2 (4)
N(3)—C(5)—C(6)	102.1 (4)
C(4)—C(5)—C(6)	112.1 (4)
N(4)—C(6)—C(1)	59.3 (3)
N(4)—C(6)—C(5)	115.4 (4)
N(4)—C(6)—C(7)	121.5 (4)
C(1)—C(6)—C(5)	110.3 (4)
C(1)—C(6)—C(7)	117.0 (4)
C(5)—C(6)—C(7)	118.6 (4)
C(6)—C(7)—C(8)	120.9 (6)
C(7)—C(8)—C(9)	121.9 (7)
C(8)—C(9)—C(10)	122.3 (7)
C(1)—C(10)—C(9)	119.9 (6)
O(1)—C(12)—N(1)	126.9 (4)
O(1)—C(12)—N(2)	126.5 (4)
N(1)—C(12)—N(2)	106.6 (4)
O(2)—C(13)—N(2)	127.7 (5)
O(2)—C(13)—N(3)	127.5 (5)
N(2)—C(13)—N(3)	104.8 (4)
N(2)—C(14)—C(15)	122.5 (5)
N(2)—C(14)—C(19)	117.5 (4)

Compound (3*B*)	
N(3)—N(1)—C(2)	109.8 (3)
N(3)—N(1)—C(12)	108.3 (3)
C(2)—N(1)—C(12)	121.0 (4)
C(12)—N(2)—C(13)	110.4 (4)
C(12)—N(2)—C(14)	124.7 (3)
C(13)—N(2)—C(14)	124.6 (4)
N(1)—N(3)—C(5)	112.7 (3)
N(1)—N(3)—C(13)	107.3 (3)
C(5)—N(3)—C(13)	119.9 (4)
C(1)—N(4)—C(6)	62.7 (3)
C(1)—N(4)—C(11)	120.7 (4)
C(6)—N(4)—C(11)	119.1 (4)
N(4)—C(1)—C(2)	116.1 (4)
N(4)—C(1)—C(6)	59.4 (3)
N(4)—C(1)—C(10)	121.1 (4)
C(2)—C(1)—C(6)	109.5 (4)
C(2)—C(1)—C(10)	119.2 (4)
C(6)—C(1)—C(10)	115.7 (5)
N(1)—C(2)—C(1)	104.1 (4)
N(1)—C(2)—C(3)	106.1 (4)
C(1)—C(2)—C(3)	112.8 (4)
C(2)—C(3)—C(4)	113.1 (5)
C(3)—C(4)—C(5)	114.1 (6)
N(3)—C(5)—C(4)	105.7 (4)
N(3)—C(5)—C(6)	103.4 (4)
C(4)—C(5)—C(6)	112.7 (4)
N(4)—C(6)—C(1)	57.9 (3)
N(4)—C(6)—C(5)	115.1 (4)
N(4)—C(6)—C(7)	121.1 (4)
C(1)—C(6)—C(5)	108.7 (4)
C(1)—C(6)—C(7)	116.9 (4)
C(5)—C(6)—C(7)	120.2 (4)
C(6)—C(7)—C(8)	121.8 (5)
C(7)—C(8)—C(9)	120.8 (7)
C(8)—C(9)—C(10)	122.2 (7)
C(1)—C(10)—C(9)	122.0 (6)
O(1)—C(12)—N(2)	127.5 (4)
O(1)—C(12)—N(1)	126.1 (4)
N(2)—C(12)—N(1)	106.3 (3)
O(2)—C(13)—N(2)	127.7 (4)
O(2)—C(13)—N(3)	125.8 (4)
N(2)—C(13)—N(3)	106.4 (4)
N(2)—C(14)—C(15)	119.3 (4)
N(2)—C(14)—C(19)	119.7 (4)

Table 7. *Comparison of angles* (°) *between planes in compounds* (1), (2), (3*A*) *and* (3*B*)

	K—*L*	*K*—*M*	*L*—*M*	*M*—*N*	*M*—*O*	*N*—*O*	*O*—*P*
(1)	106.2 (2)	114.1 (2)	139.6 (2)	126.1 (2)	113.7 (3)	120.2 (3)	136.4 (3)
(2)	110.9 (4)	112.9 (4)	136.2 (5)	126.2 (5)	113.0 (5)	120.8 (5)	135.6 (5)
(3*A*)	109.1 (5)	108.5 (5)	142.4 (6)	127.0 (6)	113.5 (5)	119.5 (5)	133.4 (5)
(3*B*)	109.1 (5)	108.9 (5)	142.1 (6)	126.8 (6)	113.8 (5)	119.4 (5)	134.0 (5)

References

ASHKENAZI, P., KAFTORY, M., ARAD, D., APELOIG, Y. & GINSBURG, D. (1981). *Helv. Chim. Acta*, **64**, 579–581.

ASHKENAZI, P., KALO, J., RUTTIMANN, A. & GINSBURG, D. (1978). *Tetrahedron*, **34**, 2161–2165.

Ashkenazi, P., Peled, M., Vogel, E. & Ginsburg, D. (1979). *Tetrahedron*, **35**, 1321–1327.
Ashkenazi, P., Vogel, E. & Ginsburg, D. (1977). *Tetrahedron*, **33**, 1169–1175.
Ashkenazi, P., Vogel, E. & Ginsburg, D. (1978). *Tetrahedron*, **34**, 2167–2169.
Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
Gleiter, R. & Ginsburg, D. (1979). *Pure Appl. Chem.* **51**, 1301–1315.
Kaftory, M. (1983). *J. Am. Chem. Soc.* **105**, 3832–3836.
Kaftory, M. & Agmon, I. (1984). *J. Am. Chem. Soc.* **106**, 7785–7793.
Kalo, J., Bloomfield, J. J. & Ginsburg, D. (1978). *Tetrahedron*, **34**, 2153–2155.
Kalo, J. & Ginsburg, D. (1978). *Tetrahedron*, **34**, 2155–2160.
Kalo, J., Vogel, E. & Ginsburg, D. (1977*a*). *Tetrahedron*, **33**, 1177–1182.
Kalo, J., Vogel, E. & Ginsburg, D. (1977*b*). *Tetrahedron*, **33**, 1183–1188.
Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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2,2′-Bis(acetamido)biphenyl

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Abstract. 2,2′-Bis(acetylamino)biphenyl, $C_{16}H_{16}N_2O_2$, $M_r = 268.32$, monoclinic, $P2_1/n$, $a = 9.808$ (4), $b = 8.780$ (5), $c = 17.248$ (6) Å, $\beta = 101.70$ (4)°, $V = 1454.44$ (7) Å^3, $Z = 4$, $D_m = 1.21$ (2), $D_x = 1.225$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418, $\mu = 6.71$ cm^{-1}, $F(000) = 568$, $T = 293$ K, $R = 0.048$ for 1084 independent reflections. The torsion angle between the phenyl rings is 91.6 (6)°. Conformational parameters are calculated and compared to those of some other 2,2′-biphenyl molecules.

Introduction. The torsion angle between aromatic rings in biphenyl compounds is of great importance for the activity of these molecules on the central nervous system (Shukla, Galy, Brouant, Galy, Soyfer & Barbe, 1985). This angle depends on the nature of substituents branching from the phenyl rings.

To gain some insight on the structure of these compounds, crystal structures of 2-acetamido-2′-diacetamidobiphenyl (ADB) (Reboul, Pèpe, Siri, Oddon, Rahal, Soyfer & Barbe, 1992), 2-nitro-2′-diacetamidobiphenyl (NDB) (Reboul, Rahal, Pèpe, Oddon, Siri, Astier, Soyfer & Barbe, 1992) and of 2,2′-bis(acetamido)biphenyl (BAB) have been investi-

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