Discussion. The benzodiazepine ring of (2) possesses an envelope-type conformation (see Fig. 1), the same as its benzylamino derivative (1). The angle between planes C4—C5—C6—C7—C8—C9—C10—C11 (plane 1) and C4—C7—N3—N1—C2 (plane 2) is 124·5 (3)° [124·7 (1)° in (1)]. The displacements of atoms from plane 1 [highest value for C8: −0·02 (1) Å] and from plane 2 [highest value for N3 0·012 (9) Å] are lower than in (1) [0·131 (7) for C7 and 0·116 (10) Å for N2]. The differences between torsion angles C2—N1—C7—C6 = −53·4 (1·3) and C2—N3—C4—C5 = 55·2 (1·3)° are also less in (2) than in (1) [−48·8 (1·0) and 65·8 (1·0)° respectively]. This indicates smaller distortions of the benzodiazepine ring in compound (2) than in compound (1). Both diazepine N atoms are protonated. The bonds N2—C2 = 1·351 (13), N1—C2 = 1·318 (13) and N3—C2 = 1·332 (12) Å are shorter than normal single C—N bonds [*e.g.* N2—C12 = 1·453 (13) Å], which is characteristic of the ionic form of the benzodiazepine group, occurring also in crystals of (1). The substitution of a Cl^- ion in (1) by an I^- ion in (2) did not change the motif of hydrogen bonds (see Fig. 2); the ionic form of (2) is stabilized by interactions N2···I1 with a distance of 3·689 (10), N(3)···I1 of 3·570 (9) and N1′*···I1 of 3·622 (9) Å (N1′* means the atom related to N1 by $\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$).

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Tetrabenzo[*b*,*g*,*k*,*p*][1,10,4,6,13,15]dioxatetraazacyclooctadeca[2,4,5,7,11,13,14,16]-octaene (III)*

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Abstract. $C_{28}H_{20}N_4O_2$, $M_r = 444{\cdot}49$, monoclinic, $C2/c$, $a = 19{\cdot}532\ (3)$, $b = 7{\cdot}858\ (1)$, $c = 14{\cdot}997\ (5)$ Å, $\beta = 98{\cdot}91\ (2)°$, $V = 2274\ (6)$ Å^3, $Z = 4$, $D_m = 1{\cdot}294$, $D_x = 1{\cdot}298\ (1)$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 5{\cdot}91$ cm^{-1}, $F(000) = 928$, room temperature, $R = 0{\cdot}0377$ for 1897 reflections with $I \geq 3\sigma(I)$. The molecule contains an 18-membered heterocyclic ring which adopts a 'saddle'-like conformation with two non-linear diimino systems. A crystallographic twofold axis passes through the centre of the ring. Two of the four benzene rings are nearly parallel to each other.

Introduction. Many examples of the biological activity of carbodiimides are known, *e.g.* their bacteriostatic and anticancer properties (Roberts, Rounds & Shankman, 1961), and its application to immunological research (Goodfriend, Levine & Fasman, 1964). At present, however, cyclic carbo-

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* Dedicated to Professor Reginald Gruehn (Justus-Liebig-Universitat-Giessen, Germany) on the occasion of his 60th birthday.

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Table 1. *Final fractional coordinates* (× 10^4) *and isotropic temperature factors* (× 10^4) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq} (Å^2)
O1	3888 (1)	3445 (2)	1317 (1)	636 (4)
O1†	6112 (1)	3445 (2)	3683 (1)	636 (4)
C1	5806 (1)	4146 (3)	4421 (1)	609 (6)
C1†	4194 (1)	4146 (3)	579 (1)	609 (6)
C3	5336 (1)	5557 (2)	4046 (1)	542 (6)
C3†	4664 (1)	5557 (2)	954 (1)	542 (6)
C4	4688 (1)	5190 (2)	3553 (1)	521 (5)
C4†	5312 (1)	5190 (2)	1447 (1)	521 (5)
N1	4499 (1)	3465 (2)	3442 (1)	584 (5)
N1†	5501 (1)	3465 (2)	1558 (1)	584 (5)
C5	3977 (1)	2821 (2)	3036 (1)	534 (5)
C5†	6023 (1)	2821 (2)	1964 (1)	534 (5)
N2	3462 (1)	1983 (2)	2766 (1)	593 (5)
N2†	6538 (1)	1983 (2)	2234 (1)	593 (5)
C6	3238 (1)	1341 (2)	1887 (1)	534 (6)
C6†	6762 (1)	1341 (2)	3113 (1)	534 (6)
C7	3468 (1)	2068 (2)	1138 (1)	546 (5)
C7†	6532 (1)	2068 (2)	3862 (1)	546 (5)
C8	3250 (1)	1382 (3)	283 (1)	669 (7)
C8†	6750 (1)	1382 (3)	4717 (1)	669 (7)
C9	2799 (1)	9 (3)	187 (2)	764 (8)
C9†	7201 (1)	9 (3)	4813 (2)	764 (8)
C10	2562 (1)	−681 (3)	928 (2)	784 (8)
C10†	7438 (1)	−681 (3)	4072 (2)	784 (8)
C11	2783 (1)	−9 (2)	1781 (1)	659 (7)
C11†	7217 (1)	−9 (2)	3219 (1)	659 (7)
C12	4248 (1)	6488 (2)	3206 (1)	633 (7)
C12†	5752 (1)	6488 (2)	1794 (1)	633 (7)
C13	4446 (1)	8158 (3)	3360 (1)	751 (8)
C13†	5554 (1)	8158 (3)	1640 (1)	751 (8)
C14	5085 (1)	8544 (3)	3855 (2)	785 (8)
C14†	4915 (1)	8544 (3)	1145 (2)	785 (8)
C15	5523 (1)	7242 (3)	4190 (1)	674 (6)
C15†	4477 (1)	7242 (3)	810 (1)	674 (6)

† Atoms in symmetrically related part of molecule.

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

C5	N1	1·215 (2)	C5	N2	1·218 (2)
N1	C4	1·407 (2)	N2	C6	1·416 (2)
C4	C3	1·393 (2)	C6	C7	1·395 (2)
C3	C15	1·382 (2)	C7	C8	1·395 (2)
C15	C14	1·378 (3)	C8	C9	1·386 (3)
C14	C13	1·383 (3)	C9	C10	1·380 (3)
C13	C12	1·378 (3)	C10	C11	1·389 (3)
C12	C4	1·382 (2)	C11	C6	1·377 (2)
C3	C1	1·493 (2)	C7	O1	1·359 (2)
O1	C1†	1·446 (3)			

C1†	O1	C7	117·4 (2)	O1†	C1	C3	107·3 (1)
O1	C7	C6	115·2 (1)	C1	C3	C4	120·1 (2)
O1	C7	C8	125·3 (2)	C1	C3	C15	121·3 (2)
C7	C8	C9	119·7 (2)	C3	C15	C14	121·3 (2)
C8	C9	C10	120·6 (2)	C15	C14	C13	119·5 (2)
C9	C10	C11	119·9 (2)	C14	C13	C12	120·3 (2)
C10	C11	C6	120·1 (2)	C13	C12	C4	119·9 (2)
C11	C6	C7	120·3 (2)	C12	C4	C3	120·5 (2)
C6	C7	C8	119·4 (2)	C4	C3	C15	118·6 (2)
C7	C6	N2	120·8 (1)	C3	C4	N1	117·5 (1)
C11	C6	N2	118·8 (2)	C12	C4	N1	122·0 (2)
C6	N2	C5	128·4 (1)	C4	N1	C5	130·1 (1)
N1	C5	N2	168·1 (2)				

† Atoms in symmetrically related part of molecule.

diimides having fused aromatic rings are less well known; the syntheses of only two such compounds have been described (Hiatt, Shaio & Fauzi, 1979; Richter, Tucker & Ulrich, 1983). This aroused our interest in this group of compounds.

In the course of synthesis of nine-membered systems of cyclic carbodiimides, 12*H*-dibenzo[*b*,*g*][1,4,6]oxadiazonine (II) was obtained by desulfuration of 6,7-dihydro-5*H*,12*H*-dibenzo[*b*,*g*][1,4,6]-oxadiazonine-6-thione (I) with yellow mercuric oxide. Both the starting material and product were investigated by means of elemental analysis and ^{1}H NMR, IR and MS [for (I)] (Glinka & Piątowska, 1984). However, the MS results for the product indicated a dimer molecule for (II), namely, tetrabenzo[*b*,*g*,*k*,*p*][1,10,4,6,13,15]dioxatetraazacyclooctadeca[2,7,11,16]tetraene-6,19-dione, which possibly forms during the process of desulfuration. The biological activity of the desulfuration product is practically zero. This fact could be connected with a relatively large molecule of (III).

To elucidate the course of the desulfuration process and the structure of the product molecule, an X-ray crystal structure analysis was undertaken.

Experimental. Colourless crystals of size ~1 mm were obtained from acetone at room temperature. The specimen used for the X-ray work, 0·3 × 0·3 × 0·4 mm, was cut from larger crystal. D_m measured by flotation. Data were recorded on a CAD-4 diffractometer using the θ–2θ scan technique, and graphite-monochromatized Cu $K\alpha$ radiation. Unit-cell parameters were obtained by least-squares refinement on setting angles of 25 reflections in the range 14·6–46·2°. Range of h,k,l was −24 to 23, −9 to 9, 0 to 18, respectively; total of 9434 reflections (half Ewald sphere) measured to $(\sin\theta)/\lambda$ = 0·063 Å^{-1}, data not corrected for absorption. Standard reflections, 424, 753, 10,4,6, mean variation 1·0, 2·3 and 1·2%, respectively; $R_{\rm int}$ = 5·8%, 2190 reflections with $I \geq 3\sigma(I)$ of which 1897 used in calculation. Solution by direct methods using *SHELX76* (Sheldrick, 1976), all H atoms located from a difference map, refinement by a full-matrix least-squares procedure on F magnitudes, 194 parameters. Refinement to final R = 0·0377, unit weights, S = 0·7585. Max. shift/e.s.d. = 0·004; the largest peaks on a final difference map were 0·15 and −0·14 e Å^{-3}. Scattering factors from *SHELX76*.

Discussion. The final atomic coordinates are listed in Table 1,* bond lengths and angles in Table 2; the molecular structure and the atom- and ring-numbering scheme is given in Fig. 1. The geometry of the molecule was calculated using *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1987).

A double-molecule compound (III), with respect to the expected form of the molecule (II), is formed

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* Lists of structure factors, thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54343 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

during the desulfuration of (I) and an 18-membered heterocyclic macro-ring is created. It seems that it is not possible to close the central ring of (II) by an —O1—C1— bridge [the C4···C6 and C3···C7 distances in (III) are 4·637 (3) and 3·640 (3) Å respectively].

The molecule lies on a special position: a crystallographic twofold axis passes through the centre of the heterocyclic ring. The 18-membered heterocyclic ring has a 'saddle'-like conformation forced by two diimino fragments and four fused benzene rings. The molecule as a whole has the same conformation, but even more pronounced with the benzene rings lengthening the form of a 'saddle' (see Fig. 1). The diimide systems have noteworthy geometry. This is a significant distortion from linearity: the valence angle at C5 is 168·0 (2)° and the C=N distance is 1·214 (2) Å.

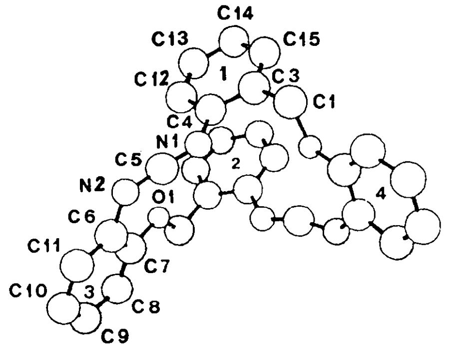

Fig. 1. The molecular structure showing the atom- and ring-numbering scheme.

It is remarkable that benzene rings 1 and 2 are nearly parallel to each other; the least-squares planes form an angle of 0·21 (7)°. The dihedral angle between benzene-ring planes 3 and 4 is 78·13 (5)°, and those between planes 1 and 3 and 1 and 4 are 108·24 (6) and 108·10 (6)°, respectively.

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Structure of *N*-(2-Phenylethenyl)pyrrolidin-2-one

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Abstract. $C_{12}H_{13}NO$, $M_r = 187·2$, orthorhombic, *Pbca*, $a = 10·896$ (3), $b = 8·033$ (2), $c = 23·376$ (8) Å, $V = 2046$ (1) Å^3, $Z = 8$, $D_x = 1·216$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·072$ mm^{-1}, $F(000) = 800$, room temperature, final $R = 0·058$ for 1290 observed reflections. The ethylene group of the title compound has a *trans* configuration and is antiperiplanar to the amide system of the lactam ring. The five-membered lactam ring is nearly planar with a mean deviation of 0·005 Å from the plane. The crystal structure is stabilized by weak intermolecular H···O interactions between the vinylic β-H atom and the carbonyl O atom of the next molecule, with a distance of 2·59 Å.

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