Tabelle 2. *Wasserstoffbrücken*

	Zu	Transl.	Symm.	Länge (Å)	Winkel (°)
O1	HN3*B*	1/0/0	1	2·53	117·3
O2	HN2*A*	0/0/0	1	2·06	178·6
O2	HN1*A*	1/0/0	3	2·17	141·9
O2	HN1*B*	0/0/0	1	2·46	132·9
O5	HO1	1/−1/0	3	1·57	178·2
O5	HN4*B*	0/0/1	2	2·48	64·5
O5	HN3*A*	0/0/1	2	2·59	60·0

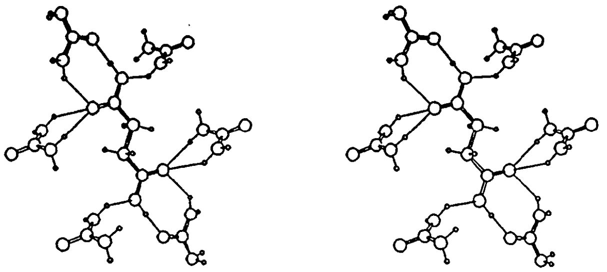

Fig. 3. Ausschnitt der Struktur als stereoskopische Abbildung (Hundt, 1987).

Anordnung, die sich durch mehrfache Quervernetzung auszeichnet. Einen Ausschnitt dieser Struktur ist in Fig. 3 als stereoskopische Abbildung (Hundt, 1987) wiedergegeben. Die Bernsteinsäure selber liegt in der erwarteten planaren Anordnung vor (Torsionswinkel alle *ca* 180°). Die Sekundärstruktur des Komplexes unterscheidet sich von den erwähnten Oxalsäure–Harnstoff Komplexen dadurch, daß in den letztgenannten Fällen kein schraubiger sondern schichtartiger Aufbau resultiert. Der Typ der H-Brücken ist jedoch in allen Fällen gleich, wobei solche von den NH-Atomen zu den Sauerstoffatomen der Säure sowie eine, die vom Carboxylproton zur C═O-Funktion des Harnstoffes reicht, anzutreffen sind. Im vorliegenden Fall liegen die Brückenlängen für die N—H···O-Brücken im Normalbereich (2·06–2·59 Å), während sich die O—H···O-Brücke durch einen extrem kurzen Abstand (1·57 Å), was eine sehr hohe Bindungsstabilität zur Folge hat, auszeichnet. Diese Stabilität dieser H-Brücke wird ebenfalls durch die stereochemische Anordnung unterstützt, da diese Brücke die energetisch günstige (stabilste) planare Anordung (O—H···O-Winkel 178·2°) einnimmt.

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Structure of (*E*,*E*)-Bis(benzylidene)-*N*-phenylsuccinimide

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Abstract. $C_{24}H_{17}NO_2$, $M_r = 351·4$, monoclinic, $C2/c$, $a = 16·047\,(1)$, $b = 14·470\,(2)$, $c = 9·046\,(3)$ Å, $\beta = 121·48\,(1)°$, $V = 1791·4$ Å^3, $Z = 4$, $D_x = 1·3$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71093$ Å, $\mu = 0·5$ cm^{-1}, $F(000) = 736$, $T = 298$ K, $R = 0·053$ for 2098 observed reflections. The dihedral angle between the *N*-phenyl ring and the plane of the succinimide ring is 44·5°. To relieve some of the steric strain imposed on the *E*,*E* configuration due to the eclipsing of the aryl rings, the exocyclic angles C(3′)—C(3)—C(5) and C(3)—C(5)—C(6) are increased to 136·1 and 129·7°, respectively. The aryl rings C(6) to C(11) and C(6′) to C(11′) have their plane normals inclined at an angle of 35°.

Introduction. The title compound 1(*a*) was synthesized as described by Crescente, Heller & Oliver (1979) and is of interest to us in our study of photochromic organic compounds. 1(*a*) undergoes photochemical *cis–trans* isomerization, thermal disrotatory and photochemical conrotatory ring-closure reactions to yield coloured 1,8a-dihydronaphthalene

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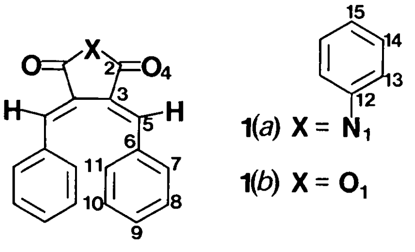
derivatives, which in turn undergo disrotatory ring-opening and 1,5-hydrogen shifts in competing thermal processes (Crescente *et al.*, 1979). To date few crystallographic data are available for these aryl-fulgide systems, and only *E,E*-diarylsuccinic anhydride systems and their geometrical and cyclic isomers have been investigated by X-ray diffraction analysis (Cohen, Kaufman, Sinnreich & Schmidt, 1970; Boeyens, Denner & Perold, 1988; Davidse, van Rooyen, Dillen, Heyns & Modro, 1989).



Converting the anhydride into the *N*-phenylimide does not, however, introduce any significant changes into the 1,4-bis(benzylidene)-1,3-butadiene system. As in the case of the *E,E*-diarylsuccinic anhydride systems, severe steric strain is introduced into the molecule of 1(*a*) due to the eclipsing of the aryl rings. Some cyclic imides, including *N*-phenylsuccinimides, show biological activity and have been investigated by X-ray analysis (Taira, Takayama & Terada, 1988). The change of the torsion angle between the benzene and the succinimide rings by introduction of substituents onto the benzene ring has been related to the biological activity of these compounds. Crystal structures of other cyclic *N*-phenylimides have also been reported, but without consideration of the biological activity in relation to molecular geometry (Wong & Watkins, 1973; Barassin, 1963; Kirino, Takayama, Fujinami, Yanagi & Minobe, 1984; Hambley, Newsom & Sternhell, 1985).

Experimental. Regular-shaped crystals, *ca* 0·54 × 0·54 × 0·65 mm, used for data collection. Intensities measured at 298 K; Enraf–Nonius CAD-4 diffractometer; graphite-monochromated Mo $K\alpha$ radiation; $\omega{:}2\theta$ scan; variable scan speed with a maximum of 5·49° min^{-1}, and a minimum corresponding to a measuring time of 100 s per reflection; ω-scan angle changed as $(0{\cdot}60 + 0{\cdot}35\tan\theta)°$, horizontal aperture as $(2{\cdot}2 + 0{\cdot}1\tan\theta)$ mm, limited to 1·3–5·9 mm; vertical slit fixed to 4 mm. Unit-cell parameters determined with 25 high-order reflections, $8 < \theta < 17°$; intensity control every hour, no significant loss. Monoclinic data set collected up to $(\sin\theta)/\lambda = 0{\cdot}705$ Å^{-1} ($h = -22{:}22$, $k = 0{:}20$, $l = 0{:}12$) resulting in 2854 reflections of which 583 $[2I < \sigma(I)]$ were unobserved. Data corrected for Lorentz and polarization effects only. Space group *C*2/*c* selected from systematically absent reflections and from statistical distributions of functions of the normalized structure factors. The structure was solved by direct methods (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All H atoms were located from subsequent Fourier maps. Non-H atoms were refined anisotropically, H atoms with a common isotropic temperature factor, $U = 0{\cdot}078\,(2)$ Å^2; *SHELX76* (Sheldrick, 1976); full-matrix method (on F) with $1/\sigma^2(F)$ weights; 2098 reflections used for 150 parameters; $R_{\rm int} = 0{\cdot}0113$; $(\Delta/\sigma)_{\rm max} = 0{\cdot}02$; $\Delta\rho_{\rm max} = 0{\cdot}18$, $\Delta\rho_{\rm min} = -0{\cdot}16$ e Å^{-3}; $R = 0{\cdot}0533$; $wR = 0{\cdot}0434$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Final fractional coordinates with equivalent isotropic temperature factors are given in Table 1;[*] bond lengths and bond angles in Table 2.

Discussion. Fig. 1 shows the crystallographically imposed twofold rotational symmetry around the N(1)—C(12) bond with atoms C(15), C(12) and N(1) lying on the rotation axes. In the benzene ring attached to N(1) the bond lengths between the C atoms and the internal angles are similar, being very close to the corresponding standard values.

The features of the C—N bonds in 1(*a*) are similar to those of the *N*-phenylsuccinimide derivatives (Taira *et al.*, 1988; Wong & Watkins, 1973; Hambley *et al.*, 1985). The sum of the bond angles around all the C atoms and the N(1) atom in 1(*a*) vary between 359·5 and 360·0° indicating, as in the case of the anhydride 1(*b*) (Davidse *et al.*, 1989), that these atoms all assume planar sp^2 hybridization. The N(1)—C(12) bond length (1·432 Å) is comparable to the mean length (1·429 Å) of the N(1)—C(12) bond in the earlier mentioned, related succinimides (Taira *et al.*, 1988). This is shorter than a normal C—N bond, and longer than a normal C═N bond. From comparison of this bond length with the bond lengths of a conjugated N—C(α) bond (1·45 Å) in peptide linkage (Marsh & Donohue, 1967), the C—N bond (1·412–1·483 Å) in aniline derivatives (Van Bellingen, Germain, Piret & Van Meerssche, 1971; Hulme & Scruton, 1968; Berndt & Schlemper, 1982), and the two C—N bonds (1·378–1·433 Å) in 1,3-diphenylspiro[imidazolidine-2,2-indan]-1,3-dione (Sheldrick, Schonberg & Singer, 1982), Taira *et al.* (1988) concluded that this C—N bond shows partial double-bond character, which suggests that the N(1)—C(12) bond participates in the conjugated

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[*] Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52548 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates* ($\times 10^4$) *and equivalent isotropic thermal factors* ($\times 10^3$ Å^2)

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

	x	y	z	U_{eq}
N(1)	5000	−219 (1)	7500	50 (1)
C(2)	4393 (1)	318 (1)	7824 (2)	50 (1)
C(3)	4564 (1)	1303 (1)	7553 (2)	47 (1)
O(4)	3836 (1)	20 (1)	8223 (2)	66 (1)
C(5)	3853 (1)	1914 (1)	7230 (2)	50 (1)
C(6)	3714 (1)	2863 (1)	6591 (2)	48 (1)
C(7)	3130 (1)	3455 (1)	6881 (2)	57 (1)
C(8)	2978 (1)	4364 (1)	6291 (2)	64 (1)
C(9)	3381 (1)	4667 (1)	5374 (2)	66 (1)
C(10)	3928 (1)	4087 (1)	5012 (2)	64 (1)
C(11)	4092 (1)	3181 (1)	5609 (2)	56 (1)
C(12)	5000	−1208 (2)	7500	54 (1)
C(13)	4130 (1)	−1680 (1)	6619 (3)	71 (1)
C(14)	4140 (2)	−2636 (1)	6639 (3)	83 (1)
C(15)	5000	−3106 (2)	7500	83 (1)

Table 2. *Bond lengths* (Å) *and valence angles* (°)

N(1)—C(2)	1·392 (2)	N(1)—C(12)	1·432 (2)
C(2)—C(3)	1·496 (2)	C(2)—O(4)	1·203 (2)
C(3)—C(5)	1·349 (2)	C(3)—C (3′)	1·452 (3)
C(5)—C(6)	1·460 (2)	C(6)—C(7)	1·392 (2)
C(6)—C(11)	1·390 (2)	C(7)—C(8)	1·392 (2)
C(8)—C(9)	1·362 (3)	C(9)—C(10)	1·372 (2)
C(10)—C(11)	1·390 (2)	C(12)—C(13)	1·374 (2)
C(13)—C(14)	1·383 (2)	C(14)—C(15)	1·360 (2)
C(2)—N(1)—C(12)	123·9 (1)	N(1)—C(2)—C(3)	106·7 (1)
C(2)—N(1)—C(2′)	112·2 (1)	C(3)—C(2)—O(4)	128·2 (1)
N(1)—C(2)—O(4)	125·1 (1)	C(2)—C(3)—C(3′)	106·1 (1)
C(2)—C(3)—C(5)	117·0 (1)	C(3)—C(5)—C(6)	129·7 (1)
C(3′)—C(3)—C(5)	136·1 (1)	C(5)—C(6)—C(11)	122·5 (1)
C(5)—C(6)—C(7)	118·9 (1)	C(6)—C(7)—C(8)	120·6 (2)
C(7)—C(6)—C(11)	118·6 (2)	C(8)—C(9)—C(10)	121·0 (2)
C(7)—C(8)—C(9)	119·6 (2)	C(6)—C(11)—C(10)	120·2 (2)
C(9)—C(10)—C(11)	119·9 (2)	C(12)—C(13)—C(14)	119·2 (2)
N(1)—C(12)—C(13)	119·8 (1)	C(13)—C(12)—C(13′)	120·4 (2)
C(13)—C(14)—C(15)	120·6 (2)		

system of the benzene ring. As in the succinimides, the N(1)—C(2) bond (1·392 Å) is longer than the corresponding C—N bond (1·32 Å), and the C(2)—O(4) double bond (1·204 Å) is shorter than the corresponding C=O bond (1·24 Å) in a peptide linkage (Marsh & Donohue, 1967).

Based on the above results (Taira *et al.*, 1988), it was suggested that the O(4′)—C(2′)—N(1)—C(2)—O(4) bond system in the succinimide ring is not fully conjugated and is not conjugated with the benzene ring bonded to the N(1) atom. The torsion angles for the moieties O(4′)—C(2′)—N(1)—C(2) and O(4)—C(2)—N(1)—C(2′) are −176·16 and 176·16° respectively. Although the N(1)—C(2) bond length (1·392 Å) and the O(1)—C(2) bond length (1·395 Å) in the anhydride 1(*b*) are almost identical, the shorter C(2)—O(4) bond length (1·184 Å) in 1(*b*) suggests a greater amount of conjugation in the anhydride O(4′)—C(2′)—O(1)—C(2)—O(4) bond system, possibly due to the extra pair of nonbonding electrons on the sp^3 oxygen O(1).

The shorter bond length (1·452 Å) for the sp^2-hybridized C(3′)—C(3) single bond in 1(*a*) and 1(*b*), in comparison to the mean bond length (1·524 Å) for the C(3′)—C(3) bond in *N*-phenylsuccinimides, indicates the expected partial double-bond character, although the torsion angle C(5′)—C(3′)—C(3)—C(5) (37·9°) would tend to reduce this effect. The exocyclic bond angles C(3′)—C(3)—C(5) and C(3)—C(5)—C(6) are increased to 136·1 and 129·7° respectively, to relieve some of the steric strain imposed on the *E*, *E* configuration of 1(*a*) due to the eclipsing of the aryl rings.

The aryl rings C(6) to C(11) and C(6′) to C(11′) in 1(*a*), having their plane normals inclined at an angle of 34°, show almost no deviation from the anhydride structure, 1(*b*), in which the plane normals are inclined at an angle of 35°. In other related anhydride systems, angles of 19° for (*E*,*E*)-bis(*p*-anisylidene)succinic anhydride (Cohen *et al.*, 1970) and 50° for (*E*,*E*)-bis(3,4-dimethoxybenzylidene)succinic anhydride (Boeyens *et al.*, 1988) have been reported. In the case of 1(*a*), the succinimide ring deviates only slightly from planarity and the exocyclic atoms O(4′) and O(4) deviate 0·17 and −0·17 Å from the calculated plane respectively.

An interesting difference between 1(*a*) and other *N*-phenylsuccinimides is the deviation from the previously reported values for the dihedral angle between the *N*-phenyl ring [C(12)—C(13)—C(14)—C(15)—C(14′)—C(13′)] and the plane of the five-

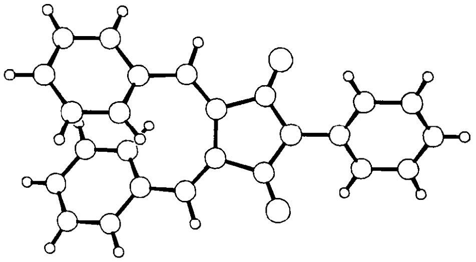

Fig. 1. Perspective drawing of (*E*,*E*)-bis(benzylidene)-*N*-phenylsuccinimide.

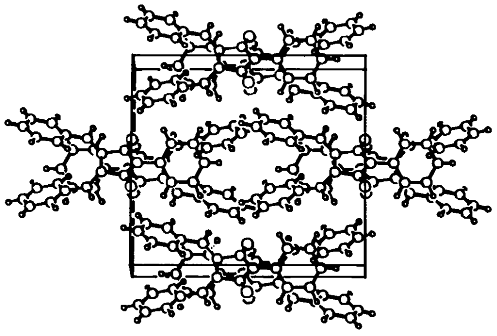

Fig. 2. Crystal packing and unit cell of the title compound viewed down the *c* axis.

membered succinimide ring [N(1)—C(2)—C(3)—C(3′)—C(2′)]. This angle varies between 55 and 71° (Taira *et al.*, 1988; Wong & Watkins, 1973; Hambley *et al.*, 1985) compared to 44·5° for the title compound. As the relation between the structure in the crystalline state and the stable molecular conformation determined by molecular-orbital calculations has not been determined for 1(*a*) as in the case of other *N*-phenylsuccinimides, it is not clear whether this reduction is due to crystal-packing effects or molecular-electronic effects. The *N*-phenyl ring adopts a perpendicular orientation relative to the neighbouring phenyl rings in the 1,4-bis(benzylidene)-1,3-butadiene fragment (Fig. 2). The shortest distance between two neighbouring non-H atoms is $C(2)\cdots O(4)(1-x,-y,2-z) = 3{\cdot}239$ Å.

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rel-(1*R*,2*S*,3*R*,4*S*,5*S*,6*R*,7*S*,8*R*)-2,3,6,7-Tetrakis(chloromethyl)-1,4:5,8-diepoxy-1,2,3,4,5,6,7,8-octahydroanthracene

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Abstract. $C_{18}H_{18}Cl_4O_2$, $M_r = 408{\cdot}2$, triclinic, $P\overline{1}$, $a = 6{\cdot}884\,(2)$, $b = 7{\cdot}782\,(2)$, $c = 9{\cdot}216\,(2)$ Å, $\alpha = 101{\cdot}32\,(2)$, $\beta = 93{\cdot}03\,(2)$, $\gamma = 115{\cdot}24\,(2)^\circ$, $V = 433$ Å^3, $Z = 1$ (the molecule possesses a centre of symmetry), $D_x = 1{\cdot}57$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}54178$ Å, $\mu = 6{\cdot}43$ mm^{-1}, $F(000) = 210$, room temperature, $R = 0{\cdot}040$ for 1144 observed reflections with $|F_o| > 3\sigma(|F_o|)$. X-ray crystallography has shown that the more chromatographically mobile and higher-melting isomer – the title compound (4) – with the 2,3,6,7-tetrakis(chloromethyl)-1,4:5,8-diepoxy-1,2,3,4,5,6,7,8-octahydroanthracene constitution obtained from reaction of *cis*-1,4-dichlorobut-2-ene with the benzo[1,2-*c*:4,5-*c*′]difuran equivalent, generated *in situ* by the thermolytic decomposition of a bisadduct of 1,4:5,8-diepoxy-1,4,5,8-tetrahydroanthracene and tetraphenylcyclone, has its two O atoms oriented *anti* to each other, whilst its four chloromethyl groups all have the *endo* configuration.

Introduction. Our current interest (Kohnke, Slawin, Stoddart & Williams, 1987; Ellwood, Mathias, Stoddart & Kohnke, 1988; Stoddart, 1988*a*,*b*, 1989;

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