

Le Tableau 5 donne la liste des liaisons inférieures à 3,5 Å. Dans la structure TeBr, il y a deux liaisons NH...O avec l'oxygène du diméthylsulfoxyde. Les Figs. 3 et 4 montrent l'empilement, respectivement dans TeCl et dans TeBr, suivant l'axe *c*.

Les auteurs remercient MM les Professeurs J. Toussaint et M. Renson pour l'intérêt qu'ils ont porté à ce travail.

Références

- BAIWIR, M., LLABRÈS, G., DENOËL, J. & PIETTE, J. L. (1973). *Mol. Phys.* **25**, 1–7.
- BAIWIR, M., LLABRÈS, G., DIDEBERG, O., DUPONT, L. & PIETTE, J. L. (1974). *Acta Cryst.* **B30**, 139–143.
- CRUICKSHANK, D. W. J. (1960). *Computing Methods and the Phase Problem in X-ray Crystal Analysis*, édité par R. PEPINSKY, J. M. ROBERTSON & J. C. SPEAKMAN. Oxford: Pergamon Press.
- HERBSTEIN, F. H., KAPON, M. & WASSERMAN, S. (1978). *Acta Cryst.* **B34**, 1613–1617.
- MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1977). *MULTAN 77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. de York, Angleterre, et Louvain-la-Neuve, Belgique.
- PREWITT, C. T. (1967). *SFLS*. State Univ. of New York, Stony Brook, Long Island, New York 11794, USA.
- TALBOT, J. M., PIETTE, J. L. & RENSON, M. (1976). *Bull. Soc. Chim. Fr.* pp. 294–296.

Acta Cryst. (1979). **B35**, 852–856

The Molecular and Crystal Structures of 2-(1-Naphthyl)-1,3-indandione and 2-(2-Naphthyl)-1,3-indandione

BY INGBORG CSÖREGH

Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-106 91 Stockholm, Sweden

AND MARIAN ECKSTEIN

Department of the Chemical Technology of Drugs, N. Copernicus Academy of Medicine, ul. Skaleczna 10, 31-065 Kraków, Poland

(Received 26 October 1978; accepted 15 December 1978)

Abstract

The crystal structures of the 2-(1-naphthyl) and 2-(2-naphthyl) derivatives of 1,3-indandione, C₁₉H₁₂O₂, were determined from three-dimensional X-ray diffractometer data. Both unit cells are monoclinic, space group *P*2₁/*c*, with *a* = 10.792 (1), *b* = 14.305 (2), *c* = 8.700 (2) Å and β = 101.96 (2)° for the 1-naphthyl derivative and *a* = 5.387 (3), *b* = 8.818 (4), *c* = 28.115 (28) Å and β = 94.20 (5)° for the 2-naphthyl derivative. The structures were solved by direct methods and refined by least squares to final linear *R* values of 0.045 (1208 reflexions) for the 1-naphthyl derivative and 0.043 (1177 reflexions) for the 2-naphthyl derivative. Both compounds crystallize in the diketone form. The crystal structures are held together by van der Waals forces.

0567-7408/79/040852-05\$01.00

Introduction

The 2-aryl-1,3-indandiones exhibit antithrombotic activity with varying efficiency. 2-(1-Naphthyl)-1,3-indandione is a strong oral anticoagulant with a mode of action and potency comparable with that of dicoumarol (Molho, 1955). However, its 2-isomer is practically inactive (Sterkowicz, Guminska & Eckstein, 1972 and references therein). A similar difference exists between the 1- and 2-naphthyl derivatives of 4-hydroxycoumarin (Morau, 1956) and also between the corresponding derivatives of dicoumarol (Guminska & Eckstein, 1961). The present X-ray structure analysis of the 1- and 2-naphthyl isomers of 1,3-indandione was performed as part of a research program concerning the relationship between anti-clotting activity and steric conformation.

© 1979 International Union of Crystallography

Experimental

The title compounds were prepared at the Department of Technology of Drugs in Kraków (Eckstein & Kocwa, 1954). Suitable single crystals were grown from methanol solutions by slow evaporation at room temperature. Preliminary investigations by X-ray photographic methods (oscillation and de Jong-Bouman techniques) showed the crystals of both compounds to be monoclinic. The systematic absences were consistent with the space group $P2_1/c$. The unit-cell dimensions were refined by least-squares fitting of the preliminary cell parameters to powder data from a Guinier photograph, taken at 298 K with strictly monochromatized Cu $K\alpha_1$ radiation ($\lambda = 1.54056 \text{ \AA}$) and KCl ($a = 6.2930 \text{ \AA}$) as an internal standard. Three-dimensional intensity data were collected by a computer-controlled four-circle diffractometer to a θ limit of 65° , with graphite-monochromatized Cu $K\alpha$ radiation. The intensities of 2370 reflexions were measured for the 1-naphthyl derivative from a single crystal of irregular shape with approximate dimensions $0.03 \times 0.8 \times 0.5 \text{ mm}$. 2331 independent reflexions were collected from a 2-(2-naphthyl)-1,3-indandione crystal with approximate dimensions $0.4 \times 0.1 \times 0.1 \text{ mm}$. The net intensities were corrected for Lorentz and polarization effects, but the rather low absorption effects ($\mu = 0.65\text{--}0.67 \text{ mm}^{-1}$) were neglected.

Structure determination and refinement

Preliminary structure-factor phases were derived by direct methods. 350 $|E|$ values and the best 2000 triple relations among them were used in both cases in the *MULTAN* program system (Main, Lessinger, Woolfson, Germain & Declercq, 1974) to generate 32 trial phase sets for the 1-naphthyl derivative and eight trial phase sets for the 2-naphthyl derivative. The E maps calculated from the solutions with the best figures of merit showed the positions of all the 21 non-hydrogen atoms for both structures. The hydrogen atoms were located from difference electron-density calculations. The structural models were refined by a full-matrix least-squares procedure in which only the most significant observations, with $I_{net} > 3\sigma(I_{net})$, were used. The calculation of $\sigma(I_{net})$ was based on conventional counter statistics. In the final refinement all carbon and oxygen atoms were allowed to vibrate anisotropically, while the hydrogen atoms were refined with isotropic temperature factors. The unweighted final reliability index R was 0.045 for the 1-naphthyl derivative (1208 reflexions) and 0.043 for 2-(2-naphthyl)-1,3-indandione (1177 reflexions). The atomic scattering factors were those given in *International*

Tables for X-ray Crystallography (1974). The fractional atomic coordinates are listed in Tables 1 and 2.*

* Lists of structure factors, anisotropic thermal parameters and bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34157 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ($\times 10^4$) for carbon and oxygen atoms with estimated standard deviations in parentheses

2-(1-Naphthyl)-1,3-indandione			
	x	y	z
O(1)	8756 (3)	7963 (2)	6066 (4)
O(2)	8831 (3)	4667 (2)	5971 (4)
C(1)	9089 (4)	7156 (2)	6284 (5)
C(2)	8473 (3)	6316 (3)	5309 (4)
C(3)	9102 (4)	5482 (3)	6258 (5)
C(4)	10965 (5)	5339 (3)	8683 (6)
C(5)	11790 (5)	5837 (3)	9779 (6)
C(6)	11805 (5)	6817 (3)	9759 (6)
C(7)	10976 (5)	7309 (3)	8630 (6)
C(8)	10126 (4)	6810 (3)	7540 (5)
C(9)	10111 (4)	5831 (3)	7542 (5)
C(10)	7050 (4)	6294 (3)	4812 (4)
C(11)	6495 (5)	6297 (4)	3260 (5)
C(12)	5162 (5)	6248 (5)	2740 (6)
C(13)	4405 (5)	6207 (3)	3785 (5)
C(14)	4930 (4)	6216 (3)	5413 (5)
C(15)	4150 (4)	6167 (3)	6513 (6)
C(16)	4663 (5)	6184 (5)	8063 (7)
C(17)	5969 (5)	6259 (5)	8606 (6)
C(18)	6759 (4)	6293 (3)	7581 (5)
C(19)	6264 (3)	6269 (2)	5934 (4)
2-(2-Naphthyl)-1,3-indandione			
	x	y	z
O(1)	504 (5)	1294 (4)	9272 (1)
O(2)	7345 (5)	4610 (4)	9209 (1)
C(1)	2250 (7)	2122 (5)	9386 (2)
C(2)	3301 (7)	3346 (5)	9071 (2)
C(3)	5749 (7)	3801 (5)	9352 (2)
C(4)	7520 (8)	3252 (6)	10213 (2)
C(5)	7098 (9)	2469 (6)	10625 (2)
C(6)	5029 (10)	1543 (6)	10651 (2)
C(7)	3324 (8)	1346 (5)	10266 (2)
C(8)	3729 (7)	2138 (4)	9850 (2)
C(9)	5779 (7)	3086 (4)	9827 (2)
C(10)	5492 (8)	1586 (6)	7952 (2)
C(11)	5377 (8)	1978 (5)	8418 (2)
C(12)	3445 (7)	2928 (5)	8552 (2)
C(13)	1684 (7)	3441 (5)	8217 (2)
C(14)	1777 (7)	3063 (5)	7729 (2)
C(15)	-3 (9)	3579 (6)	7373 (2)
C(16)	176 (10)	3206 (7)	6907 (2)
C(17)	2088 (10)	2254 (6)	6775 (2)
C(18)	3818 (10)	1722 (7)	7109 (2)
C(19)	3717 (7)	2111 (5)	7596 (2)

Results and discussion

The molecules of the 2-(1-naphthyl) and 2-(2-naphthyl) derivatives of 1,3-indandione are displayed in Fig. 1(a) and (b). They crystallize in the diketone form. The corresponding bond lengths and bond angles in these

Table 2. Fractional atomic coordinates ($\times 10^3$) for the hydrogen atoms with estimated standard deviations in parentheses

2-(1-Naphthyl)-1,3-indandione			
	x	y	z
H(2)	884 (3)	632 (3)	437 (4)
H(4)	1089 (5)	467 (3)	873 (5)
H(5)	1235 (4)	550 (3)	1060 (5)
H(6)	1246 (5)	713 (3)	1053 (5)
H(7)	1094 (5)	799 (3)	859 (5)
H(11)	705 (5)	632 (4)	249 (6)
H(12)	479 (6)	628 (5)	161 (8)
H(13)	348 (4)	619 (3)	345 (4)
H(15)	323 (4)	612 (3)	601 (4)
H(16)	412 (6)	621 (4)	883 (7)
H(17)	634 (4)	620 (3)	975 (5)
H(18)	764 (3)	633 (3)	798 (4)
2-(2-Naphthyl)-1,3-indandione			
	x	y	z
H(2)	218 (6)	423 (4)	908 (1)
H(4)	901 (8)	386 (5)	1020 (2)
H(5)	831 (9)	261 (6)	1089 (2)
H(6)	484 (8)	94 (5)	1095 (2)
H(7)	186 (7)	66 (5)	1028 (2)
H(10)	679 (8)	96 (5)	785 (2)
H(11)	680 (8)	156 (5)	867 (2)
H(13)	40 (7)	412 (5)	829 (2)
H(15)	-137 (8)	417 (5)	747 (2)
H(16)	-105 (8)	360 (5)	667 (2)
H(17)	221 (8)	195 (5)	644 (2)
H(18)	511 (9)	108 (6)	704 (2)

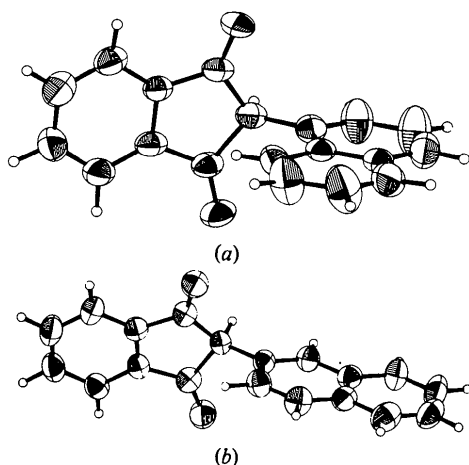


Fig. 1. Perspective drawings of the molecules of (a) 2-(1-naphthyl)-1,3-indandione and (b) 2-(2-naphthyl)-1,3-indandione. The carbon and oxygen atoms are represented by their thermal ellipsoids, while the hydrogens are drawn as spheres.

two derivatives, shown in Figs. 2 and 3, agree within experimental uncertainty. Two endocyclic angles in the benzene rings, C(5)–C(4)–C(9) and C(6)–C(7)–C(8), are significantly smaller than 120° in both structures as is characteristic for molecules having a five-membered ring fused to the phenyl ring.

The geometry of the five-membered ring, in particular around atom C(2) (*cf.* Fig. 2), shows significant variation among 1,3-indandione derivatives. The geometries found in the present studies of the two naphthyl derivatives correspond with that found for 2-phenyl- and 2-ethyl-2-phenyl-1,3-indandiones (Bravic, Gaultier & Hauw, 1974*a,b*) and for 1,3-indandione itself (Bravic, Bechtel, Gaultier & Hauw, 1976). The C(2) atom is tetrahedrally substituted in these structures, the endocyclic C(2)–C bond lengths and C–C(2)–C bond angles are 1.51–1.54 Å and 102 – 104° , respectively. In other derivatives, *e.g.* 2-methylsulfuranylidene-1,3-indandione (Christensen & Thom, 1971), 2-nitro-1,3-indandione (Simonsen & Jacobsen, 1977), 2-pivaloyl-1,3-indandione (Csöregi & Norrestam, 1976), 2-dicyanomethylene-1,3-indandione (Silverman, Krukonis & Yannoni, 1974) and 2'-dionylidene-indane-3-indanone (Bravic, Bravic, Gaultier & Hauw, 1976), the C(2) atom is sp^2 hybridized. This results in C(2)–C bond distances ranging from 1.42 to 1.50 Å and C–C(2)–C angles from 111 down to 106° within the five-membered ring, depending on the electrophilic nature of the substituent at C(2). All the other bond lengths and angles within the indandione moiety, shown in Figs. 2 and 3, agree well with the corresponding values found in other indandione derivatives (see above references). The C–C bond-length alternation within the naphthyl substituents follows the usual scheme,

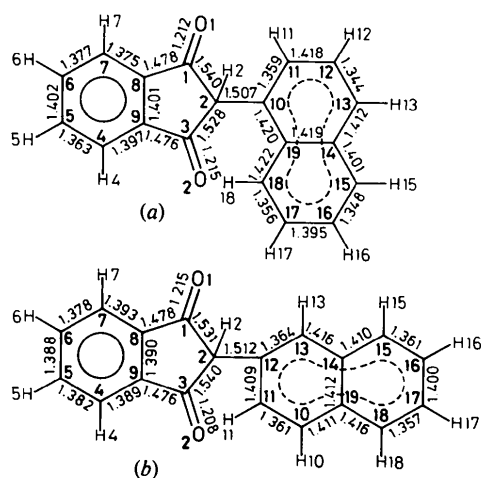


Fig. 2. Intramolecular bond distances (Å) between the non-hydrogen atoms of the (a) 2-(1-naphthyl) and (b) 2-(2-naphthyl) derivatives of 1,3-indandione. The e.s.d.'s of the bond lengths lie in the range 0.003–0.007 Å in both studies. The atoms are numbered for reference to the text.

Table 3. Equations of the least-squares planes, and deviations (Å) of the atoms from the planes

The planes are expressed as $Ax + By + Cz = D$, where x , y , and z are in Å along the axes a^* , b and c . The atoms indicated with asterisks were omitted from the calculations of the least-squares planes. A negative sign means that the atom lies between the plane and the origin.

2-(1-Naphthyl)-1,3-indandione
(1) The best LS plane through the indan rings

	Atom	Deviation	Atom	Deviation
$A = 0.650$	O(1)*	-0.187	C(5)	-0.024
$B = -0.006$	O(2)*	-0.029	C(6)	-0.007
$C = -0.760$	C(1)	-0.038	C(7)	0.025
$D = 3.600$	C(2)*	0.087	C(8)	0.022
	C(3)	0.006	C(9)	0.017
	C(4)	-0.001		

The r.m.s. deviation from the plane of the atoms without asterisks is 0.021 Å.

(2) LS plane through the naphthyl rings

	Atom	Deviation	Atom	Deviation
$A = -0.057$	C(10)	-0.025	C(15)	-0.014
$B = 0.998$	C(11)	0.003	C(16)	-0.011
$C = 0.008$	C(12)	0.012	C(17)	0.019
$D = 8.609$	C(13)	0.008	C(18)	0.011
	C(14)	0.000	C(19)	-0.004

The r.m.s. deviation from the plane of the atoms without asterisks is 0.013 Å.

2-(2-Naphthyl)-1,3-indandione
(1) The best LS plane through the indan rings

	Atom	Deviation	Atom	Deviation
$A = -0.534$	O(1)*	-0.173	C(5)	-0.010
$B = 0.766$	O(2)*	-0.055	C(6)	0.014
$C = 0.356$	C(1)	-0.025	C(7)	0.008
$D = 10.182$	C(2)*	0.170	C(8)	0.005
	C(3)	0.022	C(9)	0.005
	C(4)	-0.020		

The r.m.s. deviation from the plane of the atoms without asterisks is 0.016 Å.

(2) LS plane through the naphthyl rings

	Atom	Deviation	Atom	Deviation
$A = 0.567$	C(10)	0.003	C(15)	0.011
$B = -0.804$	C(11)	-0.005	C(16)	-0.015
$C = 0.179$	C(12)	-0.009	C(17)	-0.002
$D = 1.159$	C(13)	0.007	C(18)	0.004
	C(14)	0.001	C(19)	0.005

The r.m.s. deviation from the plane of the atoms without asterisks is 0.008 Å.

discussed by, among others, Cruickshank (1962) and Daudel (1962).

The C-H bond distances range between 0.93 and 1.07 Å, with an average value of 0.98 Å for both structures; these are normal values for C-H bond lengths determined by X-ray diffraction (Stewart, Davidson & Simpson, 1965).

Two parts of the molecules may be examined for planarity: the indandione group and the naphthyl

substituent. Deviations of the atoms from these planes are listed in Table 3. The angle between the normals of the least-squares planes is 87.1° in the 1-naphthyl derivative and 75.5° in the 2-naphthyl derivative.

The molecular packing in the two crystals is illustrated in Fig. 4. In the crystal structure of the 1-naphthyl derivative, the naphthyl rings are perpendicular to the b axis. They are stacked upon each other

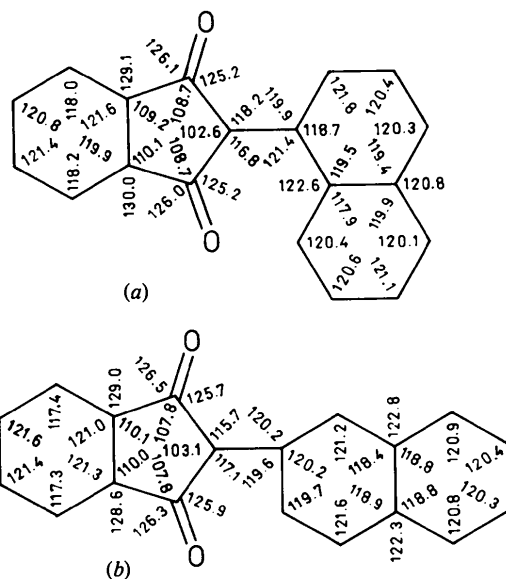


Fig. 3. Intramolecular bond angles (°) involving the non-hydrogen atoms of the (a) 2-(1-naphthyl) and (b) 2-(2-naphthyl) derivatives of 1,3-indandione. The estimated uncertainty varies from 0.2 to 0.5° in both cases.

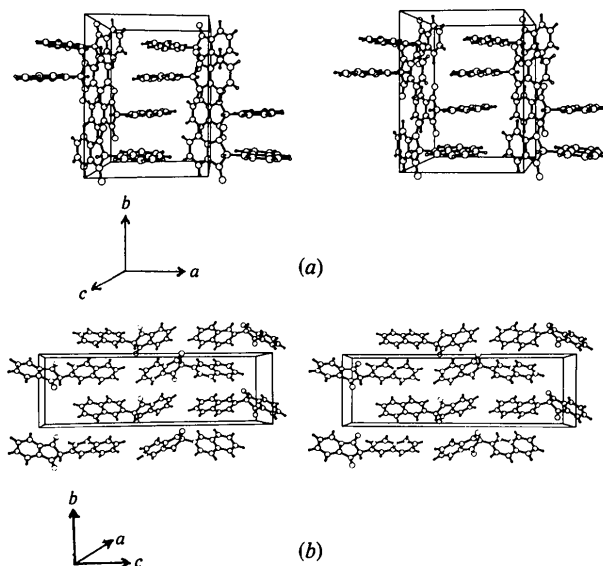


Fig. 4. Stereoscopic packing diagrams of the crystal structures of the (a) 2-(1-naphthyl) and (b) 2-(2-naphthyl) derivatives of 1,3-indandione.

Table 4. Intermolecular distances (Å) less than 3.6 Å between non-hydrogen atoms

Atom *B* is generated from the coordinates of Table 1 with the unit-cell translation shown, together with the following symmetry operations:

Atom at			Superscript
<i>x</i> , <i>y</i> , <i>z</i>			none
$-x$, $\frac{1}{2} + y$, $\frac{1}{2} - z$			(i)
$-x$, $-y$, $-z$			(ii)
x , $\frac{1}{2} - y$, $\frac{1}{2} + z$			(iii)

The estimated standard deviations are given in parentheses.

2-(1-Naphthyl)-1,3-indandione

<i>A</i>	<i>B</i>	Translation along the axes of the unit cell			Distance
		<i>a</i>	<i>b</i>	<i>c</i>	
O(2)	C(3 ⁱⁱ)	2	1	1	3.252 (5)
O(2)	C(7 ⁱ)	2	-1	1	3.393 (5)
O(1)	C(4 ⁱ)	2	0	1	3.415 (5)
O(2)	O(2 ⁱⁱ)	2	1	1	3.449 (6)
C(1)	C(7 ⁱⁱⁱ)	0	1	-1	3.466 (7)
O(1)	C(17 ⁱⁱⁱ)	0	1	-1	3.493 (6)
C(3)	C(3 ⁱⁱ)	2	1	1	3.494 (9)
O(1)	C(18 ⁱⁱⁱ)	0	1	-1	3.502 (5)
C(11)	C(18 ⁱⁱⁱ)	0	1	-1	3.519 (7)
O(1)	C(7 ⁱⁱⁱ)	0	1	-1	3.534 (6)
C(4)	C(4 ⁱⁱ)	2	1	2	3.537 (10)
O(2)	C(9 ⁱⁱ)	2	1	1	3.549 (5)
O(1)	C(11 ⁱⁱⁱ)	0	1	0	3.556 (6)
C(12)	C(16 ⁱⁱ)	1	1	1	3.561 (10)
C(14)	C(14 ⁱⁱ)	1	1	1	3.562 (9)
C(11)	C(17 ⁱⁱⁱ)	0	1	-1	3.565 (9)
C(12)	C(15 ⁱⁱ)	1	1	1	3.565 (8)
C(13)	C(14 ⁱⁱ)	1	1	1	3.579 (6)

2-(2-Naphthyl)-1,3-indandione

<i>A</i>	<i>B</i>	Translation along the axes of the unit cell			Distance
		<i>a</i>	<i>b</i>	<i>c</i>	
O(1)	O(2)	-1	0	0	3.381 (5)
C(7)	C(7 ⁱⁱ)	1	0	2	3.395 (9)
O(1)	C(3)	-1	0	0	3.403 (5)
C(3)	C(4 ⁱⁱ)	1	1	2	3.415 (7)
O(1)	C(7 ⁱⁱ)	0	0	2	3.428 (6)
O(2)	C(1)	1	0	0	3.442 (5)
O(2)	C(2)	1	0	0	3.445 (5)
O(1)	C(9)	-1	0	0	3.463 (5)
O(1)	C(6 ⁱⁱ)	1	0	2	3.467 (6)
C(7)	C(8 ⁱⁱ)	1	0	2	3.484 (6)
C(10)	C(15)	-1	0	0	3.494 (7)
C(4)	C(7)	1	0	0	3.543 (6)
C(1)	C(6 ⁱⁱ)	1	0	2	3.553 (7)
O(1)	C(5 ⁱⁱ)	1	0	2	3.565 (6)
O(2)	C(5 ⁱⁱ)	1	1	2	3.570 (6)
O(1)	C(11)	-1	0	0	3.576 (6)

with an interplanar spacing of 3.56 Å. The intermolecular distances (Table 4) are of the same magnitude as the van der Waals approach. Van der Waals interactions also play the leading part in the crystal

structure of 2-(2-naphthyl)-1,3-indandione. Remarkable in the latter case is that all the intermolecular contact distances shorter than 3.6 Å occur between molecules related by the symmetry operation *x, y, z*, or $-x, -y, -z$ (Table 4). The shortest contact distances between molecules related by the symmetry operations $-x, \frac{1}{2} + y, \frac{1}{2} - z$ (i) and $x, \frac{1}{2} - y, \frac{1}{2} + z$ (iii) are 3.648 Å [O(2)–C(17ⁱ)] and 3.789 Å [C(6)–C(17ⁱⁱⁱ)], respectively.

The authors wish to thank Professor Peder Kierkegaard for his active interest and encouragement. They also wish to thank Professor Rolf Norrestam for valuable discussions and comments on the manuscript. Thanks are further due to Docent Sven Westman for his correction of the English of this paper.

The present investigation has been performed with financial support from the Swedish Natural Science Research Council.

References

- BRAVIC, G., BECHTEL, F., GAULTIER, J. & HAUW, C. (1976). *Cryst. Struct. Commun.* **5**, 1–4.
- BRAVIC, G., BRAVIC, G., GAULTIER, J. & HAUW, C. (1976). *Cryst. Struct. Commun.* **5**, 5–8.
- BRAVIC, G., GAULTIER, J. & HAUW, C. (1974a). *Cryst. Struct. Commun.* **3**, 215–217.
- BRAVIC, G., GAULTIER, J. & HAUW, C. (1974b). *Cryst. Struct. Commun.* **3**, 219–222.
- CHRISTENSEN, A. T. & THOM, E. (1971). *Acta Cryst.* **B27**, 581–586.
- CRUICKSHANK, D. W. J. (1962). *Tetrahedron*, **17**, 155–161.
- CSÖREGH, I. & NORRESTAM, R. (1976). *Acta Cryst.* **B32**, 2450–2455.
- DAUDEL, R. (1962). *Tetrahedron*, **17**, 243–246.
- ECKSTEIN, M. & KOCWA, A. (1954). *Zesz. Probl. Nauki Pol.* **9**, 71–75.
- GUMINSKA, M. & ECKSTEIN, M. (1961). *J. Med. Pharm. Chem.* **3**, 583–595.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.
- MAIN, P., LESSINGER, L., WOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MOLHO, D. (1955). *Thrombose & Embolie*, pp. 193–199. Basel: Benno & Schwabe.
- MORAUX, J. (1956). *Thèse docteur des Sciences naturelles, Lyon*. Laboratoire de Chimie Biologique de la Faculté des Sciences, Université de Lyon, Lyon.
- SILVERMAN, J., KRUKONIS, A. P. & YANNONI, N. F. (1974). *Cryst. Struct. Commun.* **3**, 261–264.
- SIMONSEN, O. & JACOBSEN, J. P. (1977). *Acta Cryst.* **B33**, 3045–3049.
- STERKOWICZ, J., GUMINSKA, M. & ECKSTEIN, M. (1972). *Diss. Pharm. Pharmacol.* **24**, 569–576.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.