equal to 126°, contributes to the formation of a quasi-aromatic ring. This indicates sp^2 configuration of the amine N atom.

The equation of the 'best' plane for the pyridinium cation is: $7\cdot2958x - 1\cdot4437y + 6\cdot6054z - 5\cdot0191 = 0$. The deviations of the cation non-hydrogen atoms from this plane are listed in Table 3. The anion and cation planes form a dihedral angle of 25·5°.

Bond lengths in the cation (Fig. 2) are somewhat shortened with respect to those described in the literature (Serewicz, Robertson & Meyers, 1965). This shortening is probably because the correction for the librational movement of the whole cation was not applied. The anion is connected to the cation by a strong hydrogen bond N(21)—H···O(3) where the distances N(21)···O(3) and H···O(3) are 2·759 and 1·79 Å respectively and the N(21)—H···O(3) angle is 161°. Remaining intermolecular distances are greater than the sum of their respective van der Waals radii. Weak intermolecular interactions explain the fragility and softness of the crystals.

The projection of the structure along the x axis is shown in Fig. 3.

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1:1 Complex of Indan-1,2,3-trione 2-(*N*-Benzoyl-*N*-phenylhydrazone) with Indan 1,2,3-trione 2-(*N*-Phenylhydrazone)

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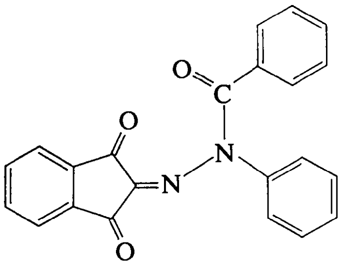
Abstract. $C_{22}H_{14}N_2O_3.C_{15}H_{10}N_2O_2$, $M_r = 604\cdot25$, orthorhombic, space group $P2_12_12_1$, $a = 12\cdot701\ (4)$, $b = 6\cdot986\ (2)$, $c = 33\cdot314\ (9)$ Å, $V = 2955\cdot9 \times 10^{-24}$ cm^3, $Z = 4$, $D_c = 1\cdot358$ g cm^{-3}, $\mu(\mathrm{Cu}\ K\alpha) = 7\cdot6$ cm^{-1}. The X-ray analysis ($R = 0\cdot053$ on 2490 reflections) shows that the complex is held together mainly by van der Waals interactions.

Introduction. In the course of studies (Puckett, Greensley, Paul & Curtin, 1977; Puckett, Paul & Curtin, 1976) on indan-1,2,3-trione 2-(*N*-benzoyl-*N*-phenylhydrazone) derivatives (1), an orange crystalline solid was obtained from indan-1,2,3-trione 2-(*N*-phenylhydrazone) (2) on treatment with benzoyl chloride. This solid was shown to be a 1:1 complex of (1) and (2) (Puckett, 1975). In order to provide more data for a systematic survey of the conformations of molecules related to (1), and to probe the types of intermolecular interactions in the complex, an X-ray structural analysis was undertaken. The cell data (given in the abstract) were obtained by a least-squares fit to the 2θ settings for ten reflections on a Picker FACS-1 diffractometer (Cu $K\alpha$, $\lambda = 1\cdot54178$ Å) at ambient room temperature. Intensity data were measured using a 2θ continuous scan method out to a maximum 2θ of 130° (Cu $K\alpha$). Out of 2927 independent reflections measured, a total of 2490 were considered significant at the $2\sigma(I)$ level; the values of $\sigma(I)$ were calculated from the equation of Corfield, Doedens & Ibers (1967). No corrections for extinction or absorption were applied. The structure was solved using the *MULTAN* direct-methods program (Germain, Main & Woolfson, 1971). All hydrogen atoms were clearly located from difference maps. The structure was refined by a full-matrix least-squares method with positional and anisotropic thermal parameters for all non-hydrogen atoms and positional and isotropic thermal parameters for the hydrogen atoms being varied. The largest peak on a

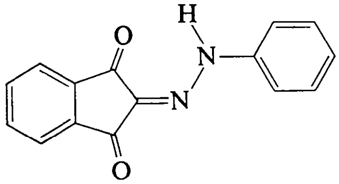
Table 1. *Final atomic coordinates*

	x	*y*	*z*
C(1)	0·7855 (3)	0·1807 (6)	0·07500 (11)
C(2)	0·8293 (5)	0·2535 (7)	0·11374 (9)
C(3)	0·9474 (3)	0·2721 (7)	0·10842 (10)
C(4)	0·9671 (3)	0·2359 (7)	0·06538 (11)
C(5)	1·0618 (4)	0·2450 (9)	0·04500 (13)
C(6)	1·0612 (5)	0·2071 (10)	0·00467 (16)
C(7)	0·9697 (5)	0·1553 (8)	−0·01500 (15)
C(8)	0·8734 (4)	0·1415 (7)	0·00552 (12)
C(9)	0·8735 (3)	0·1850 (6)	0·04590 (10)
C(10)	0·6144 (3)	0·3791 (6)	0·12517 (10)
C(11)	0·4992 (3)	0·3468 (7)	0·13187 (11)
C(12)	0·4306 (4)	0·4976 (10)	0·12431 (14)
C(13)	0·3228 (4)	0·4672 (13)	0·12775 (16)
C(14)	0·2847 (4)	0·2898 (15)	0·13703 (15)
C(15)	0·3517 (4)	0·1364 (11)	0·14416 (14)
C(16)	0·4610 (4)	0·1674 (8)	0·14150 (13)
C(17)	0·6543 (3)	0·2829 (7)	0·19516 (10)
C(18)	0·6645 (4)	0·1137 (8)	0·21544 (12)
C(19)	0·6426 (5)	0·1124 (10)	0·25671 (14)
C(20)	0·6109 (4)	0·2727 (9)	0·27612 (12)
C(21)	0·6024 (5)	0·4393 (10)	0·25586 (15)
C(22)	0·6239 (4)	0·4452 (9)	0·21478 (14)
C(1′)	0·9550 (3)	0·2950 (7)	0·35244 (11)
C(2′)	1·0587 (3)	0·2683 (6)	0·37250 (10)
C(3′)	1·1412 (3)	0·2683 (6)	0·34213 (10)
C(4′)	1·0872 (3)	0·2804 (6)	0·30259 (10)
C(5′)	1·1285 (4)	0·2769 (7)	0·26458 (10)
C(6′)	1·0602 (4)	0·2897 (7)	0·23247 (13)
C(7′)	0·9522 (4)	0·3032 (8)	0·23889 (13)
C(8′)	0·9112 (4)	0·3050 (8)	0·27614 (15)
C(9′)	0·9787 (3)	0·2947 (6)	0·30866 (11)
C(17′)	1·1690 (3)	0·1898 (6)	0·46916 (10)
C(18′)	1·0833 (4)	0·1573 (7)	0·49306 (12)
C(19′)	1·0961 (4)	0·1309 (8)	0·53360 (14)
C(20′)	1·1954 (5)	0·1410 (9)	0·55090 (14)
C(21′)	1·2812 (5)	0·1747 (9)	0·52642 (14)
C(22′)	1·2691 (4)	0·2004 (9)	0·48597 (12)
N(1)	0·7898 (2)	0·2894 (6)	0·14821 (8)
N(2)	0·6815 (2)	0·2888 (6)	0·15310 (8)
N(1′)	1·0655 (2)	0·2467 (5)	0·41174 (8)
N(2′)	1·1587 (3)	0·2182 (5)	0·42721 (8)
O(1)	0·6975 (2)	0·1167 (5)	0·06944 (8)
O(2)	1·0102 (2)	0·3123 (6)	0·13444 (8)
O(3)	0·6463 (2)	0·4765 (4)	0·09819 (8)
O(1′)	0·8692 (2)	0·3105 (6)	0·36780 (8)
O(2′)	1·2364 (2)	0·2601 (5)	0·34818 (7)
H(5)	1·129 (4)	0·299 (9)	0·0550 (16)
H(6)	1·128 (5)	0·200 (10)	−0·0200 (17)
H(7)	0·971 (3)	0·132 (6)	−0·0429 (12)
H(8)	0·804 (4)	0·081 (7)	−0·0073 (13)
H(12)	0·457 (5)	0·623 (9)	0·1234 (17)
H(13)	0·272 (6)	0·573 (12)	0·1290 (20)
H(14)	0·206 (5)	0·283 (11)	0·1343 (16)
H(15)	0·323 (4)	−0·013 (9)	0·1509 (14)
H(16)	0·506 (4)	0·040 (8)	0·1477 (14)
H(18)	0·685 (4)	0·005 (7)	0·2016 (13)
H(19)	0·648 (3)	0·009 (6)	0·2686 (12)
H(20)	0·600 (4)	0·273 (10)	0·3110 (16)
H(21)	0·577 (5)	0·530 (9)	0·2697 (16)
H(22)	0·624 (4)	0·547 (7)	0·2034 (14)
H(5′)	1·204 (3)	0·272 (6)	0·2630 (11)
H(6′)	1·089 (3)	0·285 (5)	0·2014 (10)
H(7′)	0·910 (3)	0·316 (6)	0·2068 (12)
H(8′)	0·844 (3)	0·320 (6)	0·2797 (10)
H(18′)	1·023 (3)	0·151 (6)	0·4837 (13)
H(19′)	1·038 (3)	0·100 (7)	0·5562 (12)
H(20′)	1·206 (3)	0·131 (6)	0·5794 (12)
H(21′)	1·351 (5)	0·171 (11)	0·5412 (15)
H(22′)	1·320 (3)	0·210 (7)	0·4710 (11)
H(N2′)	1·214 (3)	0·212 (7)	0·4150 (11)

final difference map was 0·25 e Å^{-3}. The final R factor on all non-zero reflections was 0·053, that for all reflections was 0·075. The value of R_w, $(\sum w\|F_o| - |F_c\|^2/\sum w|F_o|^2)^{1/2}$, was 0·051 on the non-zero reflections; the 'goodness of fit' was 2·76. The atomic scattering factors for the non-hydrogen atoms were those of Cromer & Mann (1968) and that for hydrogen was the one calculated by Stewart, Davidson & Simpson (1965). The atomic coordinates are listed in Table 1.*



(1)



(2)

Discussion. A stereoscopic view of the two molecules in the complex is shown in Fig. 1. The bond lengths and angles are listed in Table 2. These values are in general agreement with those reported previously in related compounds (Puckett, Greensley, Paul & Curtin, 1977). The results of best-plane calculations through the atoms of the five-membered rings of the indan groups and the distances therefrom of the five attached atoms for the two components of the complex are typical of similar calculations on related structures (Greensley, Curtin & Paul, 1976). In the case of (2), the carbonyl carbon atoms lie significantly to one side of the plane

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33554 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

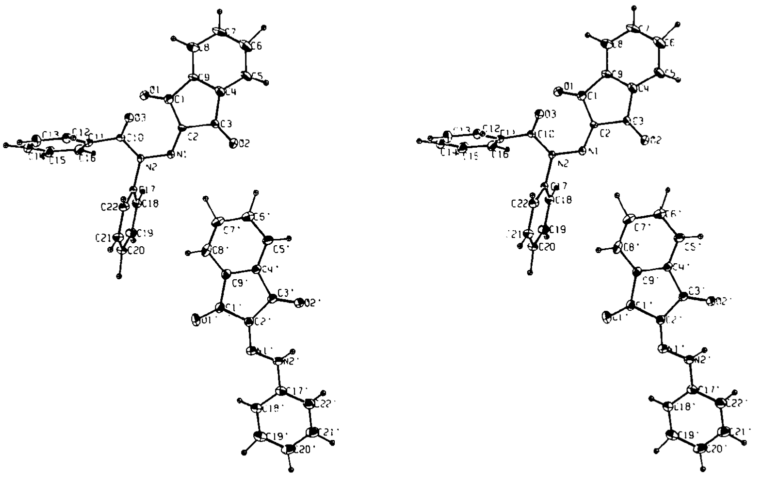

Fig. 1. Stereoscopic view of the two components of the complex.

Table 2. *Bond lengths* (Å) *and angles* (°) *in the complex*

	(1)	(2)
C(1)—C(2)	1·495 (5)	1·489 (5)
C(1)—C(9)	1·480 (5)	1·489 (5)
C(1)—O(1)	1·218 (5)	1·209 (5)
C(2)—C(3)	1·516 (7)	1·457 (5)
C(2)—N(1)	1·278 (5)	1·319 (4)
C(3)—C(4)	1·477 (5)	1·487 (5)
C(3)—O(2)	1·211 (5)	1·227 (5)
C(4)—C(5)	1·383 (6)	1·371 (5)
C(4)—C(9)	1·400 (5)	1·396 (5)
C(5)—C(6)	1·369 (7)	1·380 (6)
C(6)—C(7)	1·382 (9)	1·391 (7)
C(7)—C(8)	1·404 (8)	1·346 (7)
C(8)—C(9)	1·379 (5)	1·383 (6)
C(10)—C(11)	1·497 (5)	–
C(10)—N(2)	1·411 (5)	–
C(10)—O(3)	1·198 (5)	–
C(11)—C(12)	1·390 (8)	–
C(11)—C(16)	1·382 (7)	–
C(12)—C(13)	1·390 (7)	–
C(13)—C(14)	1·366 (13)	–
C(14)—C(15)	1·389 (11)	–
C(15)—C(16)	1·408 (7)	–
C(17)—N(2)	1·444 (4)	1·418 (4)
C(17)—C(18)	1·368 (7)	1·368 (6)
C(17)—C(22)	1·385 (5)	1·391 (6)
C(18)—C(19)	1·403 (6)	1·373 (6)
C(19)—C(20)	1·354 (9)	1·388 (8)
C(20)—C(21)	1·350 (9)	1·381 (8)
C(21)—C(22)	1·396 (7)	1·368 (6)
N(1)—N(2)	1·385 (5)	1·306 (4)
C(2)—C(1)—C(9)	106·1 (3)	105·1 (3)
C(2)—C(1)—O(1)	126·7 (4)	128·2 (4)
C(9)—C(1)—O(1)	126·9 (4)	126·7 (4)
C(3)—C(2)—N(1)	118·5 (4)	129·9 (3)
C(3)—C(2)—C(1)	107·2 (3)	108·9 (3)
N(1)—C(2)—C(1)	134·2 (4)	121·1 (3)
C(4)—C(3)—O(2)	128·5 (4)	127·0 (4)
C(4)—C(3)—C(2)	105·4 (3)	106·5 (3)
O(2)—C(3)—C(2)	126·0 (4)	126·5 (4)
C(5)—C(4)—C(9)	121·5 (4)	120·8 (4)
C(5)—C(4)—C(3)	128·0 (4)	129·8 (4)
C(9)—C(4)—C(3)	110·5 (4)	109·3 (3)
C(6)—C(5)—C(4)	117·9 (5)	118·3 (4)
C(7)—C(6)—C(5)	121·4 (6)	120·3 (4)
C(8)—C(7)—C(6)	121·3 (5)	121·6 (5)
C(9)—C(8)—C(7)	117·3 (4)	118·8 (5)
C(1)—C(9)—C(4)	110·0 (3)	110·0 (3)
C(1)—C(9)—C(8)	129·3 (4)	129·9 (4)
C(4)—C(9)—C(8)	120·6 (4)	120·1 (4)
C(11)—C(10)—N(2)	115·1 (3)	–
C(11)—C(10)—O(3)	121·9 (4)	–
N(2)—C(10)—O(3)	123·0 (3)	–
C(12)—C(11)—C(16)	120·6 (4)	–
C(10)—C(11)—C(12)	118·1 (4)	–
C(10)—C(11)—C(16)	121·0 (4)	–
C(11)—C(12)—C(13)	119·1 (5)	–
C(12)—C(13)—C(14)	120·4 (6)	–
C(13)—C(14)—C(15)	121·4 (7)	–
C(14)—C(15)—C(16)	118·3 (6)	–
C(11)—C(16)—C(15)	120·0 (5)	–
C(18)—C(17)—C(22)	120·6 (4)	120·1 (4)
C(18)—C(17)—N(2)	118·8 (4)	121·6 (4)
C(22)—C(17)—N(2)	120·6 (4)	118·3 (4)
C(17)—C(18)—C(19)	118·1 (5)	120·1 (4)
C(18)—C(19)—C(20)	121·4 (5)	120·6 (5)
C(19)—C(20)—C(21)	119·9 (5)	118·7 (5)
C(20)—C(21)—C(22)	120·0 (5)	121·0 (5)
C(17)—C(22)—C(21)	120·0 (5)	119·5 (5)
N(2)—N(1)—C(2)	119·7 (4)	117·9 (3)
C(10)—N(2)—C(17)	120·6 (3)	–
C(10)—N(2)—N(1)	121·4 (3)	119·6 (3)
C(17)—N(2)—N(1)	110·6 (3)	–

and the other three carbon atoms lie to the other side; the attached atoms lie further on the respective sides of the plane [Fig. 2(*a*)]. However, the magnitudes of the deviations are approximately symmetrical about the C=N vector. By contrast, the deviations of the five ring atoms from the corresponding best plane in (1) are much greater and the distances of the attached atoms from the plane are unsymmetrical [Fig. 2(*b*)]; this effect is almost certainly due to the intramolecular carbonyl—carbonyl (amide) interaction (Puckett, Greensley, Paul & Curtin, 1977) that is present in (1) [O(1)···O(3) 2·767 (4) Å] but not in (2).

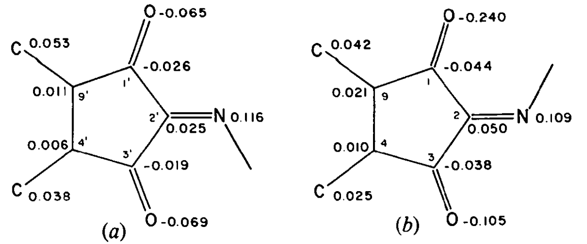

Fig. 2. (*a*) Deviations (Å) from the weighted best plane calculated through the atoms of the five-membered ring in the indan group in the molecule (2). (*b*) Deviations (Å) from the weighted best plane calculated through the atoms of the five-membered ring in the indan group in the molecule (1).

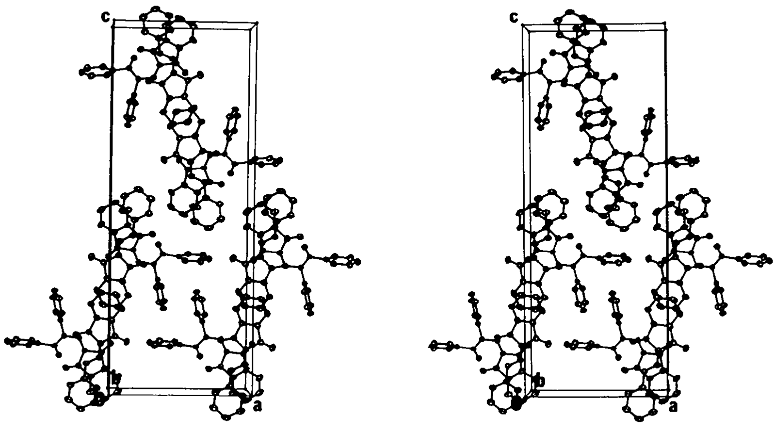

Fig. 3. Stereoscopic view of the molecular packing. The reference molecules of the complex are those furthest away at the bottom right of the figure.

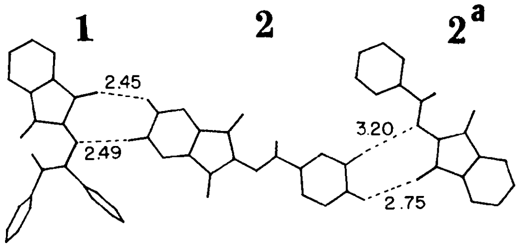

Fig. 4. Drawing showing the C—H···O and C—H···N interactions. The H···O and H···N distances are in Å. Molecule (2^a) is at $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$.

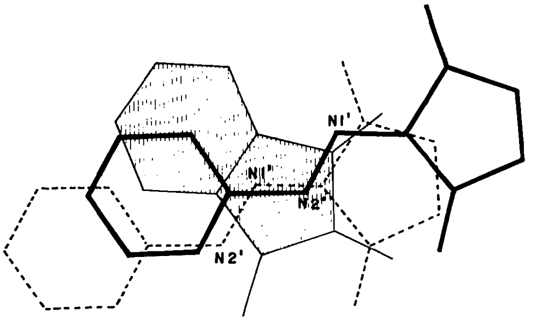

Fig. 5. Projection of part of the molecules of (2) onto the best plane (shaded) through the nine atoms of the indan group of the reference molecules of (1). The solid line depicts the molecule of (2) at $2-x, \frac{1}{2}+y, \frac{1}{2}-z$, the discontinuous line depicts the molecule of (2) at $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$.

The packing (Fig. 3) accommodates a very approximately planar molecule (deviations from the best plane range from −0·187 to +0·325 Å), the phenylhydrazone (2), and a definitely non-planar one, the benzoylphenylhydrazone (1), but one which has a large planar region. The complex may be described as a roughly bilayered structure, the layers being parallel to the *ac* plane with molecular centers [C(2) and C(2′)] in one layer at $y \simeq \frac{1}{4}$ and in the other at $y \simeq \frac{3}{4}$. The reference molecule (2) is associated with the reference molecule (1) with C—H···O and C—H···N interactions (Fig. 4) which are reminiscent of the C—H···O interactions of quinones and related molecules discussed by Bernstein, Cohen & Leiserowitz (1974). There are geometrically similar but weaker interactions between the reference molecule of (2) and the molecule of (2) at $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$. A further specific interaction which may share responsibility for complex formation between (1) and (2) is a weak hydrogen bond which is suggested by the orientation of the amide oxygen atom O(3) of the molecule (1) at $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$ toward the hydrazone hydrogen atom H(N2) of the reference molecule (2); the N(2′)···O(3) and H(N2′)···O(3) distances are 3·115 (4) and 2·46(4) Å, respectively, and the N(2′)—H(N2′)···O(3) angle is 138 (4)°. There is considerable overlap between the planar portion of (1) and the molecule (2), with the phenyl rings of (1) (roughly at right angles to the planar portion) occupying channels through the crystals in the *b* direction. The overlap of the two adjacent molecules of (2) with the planar portion of (1) is shown in Fig. 5. The atoms N(1′) and N(2′) in the molecule of (2) at $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$ lie 3·383 and 3·254 Å from the best plane through the ring atoms of the indan ring system in the reference molecule of (1), while N(1′) and N(2′) in the molecule of (2) at $2-x, \frac{1}{2}+y, \frac{1}{2}-z$ lie 3·325 and 3·450 Å from the same plane. It is possible that there may be some charge transfer in this interaction.

We thank Dr Sherrill Puckett for providing the crystals used in this study.

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Indan-1,2,3-trione 2-(*N*-*p*-*tert*-Butylbenzoyl-*N*-phenylhydrazone)

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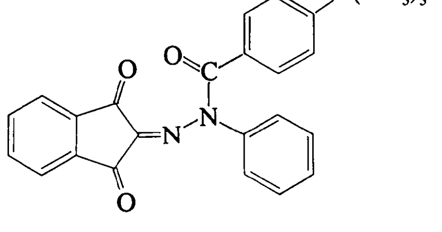
Abstract. $C_{26}H_{22}N_2O_3$, M_r = 410·5, triclinic, space group $P\bar{1}$, a = 10·076 (7), b = 11·854 (6), c = 10·463 (5) Å, α = 99·61 (4), β = 115·29 (4), γ = 87·75 (5)°, $V = 1113·4 \times 10^{-24}$ cm³, $Z = 2$, $D_c = 1·22$ g cm^{-3}, μ(Cu $K\alpha$) = 6·6 cm^{-1}. The X-ray analysis (R = 0·071 on 2935 reflections) provides the crystal structure of the form of indan-1,2,3-trione 2-(*N*-*p*-*tert*-butylbenzoyl-*N*-phenylhydrazone) that is obtained by crystallization from ethyl ether/isooctane.

Introduction. When indan-1,2,3-trione 2-(*N*-*p*-*tert*-butylbenzoyl-*N*-phenylhydrazone) (1) is crystallized from acetone, orange crystals (m.p. 160–165 °C) of a 1 : 1 acetone solvent of (1) are obtained (Puckett, 1975). The crystal structure of this acetone solvate has been determined and the course of loss of acetone from the crystals has been investigated (Puckett, Paul & Curtin, 1976). X-ray powder patterns of the material left after loss of acetone show that this red material has a different crystal structure from the red solvent-free crystals (m.p. 159–161 °C) obtained by crystallization from ethyl ether/isooctane mixtures. The present paper reports the crystal structure of the red solvent-free crystals. Cell data were obtained from a least-squares fit to the 2θ settings for thirteen reflections centered on a Syntex $P2_1$ diffractometer (λ = 1·5418 Å, ambient room temperature). Intensity data to a maximum 2θ of 130° were collected on a Syntex $P2_1$ diffractometer using a continuous 2θ-scan procedure; scan speeds ranged from 2–10° min^{-1}. A total of 2935 reflections out of a possible 3784 were considered non-zero at the 1·96σ level of significance. No corrections were applied for absorption or extinction. The structure was solved by the use of the *MULTAN* direct methods program (Germain, Main & Woolfson, 1971) that is incorporated in the Syntex *EXTL* system. Refinement was carried out by block-diagonal and finally by full-matrix least-squares methods using the programs in the *EXTL* system. All hydrogen atoms except those for the *tert*-butyl group were located from a difference map. The hydrogen atoms for the *tert*-butyl group could not be located with certainty and were not included in the model. The final values of R and R_w $[(\sum w||F_o| - |F_c||^2/\sum w|F_o|^2)^{1/2}]$ for the non-zero reflections are 0·071 and 0·099; the 'goodness of fit' is 3·00. Scattering factors were those from *International Tables for X-ray Crystallography* (1974). The final atomic coordinates are listed in Table 1.*



(1)

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33555 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.