

ring is all-*S* or all-*R*. This means that the molecule has the *meso*-(*R,R,S,S*)-configuration. The molecule $C_6H_{10}O_6S_2$ lies on a crystallographic centre of symmetry. The five-membered rings have the envelope conformation; C(3) is 0.602 Å out of the plane of the other four atoms. The perpendicular distance of the best planes through the two rings in the molecule $C_6H_{10}O_6S_2$ is 1.057 Å. The hydroxy groups in the ring are in *trans* positions.

The molecule $C_6H_{10}O_6S_2$ crystallizes with two molecules of dimethylacetamide. Sheets of the five-membered ring molecules alternate with those of dimethylacetamide, linked by hydrogen bonds [O(4)···HO(3) 2.695 Å] parallel to the *x* axis (Fig. 2).

There is another hydrogen bond between the five-membered ring molecules [O(2)H···O(3) 2.841 Å] parallel to the *y* axis. In this manner the hydrogen-bond system controls the architecture of the crystal.

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Acta Cryst. (1992). **C48**, 1269–1272

Structure of *N*-{2-[2,4-Bis(1,1-dimethylpropyl)phenoxy]ethyl}-1-hydroxy-2-naphthalenecarboxamide

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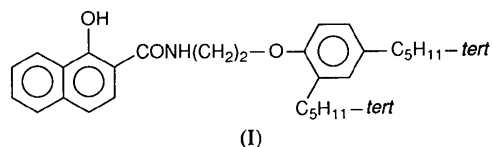
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(Received 16 May 1991; accepted 21 November 1991)

Abstract. $C_{29}H_{37}NO_3$, $M_r = 447.62$, monoclinic, $P2_1/n$, $a = 13.391$ (2), $b = 28.817$ (3), $c = 6.667$ (2) Å, $\beta = 97.256$ (17)°, $V = 2552$ (2) Å³, $Z = 4$, $D_x = 1.165$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.69$ cm⁻¹, $F(000) = 968$, $T = 295$ K, $R = 0.072$ for 2432 observed reflections [$I > \sigma(I)$]. The carbonyl O atom is intramolecularly hydrogen bonded to the hydroxyl proton. Intermolecular hydrogen bonding is absent. Disorder is evident in the *p-tert*-pentyl group. The molecules are packed in (010) sheets bounded on each surface by *tert*-pentyl groups and by naphthol groups. There is no intermolecular overlap of naphthol groups.

Introduction. The title compound (I) is a photographic coupler and reacts with oxidized color developer to yield cyan indoaniline image dyes in color films (Tong, 1977). The usual method of incorporating this compound in a sensitized film layer is to prepare a (high-vapor-pressure solvent and low-vapor-pressure plasticizer) solution of it and then to emulsify it as an oil-in-water emulsion (submicron particle sizes). After emulsification, the high-vapor-pressure solvent is removed by evapor-

ation or washing procedures. The resulting amorphous particles generally consist of coupler and plasticizer (e.g. di-*n*-butyl phthalate). Intraparticle nucleation of coupler can lead to microcrystallite formation, with dramatic effects on image dye formation kinetics. The definition of this crystalline structure (molecular packing) provides a basis for extending our understanding (Luss, Garti & Texter, 1992) of crystalline ripening and growth phenomena in photographic coupler dispersions (Sakamoto, Tanaka, Hanzawa & Kasai, 1972).



Experimental. The title compound was obtained from Mr W. F. Coffey of the Photographic Research Laboratories, Eastman Kodak Company. Large crystals were grown in saturated ethyl acetate solution at room temperature. One of these crystals was cut to a size of 0.27 × 0.28 × 0.38 mm for data col-

lection. Data (ω - 2θ scans) were collected on an Enraf-Nonius CAD-4 diffractometer using graphite-monochromated Mo $K\alpha$ radiation. A total of 3549 reflections were measured for $(\sin\theta)/\lambda < 0.55 \text{ \AA}^{-1}$. One quadrant was measured with maximum $h = \pm 12$, $k = 31$ and $l = 7$. Unit-cell parameters were refined from the setting angles after computer centering of 25 reflections ($6.5 < \theta < 12.6^\circ$). The scan range was from 2θ (Mo $K\alpha_1$) -0.65° to 2θ (Mo $K\alpha_2$) $+0.65^\circ$. Backgrounds were measured by moving-crystal moving-counter counts at each end of the scan range. The ratio of scan time to background time was 2.0, which extended the scan range to 25% at each end. To check crystal and electronic stability, three reflections were remeasured periodically.

Intensities were calculated according to $I = ASK(C - RB)$, where A is the attenuation factor, S is the scan rate, K is a scale factor, C is the total integrated peak count, R is the ratio of peak time to background time, and B is the total background count. Standard deviations were calculated according to $\sigma^2 = (ASK)^2(C + R^2B)$ and $\sigma_o = \{[I + \sigma(I)]/Lp\}^{1/2} - F_o$, where $(Lp)^{-1}$ is the Lorentz-polarization correction and $F_o = (I/Lp)^{1/2}$ is the observed structure factor. Lorentz and polarization corrections were made. Data were corrected for changes in intensity over time. The maximum correction factor on I was 1.23. Owing to the small absorption coefficient ($\mu = 0.69 \text{ cm}^{-1}$) and the uniform shape of the crystal, absorption corrections were not necessary. The structure was solved by direct methods (*MULTAN11/82*;

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2) with e.s.d.'s in parentheses

$$B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B_{eq}
O(1)	0.1812 (3)	0.1881 (1)	-0.3012 (5)	5.87 (9)
O(2)	0.0708 (2)	0.1467 (1)	-0.0790 (5)	5.34 (8)
O(3)	0.1115 (2)	0.0472 (1)	0.4252 (5)	4.14 (7)
N	0.1213 (3)	0.1358 (1)	-0.2503 (6)	4.48 (9)
C(1)	0.2531 (4)	0.1887 (2)	-0.1401 (7)	4.1 (1)
C(2)	0.3467 (4)	0.2104 (2)	-0.1665 (7)	4.1 (1)
C(3)	0.3617 (4)	0.2336 (2)	-0.3465 (8)	5.6 (1)
C(4)	0.4515 (5)	0.2543 (2)	-0.3655 (9)	6.5 (1)
C(5)	0.5304 (4)	0.2527 (2)	-0.207 (1)	6.3 (1)
C(6)	0.5174 (4)	0.2307 (2)	-0.0327 (9)	5.7 (1)
C(7)	0.4260 (4)	0.2092 (2)	-0.0063 (7)	4.4 (1)
C(8)	0.4096 (4)	0.1874 (2)	0.1768 (8)	5.6 (1)
C(9)	0.3202 (4)	0.1688 (2)	0.2009 (8)	5.2 (1)
C(10)	0.2382 (3)	0.1691 (2)	0.0424 (7)	4.0 (1)
C(11)	0.1382 (3)	0.1498 (2)	0.0666 (7)	4.0 (1)
C(12)	0.0282 (3)	0.1144 (2)	0.2918 (7)	4.6 (1)
C(13)	0.0329 (3)	0.0626 (2)	0.2757 (7)	4.2 (1)
C(14)	0.1421 (3)	0.0015 (2)	0.4231 (6)	3.46 (9)
C(15)	0.2268 (3)	-0.0111 (2)	0.5596 (6)	3.40 (9)
C(16)	0.2573 (3)	-0.0573 (2)	0.5532 (7)	3.9 (1)
C(17)	0.2100 (3)	-0.0907 (2)	0.4222 (7)	4.0 (1)
C(18)	0.1267 (3)	-0.0767 (2)	0.2946 (7)	4.3 (1)
C(19)	0.0931 (3)	-0.0312 (2)	0.2964 (7)	4.0 (1)
C(20)	0.2833 (3)	0.0237 (2)	0.7093 (7)	3.9 (1)
C(21)	0.3232 (4)	0.0653 (2)	0.5966 (8)	5.0 (1)
C(22)	0.3903 (5)	0.0525 (2)	0.441 (1)	7.3 (2)
C(23)	0.2106 (4)	0.0425 (2)	0.8534 (8)	5.8 (1)
C(24)	0.3718 (4)	0.0013 (2)	0.8431 (8)	5.3 (1)
C(25)	0.2530 (4)	-0.1397 (2)	0.4197 (8)	5.2 (1)
C(27)	0.3876 (6)	-0.1140 (3)	0.197 (1)	9.3 (2)
C(26A)*	0.3672 (6)	-0.1384 (3)	0.393 (1)	6.4 (2)†
C(28A)*	0.1970 (7)	-0.1713 (3)	0.262 (1)	7.5 (2)†
C(29A)*	0.2528 (6)	-0.1622 (3)	0.641 (1)	7.0 (2)†
C(26B)*	0.297 (2)	-0.1438 (9)	0.196 (4)	6.9 (6)†
C(28B)*	0.160 (1)	-0.1760 (6)	0.368 (3)	3.8 (3)†
C(29B)*	0.320 (2)	-0.1546 (8)	0.588 (3)	5.5 (5)†
H(N)	0.171 (3)	0.141 (1)	0.354 (6)	1.4 (9)†
H(O(1))	0.124 (4)	0.172 (2)	-0.248 (8)	4 (1)†

* Alternate A and B positions were modeled to fit the disorder. The relative occupancy of these positions was assumed to be in the ratio 3:1 for A and B , respectively, so as to make the temperature factors approximately equal for the A/B atom pairs.

† Atoms refined isotropically.

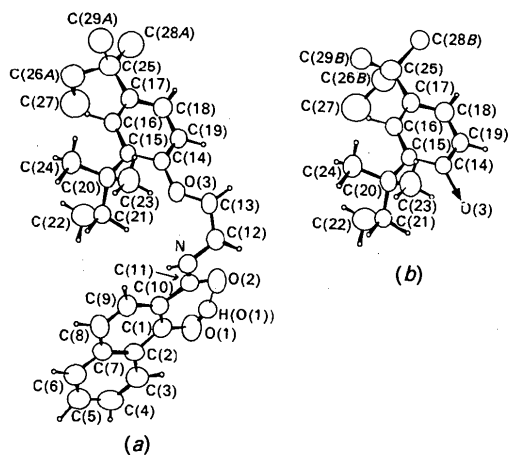


Fig. 1. (a) Conformation where the p -*tert*-pentyl group is in the A position. The labeling of the non-H atoms is illustrated. The thin bond represents the intramolecular hydrogen bonding $\{O(1)-H(O(1))\cdots O(2)\}$. The hydrogen-bond length and the corresponding angles are given in Table 2. (b) Conformation where the p -*tert*-pentyl group is in the B position. Thermal ellipsoids for C, N and O atoms and the hydrogen-bonded H atom are drawn at the 50% probability level. The other H atoms illustrated were drawn arbitrarily small for clarity. Structures were plotted using the program *ORTEPII* (Johnson, 1976).

Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The E map calculated with 310 reflections ($|E| > 1.75$) and the phase set having the best combined figure of merit gave all non-H atoms except those on the *tert*-pentyl groups. Subsequently, difference electron density maps yielded the remaining C atoms and all H atoms not involved in the disorder of the p -*tert*-pentyl group. Refinement of 375 parameters was performed by full-matrix least squares on F using 2432 observed reflections with $I > \sigma(I)$ (*SDP-Plus*; Frenz, 1985). Anisotropic thermal parameters were applied to all atoms, except those involved in the disorder and the H atoms. The function minimized was $\sum w(|F_o| - k|F_c|)^2$, where $w^{-1} = \sigma^2(F_o) + (0.04F_o)^2 + 2$ (Killean & Lawrence, 1969). The constant, 2, in this weighting expression was chosen so as to keep S on the order of unity. Scattering factors and anomalous-dispersion corrections for all atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The agreement indices are $R = \sum ||F_o| - k|F_c|/|$

Table 2. Selected bond lengths (Å) and angles (°) with *e.s.d.'s* in parentheses

O(1)—C(1)	1.349 (5)	O(1)—H[O(1)]	1.00 (5)
O(2)—C(11)	1.241 (5)	O(2)—H[O(1)]	1.58 (5)*
O(3)—C(13)	1.426 (5)	O(3)—C(14)	1.378 (5)
N—C(11)	1.335 (5)	N—C(12)	1.449 (5)
N—H(N)	0.91 (4)	C(1)—C(2)	1.431 (6)
C(1)—C(10)	1.378 (6)	C(2)—C(3)	1.410 (6)
C(2)—C(7)	1.408 (6)	C(3)—C(4)	1.362 (7)
C(4)—C(5)	1.395 (8)	C(5)—C(6)	1.356 (7)
C(6)—C(7)	1.402 (6)	C(7)—C(8)	1.414 (6)
C(8)—C(9)	1.340 (6)	C(9)—C(10)	1.424 (6)
C(10)—C(11)	1.478 (6)	C(12)—C(13)	1.497 (6)
C(14)—C(15)	1.409 (5)	C(14)—C(19)	1.376 (5)
C(15)—C(16)	1.393 (5)	C(15)—C(20)	1.544 (5)
C(16)—C(17)	1.396 (5)	C(17)—C(18)	1.374 (6)
C(17)—C(25)	1.527 (6)	C(18)—C(19)	1.387 (6)
C(20)—C(21)	1.546 (6)	C(20)—C(23)	1.549 (6)
C(20)—C(24)	1.533 (6)	C(21)—C(22)	1.502 (7)
C(25)—C(26A)	1.562 (8)	C(25)—C(28A)	1.517 (9)
C(25)—C(29A)	1.612 (9)	C(25)—C(26B)	1.67 (2)
C(25)—C(28B)	1.63 (2)	C(25)—C(29B)	1.42 (2)
C(27)—C(26A)	1.536 (9)	C(27)—C(26B)	1.49 (2)
C(1)—O(1)—H[O(1)]	103 (3)	C(11)—O(2)—H[O(1)]	100 (2)*
C(13)—O(3)—C(14)	119.0 (3)	C(11)—N—C(12)	123.5 (4)
C(11)—N—H(N)	117 (2)	C(12)—N—H(N)	119 (2)
O(1)—C(1)—C(2)	117.1 (4)	O(1)—C(1)—C(10)	121.9 (4)
C(2)—C(1)—C(10)	121.0 (4)	C(1)—C(2)—C(3)	122.2 (4)
C(1)—C(2)—C(7)	119.0 (4)	C(3)—C(2)—C(7)	118.8 (4)
C(2)—C(3)—C(4)	120.6 (5)	C(3)—C(4)—C(5)	120.5 (5)
C(4)—C(5)—C(6)	119.8 (5)	C(5)—C(6)—C(7)	121.5 (5)
C(2)—C(7)—C(8)	118.7 (4)	C(2)—C(7)—C(8)	118.8 (4)
C(6)—C(7)—C(8)	122.5 (4)	C(7)—C(8)—C(9)	121.3 (4)
C(8)—C(9)—C(10)	121.7 (5)	C(1)—C(10)—C(9)	118.2 (4)
C(1)—C(10)—C(11)	119.0 (4)	C(9)—C(10)—C(11)	122.7 (4)
O(2)—C(11)—N	120.4 (4)	O(2)—C(11)—C(10)	121.3 (4)
N—C(11)—C(10)	118.3 (4)	N—C(12)—C(13)	111.4 (4)
O(3)—C(13)—C(12)	107.2 (4)	O(3)—C(14)—C(15)	117.2 (3)
O(3)—C(14)—C(19)	122.7 (3)	C(15)—C(14)—C(19)	120.1 (4)
C(14)—C(15)—C(16)	116.3 (3)	C(14)—C(15)—C(20)	122.5 (3)
C(16)—C(15)—C(20)	121.2 (3)	C(15)—C(16)—C(17)	124.4 (4)
C(16)—C(17)—C(18)	117.1 (4)	C(16)—C(17)—C(25)	120.1 (4)
C(18)—C(17)—C(25)	122.8 (4)	C(17)—C(18)—C(19)	120.6 (4)
C(14)—C(19)—C(18)	121.6 (4)	C(15)—C(20)—C(21)	111.2 (3)
C(15)—C(20)—C(23)	109.5 (3)	C(15)—C(20)—C(24)	112.5 (4)
C(21)—C(20)—C(23)	108.3 (4)	C(21)—C(20)—C(24)	108.6 (4)
C(23)—C(20)—C(24)	106.7 (4)	C(20)—C(21)—C(22)	114.9 (4)
C(17)—C(25)—C(26A)	110.9 (4)	C(17)—C(25)—C(28A)	114.1 (5)
C(17)—C(25)—C(29A)	108.5 (4)	C(17)—C(25)—C(26B)	104.5 (8)
C(17)—C(25)—C(28B)	108.6 (6)	C(17)—C(25)—C(29B)	118.3 (9)
C(26A)—C(25)—C(28A)	109.8 (5)	C(26A)—C(25)—C(29A)	103.3 (5)
C(28A)—C(25)—C(29A)	109.7 (5)	C(26B)—C(25)—C(28B)	96 (1)
C(26B)—C(25)—C(29B)	115 (1)	C(28B)—C(25)—C(29B)	112 (1)
C(25)—C(26A)—C(27)	113.0 (6)	C(25)—C(26B)—C(27)	109 (1)
O(1)—H[O(1)]—O(2)	154 (1)*		

* These bond lengths and angles involve the hydrogen bond illustrated in Fig. 1.

$\sum |F_o|$, $wR = [\sum w(|F_o| - k|F_c|)^2 / \sum wF_o^2]^{1/2}$, and $S = |\sum w(|F_o| - k|F_c|)^2 / (n_o - n_v)|^{1/2}$. At convergence, the final $R = 0.072$, $wR = 0.099$, $S = 1.40$, and $k = 1.980$ (9). The maximum shift in the final cycle was 0.03σ . Residual electron density in the final difference Fourier map was -0.20 to 0.44 e Å⁻³.

Discussion. The atom-numbering scheme is illustrated in Fig. 1. The refinement was complicated by disorder in the *p-tert-pentyl* group. This disorder was modeled with two alternate positions (*A* and *B*, ratio 3:1) for atoms C(26), C(28) and C(29). This 3:1 ratio was chosen to make the temperature factors approximately equal for the *A/B* atom pairs. Atom C(27) is also disordered; however, its alternate positions could not be resolved.

Atomic positional and isotropic thermal parameters and bond distances and angles are listed in Tables 1 and 2,* respectively. The molecular structure is illustrated in Fig. 1. The molecules pack in (010) sheets containing pairs of molecules related by a center of symmetry. This sheet structure, illustrated in the (100) and (001) stereographic plots in Fig. 2, is bounded on either surface by *p-tert-pentyl* and naphthol groups, where contact occurs with adjacent sheets. Intrasheet contacts are between *p-tert-pentyl* and naphthol groups. This sheet structure differs markedly from that of the (010) sheets found (Luss & Texter, 1991) in the analogous (coupler) structure, (II), where two additional methylene groups tie the phenoxy group to the N atom. In (II), the naphthol rings are buried within the (010) sheet (no overlap between adjacent naphthol rings) and the sheet surfaces comprise *p-tert-pentyl* groups. The intramolecular hydrogen bond in (I) {O(1)—H[O(1)]···O(2)} is illustrated in Fig. 1, and the relevant distances and angles are given in Table 2.

* Lists of anisotropic thermal parameters, structure factors and H-atom positional parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54880 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0359]

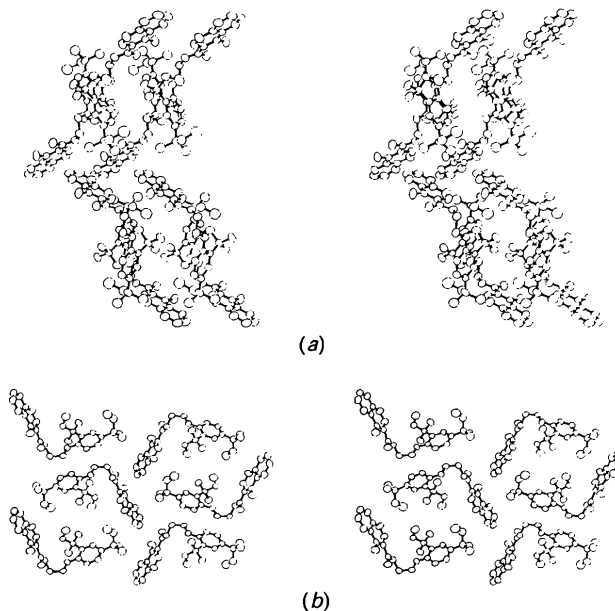


Fig. 2. (a) Stereographic plot (100) of the molecular packing. The *c* axis is horizontal (left to right) and the *b* axis is vertical (top to bottom); (b) stereographic plot (001) of the molecular packing. The *a* axis is vertical (top to bottom) and the *b* axis is horizontal (left to right). Thermal ellipsoids are drawn at the 50% probability level. Structures were plotted using the program ORTEPII (Johnson, 1976).

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Acta Cryst. (1992). **C48**, 1272–1275

The Structure of Triphenylmethanol, Ph₃COH

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(Received 22 October 1991; accepted 20 December 1991)

Abstract. C₁₉H₁₆O, $M_r = 260.3$, trigonal, $R\bar{3}$, $a = 19.307(3)$, $c = 26.735(4)$ Å, $V = 8631(2)$ Å³, $Z = 24$, $D_x = 1.20$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.7$ cm⁻¹, $F(000) = 3312$, $T = 294$ K, $R = 0.083$, $wR = 0.068$ for 1022 observed reflections. The structure contains hydrogen-bonded pyramidal tetramers (which have approximate 32 symmetry) with one molecule lying on a crystallographic threefold axis and another in a general position. The analysis was complicated by disorder in the crystal lattice where 71% of the tetramers occupy one orientation and 29% another; these moieties have their phenyl rings in the same volume elements in the lattice. The O···O separations in the hydrogen-bond systems are 2.884(10) and 2.896(11) Å in the major tetramer and 2.80(3) and 2.90(3) Å in the minor tetramer.

Introduction. We have been interested for some time in the structures of the series Ph₃MOH ($M = \text{C, Si, Ge, Sn, Pb}$). Although full structural data are available (*see below*) for the Si, Ge, Sn and Pb derivatives, no structural data are available for the Ph₃COH

compound [a recent search of the Cambridge Structural Database (October 1991, 90295 entries) for the unsolvated triphenylmethanol molecule yielded no hits]. In a brief footnote to a paper on the structures of methanol and dimethyl sulfoxide hydrogen-bonded clathrates of triphenylmethanol, Weber, Skobridis & Goldberg (1989) mention that a crystal structure analysis of the unsolvated triphenylmethanol led to a structural model which would not refine satisfactorily. This was attributed to poor crystal quality, possibly resulting from twinning or the inclusion of small non-stoichiometric amounts of solvent; no structural data or details were given apart from the statement that the compound 'self-associates by forming H-bonded tetramers which pack rather loosely in the crystalline state'. We have obtained well formed unsolvated crystals of triphenylmethanol by slow evaporation of a saturated benzene solution and report the crystal structure.

Experimental. A crystal measuring $0.45 \times 0.30 \times 0.30$ mm was mounted on a glass fibre with its long