

compounds. The resonance form as drawn in (I)–(IV) predominates. The effect is more pronounced in the present case, especially in the C(4)–C(5) and C(5)–C(6) bonds, which have lengths typical of a pure double bond and a pure sp^2 – sp^2 carbon atom single bond, respectively. Bond angles in the phenyl and pyridyl rings are as expected.

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Third (γ) Polymorph of Tri-*tert*-butylmethyl *p*-Nitrobenzoate, C₂₀H₃₁NO₄

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Abstract. $M_r = 349.5$, monoclinic, $P2_1/c$, $a = 12.655$ (6), $b = 26.523$ (5), $c = 12.134$ (6) Å, $\beta = 105.8$ (2)°, $U = 3919$ (3) Å³, $Z = 8$, $D_x = 1.194$ g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 6.7$ cm⁻¹. $F(000) = 1520$; $T = 298$ K. Final $R = 0.087$ for 3453 significant reflections. The two independent molecules of this polymorph have virtually identical structures. They differ from that of the α polymorph in that the central C(1)–C(7)–O(1)–C(8) system has a torsional angle of 178° in the former and 162° in the latter.

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Introduction. Molecular overcrowding caused by bulky substituents on *p*-nitrobenzoate esters such as the title compound increases their rate of solvolysis (Cheng, Nyburg, Thankachan & Tidwell, 1977). The strain found in molecules of the α polymorph of the title compound has been reported (Cheng & Nyburg, 1978). This paper reports structural results for the molecules of the γ polymorph.

Experimental. The first crystals to form from acetone–water solutions were colorless and of the γ polymorph. Crystals deposited later consist of pale-yellow clusters composed of two mutually-imbedded different poly-

morphic forms (now designated α and β). Because of the superposition of certain X-ray reflections, only the crystal structure of the α polymorph could be solved accurately (Cheng & Nyburg, 1978). The structure of the β polymorph was solved approximately.

Crystal $0.16 \times 0.30 \times 0.62$ mm sealed in a capillary, Picker four-circle diffractometer using Ni-filtered Cu $K\alpha$ radiation, no absorption corrections made; data collected in θ - 2θ scan mode to $2\theta = 124^\circ$ in the quadrant $h = -13$ to $+13$, $k = 0$ to 27 , $l = 0$ to 12 . For the first 2000 reflections measured at 1° min^{-1} , the intensity of a standard reflection fell by 10%; a correction linear with time was applied. For the remaining 3400 reflections, the scan speed was increased to 2° min^{-1} . During this latter period both the standard reflection and the last 200 reflections which were remeasured showed a further fall in intensity of 25%. For each successive set of 500 reflections in this latter period, a separate correction, linear with time was applied. 3453 reflections out of 5403 considered significant on criterion $F_o > 2\sigma(F_o)$ where $\sigma(F_o) = \{[(I/Lp)^2 + 0.02F_o^4]^{1/2}\}$; merging consistency for $0kl$, 0.080. Structure solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971) with 328 E values > 1.4 ; refinement on F by *ORXFLS3* (Busing, Martin & Levy, 1971), atomic scattering factors from Cromer & Mann (1968); H atoms fixed in calculated positions, C-H equal to 1 \AA , with isotropic temperature factors varied; $R = 0.087$, $R_w = 0.12$ ($w = 1/\sigma_F^2$); final cycle, $(\Delta/\sigma)_{\text{max}} = 0.012$ [temperature factor of H(A18A)], final $\Delta\rho$ excursions $\leq |0.4| e \text{ \AA}^{-3}$.

Discussion. Fractional coordinates and equivalent isotropic temperature factors of non-hydrogen atoms are given in Table 1.* Table 2 shows that the bond lengths in the two independent molecules and in that of the α polymorph are closely concordant (root-mean-square differences: γ_A vs γ_B 0.0037 \AA , γ_A vs α 0.0032 \AA , γ_B vs α 0.0036 \AA).

There seems to be no simple relation between the packing of the molecules in this polymorph (Fig. 1) and that present in either the α or β polymorphs. The latter are however slightly more densely packed, having densities 1.22 as against 1.19 g cm^{-3} .

The A and B molecules of this polymorph are related in the crystal by an almost exact non-crystallographic center of symmetry. [In Table 1, for every atom, $(x_A + x_B) \simeq 0.72$, $(y_A + y_B) \simeq 0.49$ and $(z_A + z_B) \simeq 0.12$.] One such pair of molecules, disposed exactly as in the crystal, is illustrated in Fig. 2.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38812 (17 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 ($\times 10^4$) with e.s.d.'s of non-hydrogen atoms and equivalent isotropic temperature factors

$$T. F. = \exp(-\sum_i \sum_j \beta_{ij} h_i h_j), B_{\text{eq}} = \frac{1}{3} \sum_i \sum_j \beta_{ij} a_i a_j$$

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
O(A1)	1747 (2)	1300 (1)	1094 (2)	4.0 (1)
O(A2)	2044 (3)	2088 (1)	481 (3)	6.1 (1)
O(A3)	5653 (4)	342 (2)	-1238 (5)	10.8 (2)
O(A4)	6285 (3)	1093 (2)	-1362 (4)	8.3 (2)
N(A)	5640 (4)	798 (2)	-1100 (4)	7.4 (2)
C(A1)	3108 (3)	1400 (2)	144 (4)	4.2 (1)
C(A2)	3852 (4)	1722 (2)	-142 (4)	5.1 (2)
C(A3)	4713 (4)	1523 (2)	-525 (4)	5.4 (2)
C(A4)	4766 (4)	1013 (2)	-649 (4)	5.2 (2)
C(A5)	4001 (4)	682 (2)	-413 (4)	6.0 (2)
C(A6)	3183 (4)	887 (2)	13 (4)	5.4 (2)
C(A7)	2216 (3)	1645 (2)	582 (4)	4.3 (1)
C(A8)	813 (3)	1338 (2)	1656 (4)	4.0 (1)
C(A9)	-296 (4)	1330 (2)	629 (4)	5.3 (2)
C(A10)	1083 (4)	813 (2)	2387 (4)	5.2 (2)
C(A11)	975 (3)	1836 (2)	2474 (4)	4.5 (1)
C(A12)	-286 (4)	1723 (2)	-314 (4)	7.3 (2)
C(A13)	-1323 (4)	1431 (2)	1067 (5)	6.7 (2)
C(A14)	-533 (4)	843 (2)	-65 (5)	7.7 (2)
C(A15)	97 (4)	623 (2)	2810 (5)	7.1 (2)
C(A16)	1401 (5)	374 (2)	1682 (6)	8.8 (3)
C(A17)	2073 (4)	854 (2)	3457 (5)	7.5 (2)
C(A18)	483 (4)	2322 (2)	1914 (5)	6.1 (2)
C(A19)	440 (4)	1770 (2)	3470 (4)	6.5 (2)
C(A20)	2212 (4)	1964 (2)	3022 (4)	6.2 (2)
O(B1)	5519 (2)	3617 (1)	120 (2)	4.0 (1)
O(B2)	5226 (3)	2826 (1)	722 (3)	6.0 (1)
O(B3)	1525 (4)	4544 (2)	2332 (5)	11.9 (2)
O(B4)	996 (3)	3809 (2)	2573 (3)	7.9 (1)
N(B)	1587 (4)	4096 (2)	2256 (4)	7.1 (2)
C(B1)	4157 (3)	3508 (2)	1052 (3)	3.8 (1)
C(B2)	3393 (4)	3184 (2)	1317 (4)	4.9 (2)
C(B3)	2538 (4)	3388 (2)	1709 (4)	5.0 (2)
C(B4)	2483 (4)	3887 (2)	1833 (4)	4.6 (1)
C(B5)	3234 (4)	4215 (2)	1604 (5)	5.9 (2)
C(B6)	4081 (4)	4014 (2)	1197 (4)	4.9 (2)
C(B7)	5039 (3)	3266 (2)	619 (4)	4.1 (1)
C(B8)	6459 (3)	3581 (2)	-430 (3)	3.7 (1)
C(B9)	7545 (4)	3616 (2)	614 (4)	5.8 (2)
C(B10)	6181 (4)	4088 (2)	-1194 (4)	5.4 (2)
C(B11)	6354 (3)	3077 (2)	-1204 (4)	4.2 (1)
C(B12)	7569 (5)	3233 (2)	1596 (4)	7.5 (2)
C(B13)	8629 (4)	3518 (2)	232 (5)	7.5 (2)
C(B14)	7713 (5)	4118 (2)	1244 (5)	8.9 (2)
C(B15)	7161 (4)	4292 (2)	-1607 (5)	6.8 (2)
C(B16)	5789 (6)	4537 (2)	-563 (6)	9.0 (3)
C(B17)	5206 (4)	4024 (2)	-2285 (5)	7.2 (2)
C(B18)	6864 (4)	2586 (2)	-590 (4)	5.4 (2)
C(B19)	6923 (4)	3137 (2)	-2183 (4)	5.6 (2)
C(B20)	5161 (4)	2930 (2)	-1772 (4)	5.9 (2)

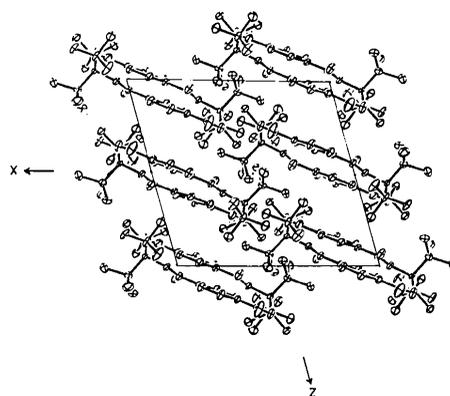


Fig. 1. ORTEP plot (Johnson, 1965) showing molecular packing in the y projection. Only non-hydrogen atoms are shown, with 50% probability ellipsoids.

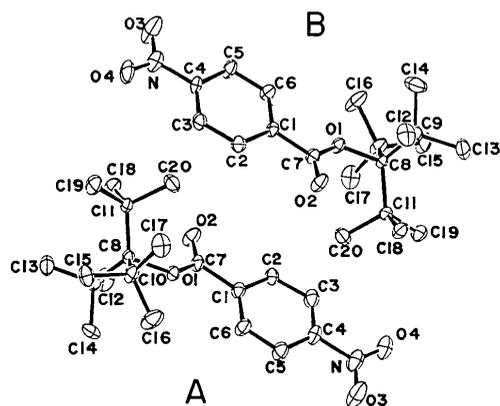


Fig. 2. ORTEP plot (Johnson, 1965) of a pair of molecules *A* and *B*, showing atomic numbering, exactly as disposed in the crystal.

Table 2. Bond lengths (Å) in the α -polymorph molecule and in the *A* and *B* molecules of the γ polymorph

	α	γ_A	γ_B
O(1)–C(7)	1.332 (5)	1.333 (5)	1.343 (5)
O(1)–C(8)	1.496 (5)	1.520 (5)	1.517 (5)
O(2)–C(7)	1.207 (6)	1.195 (5)	1.190 (5)
O(3)–N	1.219 (6)	1.223 (7)	1.197 (7)
O(4)–N	1.227 (7)	1.232 (6)	1.201 (6)
N–C(4)	1.468 (7)	1.476 (7)	1.473 (7)
C(1)–C(2)	1.387 (6)	1.383 (6)	1.394 (6)
C(1)–C(6)	1.386 (6)	1.375 (6)	1.361 (6)
C(1)–C(7)	1.503 (7)	1.517 (6)	1.500 (6)
C(2)–C(3)	1.393 (7)	1.400 (7)	1.404 (7)
C(3)–C(4)	1.369 (7)	1.363 (7)	1.336 (7)
C(4)–C(5)	1.365 (6)	1.392 (7)	1.373 (7)
C(5)–C(6)	1.398 (6)	1.388 (7)	1.401 (7)
C(8)–C(9)	1.612 (6)	1.604 (6)	1.598 (6)
C(8)–C(10)	1.636 (5)	1.634 (6)	1.617 (6)
C(8)–C(11)	1.605 (6)	1.633 (6)	1.618 (6)
C(9)–C(12)	1.571 (6)	1.549 (7)	1.561 (7)
C(9)–C(13)	1.551 (7)	1.554 (7)	1.585 (7)
C(9)–C(14)	1.539 (7)	1.527 (7)	1.521 (8)
C(10)–C(15)	1.543 (7)	1.556 (8)	1.555 (8)
C(10)–C(16)	1.561 (6)	1.562 (8)	1.569 (8)
C(10)–C(17)	1.542 (7)	1.544 (8)	1.555 (8)
C(11)–C(18)	1.560 (7)	1.510 (7)	1.551 (6)
C(11)–C(19)	1.559 (6)	1.547 (7)	1.557 (7)
C(11)–C(20)	1.565 (8)	1.563 (7)	1.530 (7)

Table 3. The results of best molecular fitting (Yuen & Nyburg, 1979), expressed as the sum of squared deviations (\AA^2) for the *A* and *B* molecules of the γ polymorph with that of the α polymorph

(a) All 25 non-hydrogen atoms. (b) The nine non-hydrogen atoms of the nitrophenyl group plus C(7). (c) The 12 non-hydrogen atoms of the *tert*-butyl group plus O(1).

	γ_A vs γ_B	γ_A vs α	γ_B vs α
(a)	0.056	4.568	3.917
(b)	0.023	0.061	0.097
(c)	0.009	0.052	0.034

Table 4. Bond and dihedral angles of the central bond system

	Bond angles ($^\circ$)		Dihedral angles ($^\circ$)
	C(1)–C(7)–O(1)	C(7)–O(1)–C(8)	C(1)–C(7)–O(1)–C(8)
α	111.6	132.8	162.3
γ_A	109.7	131.5	–178.8
γ_B	109.4	131.2	178.6

The three molecules, *A* and *B* of this polymorph and the single independent molecule of the α polymorph, were compared by the least-squares rigid-body fitting program *BMFIT* (Yuen & Nyburg, 1979). It is seen, Table 3, that the fit between molecule γ_A and (the mirror image of) γ_B is excellent whereas, despite concordant bond lengths, both γ molecules fit poorly with the α molecule. This is due to important differences between the γ and α molecules in the C(1)–C(7)–O(1)–C(8) bond system. When the *p*-nitrophenyl moieties together with C(7) are compared, the fit is good [Table 3, line (b)] as it is also when the *tert*-butyl groups together with O(1) are compared [Table 3, line (c)]. Thus these two halves of the molecules match well. The reason for the poor overall matching of the γ and α molecules is due partly to small differences in interbond angles but, principally, to a large difference, 16° , in the torsion angles of the C(1)–C(7)–O(1)–C(8) bond system (Table 4).

Although best molecular fitting shows the *A* and the mirror image of the *B* molecules to fit closely, it is of interest to see to what extent they are related by a true center of symmetry in the crystal. Using *BMFIT*, the signs of all the coordinates of the *A* + *B* pair were reversed (giving the enantiomorphic pair *A'* + *B'*) and this latter pair, in the order *B'* + *A'* was matched with the original *A* + *B* pair. The sum of squares of deviations was 0.445 \AA^2 , which, considering the large number of atoms involved, is a good fit. However, there are clearly significant deviations from exact centrosymmetry.

Apart from differences in the torsional angle of the central bond system, referred to above, all three molecules are closely similar, as the concordant bond lengths in Table 2 indicate. In particular, due to overcrowding, the three C(8)–C(methyl) bonds are again long, averaging $1.623 (6)$ for γ_A , $1.611 (6)$ for γ_B , and $1.618 (6) \text{ \AA}$ for α . The C(methyl)–C(8)–C(methyl) angles are again opened up, $114.2 (3)^\circ$ in γ_A , $114.0 (3)^\circ$ in γ_B matching $113.8 (3)^\circ$ in α . The methyl H atoms are closely enmeshed, ten H...H distances lying between 1.85 and 1.90 \AA .

There are no abnormally short intermolecular distances.

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α -(4-Methyl-2-nitrophenylhydrazono)acetanilide, $C_{15}H_{14}N_4O_3$

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Abstract. $M_r = 298.3$, monoclinic, $P2_1/a$, $a = 23.344$ (15), $b = 5.905$ (8), $c = 12.164$ (12) Å, $\beta = 121.07$ (10)°, $V = 1436.21$ Å³, $Z = 4$, $D_x = 1.380$, $D_m = 1.378$ Mg m⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 0.84$ mm⁻¹, $F(000) = 624$, $T = 293$ K. Final $R = 0.045$ for 1183 observed film reflexions. The almost planar molecules exist as hydrogen-bonded dimers with also one internal hydrogen bond per molecule. The C atom of acetanilide to which the azo compound couples is bonded trigonally, transferring its H atom elsewhere; bond lengths indicate this H to be attached to an N of the diazo group.

Introduction. This title compound was obtained as an unwelcome impurity during the coupling of diazotized 2-nitro-*p*-toluidine to acetoacetanilide at 328 K. In this reaction the acetyl group became detached and the product was precipitated as large red crystals. The crystal-structure determination was undertaken in order to characterize this previously unknown substance. A preliminary mention of the crystal data is given by Brown (1967).

Experimental. Sample prepared by J. Mitchell of Imperial Chemical Industries Limited (Organics Division); purified acicular crystals grown from acetone solution, m.p. 476 (5) K; found: C 60.7, H 4.8, N 18.0%; required for $C_{15}H_{14}N_4O_3$: C 60.4, H 4.7, N 18.8%. D_m measured by flotation in aqueous NaI solution. Unit-cell dimensions obtained initially from rotation photographs and more accurately from $\alpha_1\alpha_2$ separations of high-angle reflexions on Weissenberg photographs. Crystal 4.0 × 0.15 × 0.05 mm used for intensities. Stoe integrating Weissenberg camera; multiple-film photographs taken of levels $h0l \rightarrow h3l$ and $hk0$; visual estimations using calibrated scale; interlayer scaling by correlation of common reflexions and comparison of Wilson plots. No corrections for absorption or extinction except for $F(41\bar{3})$. 2090

measured reflexions, 907 with $I < 3\sigma(I)$; index range of observed reflexions: $h = 0$ to 28, $k = 0$ to 7, $l = -14$ to 13. Structure solved by manual enhancement of Σ_2 relationships. Least-squares refinement using $\sqrt{w} = 1/F_o$ with B_{iso} initially and B_{ij} finally until shifts $< 0.3\sigma$. H parameters obtained from difference Fourier sections included with $B_{iso} = 6.0$ Å² but not refined. $R = 0.045$, $R_w = 0.094$. Final $\Delta\rho$ excursions = ± 0.1 e Å⁻³. Scattering factors from *International Tables for X-ray Crystallography* (1962). NRC computer programs used (Ahmed, Hall, Pippy & Huber, 1970) implemented on the ICL 1905E at the London Polytechnics Computer Centre, and on our own DEC-10.

Discussion. The final positional and equivalent isotropic temperature parameters are listed in Table 1.* Numbering of the atoms and the packing arrangement of the molecules in the unit cell are shown in Fig. 1. Bond lengths and interbond angles are given in Table 2.

The molecule is almost planar, the average distance of the 22 non-H atoms out of the mean plane being 0.046 Å (r.m.s. deviation 0.057 Å) with a maximum displacement of 0.111 (4) Å for C(1). Despite this planarity, this substance does not show pigmentary properties such as those possessed by groups of azo dyes which have, for example, an acetyl group substituted on C(8), the atom to which coupling has occurred. C(8) is trigonally bonded, confirming that C(8)–N(3) is formally a double bond [1.284 (5) Å], and N(2)–N(3) is only slightly shorter [1.334 (4) Å] than a single bond. Both of these agree with the corresponding dimensions in the dibromo derivative (Brown, 1967) where the lengths are 1.29 and 1.36 Å

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and mean-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38816 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.