a basic set in the calculations are expected to be reduced.

Taking into account the value of $d$ for 16 crystal structures of carboxylic acids analyzed elsewhere (Grabowski & Krygowski, 1984, 1989) it is clear that an increase in $d$ associated with an increase in $\Delta E(d)$ results in a decrease in the probability, $p(d)$, of finding dimers that could realize such a situation. Fig. 2 illustrates this clearly; for $d$ values between 0 and 0·1 Å, $p(d) = 0·57$; for $d$ values between 0·1 and 0·2 Å, $p(d) = 0·36$; and for $d > 0·2$ Å, $p(d) = 0·07$. This supports the hypothesis of Jeffrey & Sax (1963), since the greater the energy loss of the dimer, the lower is the probability of finding it.

There is no disorder for the carboxyl group of *p*-NPAA. According to Dieterich, Paul & Curtin (1974) the disorder exists if there is an equalization of the C=O and C—O(H) bond lengths and of the appropriate valence angles of the carboxyl group. Fig. 1 and Table 2 show that the difference between the bond lengths of the carboxyl groups is 0·057 Å and the difference between the angles O4—C8—C7 and O3—C8—C7 is 8·5°, *i.e.* that the geometry of the COOH group is not affected by disorder.

The angle between the best plane of the benzene ring and the plane of the $NO_2$ group is 7·25°; the angle between the plane of the COOH group and the plane of the benzene ring is 69·90°. The latter value is not caused by the lattice forces but rather by hybridization of C3, C7 and C8 atoms of the molecule (Fig. 1). The torsional angles O3—C8—C7—C3, O4—C8—C7—C3, C2—C3—C7—C8 and C4—C3—C7—C8 are 171·5, 10·2, 105·9 and −74·6°, respectively.

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# Structure of 4,4'-Dimethylbenzophenone

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**Abstract.** $C_{15}H_{14}O$, $M_r = 210·28$, orthorhombic, $P2_12_12_1$, $a = 7·957$ (1), $b = 12·169$ (2), $c = 12·268$ (2) Å, $V = 1187·89$ (3) Å$^3$, $Z = 4$, $D_x = 1·176$ Mg m$^{-3}$, $\lambda(Mo\ K\alpha) = 0·71073$ Å, $\mu = 0·68$ cm$^{-1}$, $T = 293$ K, $F(000) = 448$, $R = 0·049$ for 1184 independent reflections with $I > 1·5\sigma(I)$. The

Table 1. *Final atomic coordinates and equivalent iso-tropic thermal parameters* ($\times 10^4$) *for non-hydrogen atoms*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{eq}$ (Å$^2$) |
|---|---|---|---|---|
| C1 | 0·1989 (3) | 0·2587 (2) | 0·1519 (2) | 450 (4) |
| C2 | 0·2937 (3) | 0·2483 (2) | 0·0568 (2) | 498 (5) |
| C3 | 0·2947 (3) | 0·3321 (2) | −0·0192 (2) | 512 (5) |
| C4 | 0·2030 (3) | 0·4275 (2) | −0·0041 (2) | 508 (5) |
| C5 | 0·1078 (3) | 0·4376 (2) | 0·0903 (2) | 529 (5) |
| C6 | 0·1061 (3) | 0·3553 (2) | 0·1674 (2) | 516 (5) |
| C7 | 0·1947 (4) | 0·1727 (2) | 0·2386 (2) | 562 (5) |
| O7 | 0·1579 (4) | 0·1986 (2) | 0·3317 (2) | 900 (5) |
| C8 | 0·2039 | 0·5178 | −0·0882 | 753 (5) |
| C11 | 0·2374 (3) | 0·0561 (2) | 0·2132 (2) | 490 (5) |
| C21 | 0·1948 (4) | 0·0057 (2) | 0·1156 (2) | 543 (5) |
| C31 | 0·2299 (4) | −0·1045 (2) | 0·0992 (2) | 570 (5) |
| C41 | 0·3098 (3) | −0·1660 (2) | 0·1781 (2) | 573 (5) |
| C51 | 0·3533 (4) | −0·1154 (3) | 0·2746 (2) | 589 (5) |
| C61 | 0·3163 (4) | −0·0063 (3) | 0·2933 (2) | 562 (5) |
| C81 | 0·3492 | −0·2850 | 0·1581 | 898 (6) |

benzophenone molecule is not planar; the torsion angles about the bonds connecting the phenyl rings and the keto group are 22·6 (4) and 32·2 (4)°. Molecular packing is realized *via* van der Waals interactions.

**Introduction.** Study of vibration spectra of substituted benzophenones (Volovšek & Baranović, 1987) initiated the X-ray structure determination of the title compound. The space group and unit-cell parameters of the title compound are in agreement with those given by Toussaint (1952). Steric hindrance between the two *ortho*-positioned H atoms in benzophenone and its other derivatives prevents the benzophenone molecule from being planar (Pattabhi & Venkatesan, 1973; van der Velden & Noordik, 1979). The twist angles between the phenyl rings are in the range 20–35°. From phosphorescence emission spectra and optically detected magnetic resonance spectra of benzophenone and its derivatives, correlations between ring twisting and $\pi^*$ electron delocalization and O—O band shifts were found (van der Velden & Noordik, 1979, 1980). The present structure determination confirms the non-planar geometry of a 4,4′-substituted benzophenone.

Ito *et al.* (1987) also reported the structure of 4,4′-dimethylbenzophenone, but disorder of the methyl groups was not detected. The final disagreement factor revealing the accuracy of the structure determination was not given.

**Experimental.** Weissenberg photographs (Cu $K\alpha$ radiation) determined space group $P2_12_12_1$ uniquely. Cell dimensions were based on diffractometer measurements of 20 reflections in $\theta$ range 6·3–17·5°. A crystal of dimensions 0·6 × 0·4 × 0·6 mm was used for intensity data collection with a Nonius CAD-4F diffractometer, Mo $K\alpha$ radiation, graphite mono-

chromator, $\omega/2\theta$ scan (scan angle: 1·1 + 0·35 tan$\theta$) in the range 2·5 < $\theta$ < 28°. No significant variation was detected in the intensities of three standard reflections (283, 431, 251). No absorption or secondary extinction corrections were made. The structure was solved by direct methods (*SHELX*86: Sheldrick, 1985), revealing non-hydrogen atoms. Refinement by full-matrix least squares with anisotropic thermal parameters for non-hydrogen atoms (*SHELX*76: Sheldrick, 1983). 1184 independent reflections with $I > 1·5\sigma(I)$. $\Sigma w(|F_o| - k|F_c|)^2$ was minimized with final $w = [\sigma^2(F_o) + 0·006F_o^2]^{-1}$. The H-atom positional and isotropic thermal parameters were also refined. A difference Fourier map reveals two orientations of methyl (C8, C81) H atoms; both orientations were included with population parameter of 0·5 and treated as rigid groups in the refinement. Final values $R = 0·049$ and $wR = 0·060$ were obtained. The residual electron density in the final map was $-0·23 < \Delta\rho < 0·12$ e Å$^{-3}$ with max. shift/e.s.d. = 0·18 (C5, $z$). Scattering factors are those included in *SHELX*76 (Sheldrick, 1983). Final atomic coordinates of the non-H atoms and equivalent isotropic thermal parameters are given in Table 1.* Calculations were carried out on an IBM 4341 with *SHELX*76, *SHELX*86 and the program for

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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 52306 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
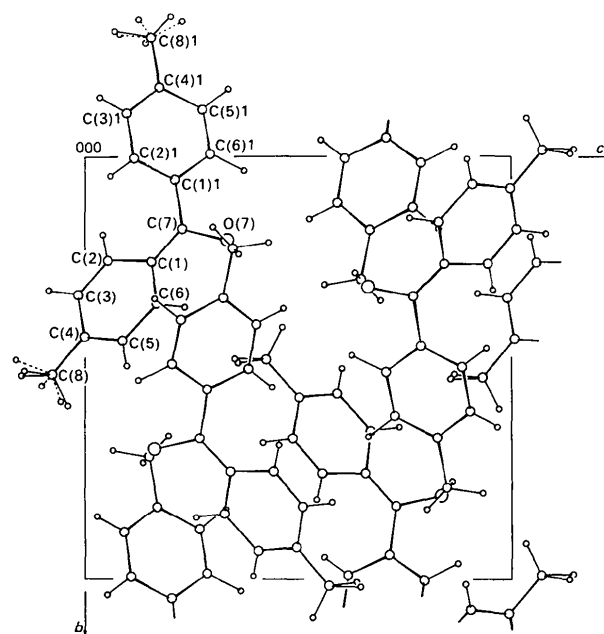


Fig. 1. Molecular packing viewed along **a** with the atom numbering.

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

| | | | |
|---|---|---|---|
| C1—C2 | 1·395 (3) | C11—C21 | 1·387 (4) |
| C1—C6 | 1·401 (3) | C11—C7 | 1·492 (4) |
| C1—C7 | 1·493 (4) | C11—C61 | 1·392 (4) |
| C2—C3 | 1·382 (4) | C21—C31 | 1·384 (4) |
| C3—C4 | 1·384 (3) | C31—C41 | 1·379 (4) |
| C4—C5 | 1·389 (3) | C41—C51 | 1·379 (4) |
| C4—C8 | 1·507 (2) | C51—C61 | 1·379 (5) |
| C5—C6 | 1·378 (4) | C41—C81 | 1·502 (2) |
| C7—O7 | 1·220 (4) | | |
| | | | |
| C2—C1—C7 | 123·0 (2) | C11—C7—O7 | 119·7 (2) |
| C2—C1—C6 | 118·3 (2) | C7—C11—C61 | 118·3 (2) |
| C6—C1—C7 | 118·7 (2) | C21—C11—C61 | 118·6 (3) |
| C1—C2—C3 | 120·0 (2) | C21—C11—C7 | 123·0 (2) |
| C2—C3—C4 | 121·7 (2) | C11—C21—C31 | 120·3 (2) |
| C3—C4—C5 | 118·2 (2) | C21—C31—C41 | 121·1 (2) |
| C3—C4—C8 | 121·2 (2) | C31—C41—C81 | 120·3 (2) |
| C5—C4—C8 | 120·6 (2) | C51—C41—C81 | 121·2 (2) |
| C4—C5—C6 | 120·9 (2) | C51—C41—C31 | 118·4 (3) |
| C1—C6—C5 | 120·8 (2) | C41—C51—C61 | 121·3 (2) |
| C1—C7—O7 | 119·4 (2) | C11—C61—C51 | 120·3 (2) |
| C1—C7—C11 | 120·9 (2) | | |

analysis of molecular geometry (Nardelli, 1983) at the University Computing Centre in Zagreb.

**Discussion.** The molecule with atom numbering is shown in the packing diagram (Fig. 1). Table 2 lists the bond lengths and angles.

The molecular geometry does not deviate from the geometry expected from standard atom types and hybridization (Table 2). Various symmetrically disubstituted aryl ketones, the title compound, and benzophenone by itself are not planar molecules. The ring twisting influences the delocalization of the $\pi^*$ electrons and can explain the changes of optical properties of benzophenones in relation to the electronic properties of the substituents (Batley, Bramley & Robinson, 1979).

The torsion angle about the bond connecting the phenyl ring and keto group is 26·0° in benzophenone (Fleischer, Sung & Hawkinson, 1968), 27·9° in 4,4′-

dichlorobenzophenone (Shields & Kennard, 1977), 25·2° in 4,4′-diiodobenzophenone (van der Velden & Noordik, 1979), 24 and 33° in 4,4′-diaminobenzophenone (van der Velden & Noordik, 1980), and 22·6 (4) (C6—C1—C7—O7) and 32·2 (4)° (C61—C11—C7—O7) in the present structure. The 4,4′-halogen substituted molecules exhibit a crystallographic twofold axis. Molecular packing is realized *via* van der Waals interactions (Fig. 1).

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# Structure of Diethyl 1-(8-Dimethylamino-1-naphthyl)-1H-1,2,3-triazole-4,5-dicarboxylate

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**Abstract.** $C_{20}H_{22}N_4O_4$, $M_r = 382·42$, monoclinic, $P2_1/n$, $a = 9·498$ (1), $b = 22·241$ (1), $c = 9·589$ (1) Å, $\beta = 97·47$ (1)°, $V = 2008·4$ (1) Å$^3$, $Z = 4$, $D_m = 1·27$, $D_x = 1·265$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu =$ 0·0971 mm$^{-1}$, $F(000) = 808$, $T = 295$ K, $R = 0·049$ for 2487 observed independent reflections. The dihedral angle between the mean plane of the naphthalene ring and that of the triazole ring in the title