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Short Communications

The Crystal Structure of a New Modification of *p*-Nitrobenzoic Acid at —150 °C

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A few colorless crystals were received (from our organic chemistry department) for X-ray investigation. To the suppliers disappointment, the crystals turned out to be *p*-nitrobenzoid acid. However, when checking the literature,[1-2] they revealed themselves as a new modification, (II), and the results are therefore reported.

The crystals belong to the monoclinic system with space group $P2_1/c$, cell dimensions $a=5.403(2)$, $b=5.153(2)$, $c=24.692(7)$ Å, $\beta=96.89(2)°$, and $Z=4$ ($D_x=1.62$ g cm^{-3}). Crystal data reported[1] (for modification (I) are: space group $A2/a$, $a=12.97$, $b=5.07$, $c=21.43$ Å, $\beta=96.4°$, $Z=8$ ($D_x=1.585$ g cm^{-3}, $D_m=1.600$ g cm^{-3}).

With $2\theta_{max}=50°$, Mo$K\alpha$-radiation, and an observed-unobserved cut off at $2\sigma(I)$, 506 independent reflections were recorded as observed on an automatic four-circle diffractometer at −150 °C. The structure was solved by direct methods[3] and refined by full-matrix least squares technique.[4,*] Hydrogen atoms were found in a difference Fourier map. Anisotropic temperature factors were introduced for O, N and C atoms and the weights in least squares were calculated from the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I)=[C_T+(0.02C_N)^2]^{\frac{1}{2}}$$

where C_T is the total number of counts and C_N the net count. The form factors used were those of Hanson *et al.*[5] except for hydrogen.[6] The final *R*-value was 6.5 % (weighted value $R_w=4.3$ %) for 506 observed reflections.

Final fractional coordinates with estimated standard deviations are listed in Table 1. In Fig. 1 bond lengths and angles of the two modifications are compared. Estimated standard deviations are 0.006 Å and 0.4° for (I) and 0.01 Å and 0.8° for (II), re-

* All programs used (except those for phase determination) are included in this reference.

Table 1. Final fractional coordinates with estimated standard deviations.

ATOM	X	Y	Z
O1	-.6201(12)	-.6784(11)	.3832(2)
O2	-.2786(11)	-.6518(10)	.2664(2)
O3	-.2111(10)	.2618(9)	.4981(2)
O4	.1011(11)	.3571(10)	.4410(2)
N	-.4169(15)	-.5814(12)	.2995(2)
C1	-.3313(13)	-.3694(14)	.3372(2)
C2	-.4608(16)	-.3307(16)	.3817(3)
C3	-.3615(16)	-.1362(16)	.4188(3)
C4	-.1768(15)	-.0149(14)	.4098(3)
C5	-.0496(14)	-.0305(15)	.3649(2)
C6	-.1245(15)	-.2254(13)	.3288(3)
C7	-.2956(16)	.2281(13)	.4581(3)
H2	-.612(16)	-.435(15)	.384(3)
H3	-.472(15)	-.099(17)	.449(3)
H5	.078(11)	.073(11)	.362(2)
H6	-.047(11)	-.248(10)	.298(2)
HO4	.163(28)	.499(22)	.474(4)

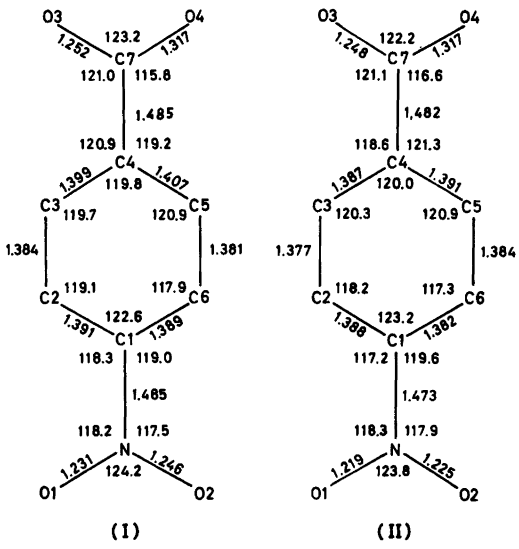

Fig. 1. Bond lengths and angles in the modification determined earlier, (I), and in the present modification, (II).

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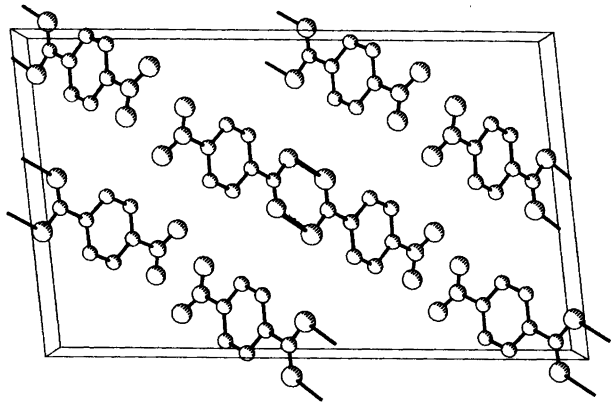

Fig. 2. Packing arrangement in modification (I).

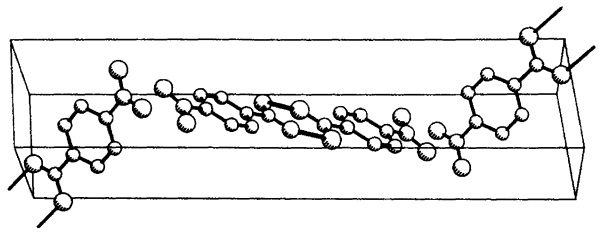

Fig. 3. Packing arrangement in modification (II).

spectively. Within these error limits no significant differences occur. The two oxygen atoms of the nitro group are displaced about 0.25 Å (in opposite directions) from the least squares plane defined by the rest of the atoms which are coplanar to within 0.01 Å. The same effect was observed for modification (I). Dimers are formed by hydrogen bonds O3···O4′ of length 2.622 Å. The corresponding distance of (I), 2.653 Å, is not significantly longer. The main difference between the two modifications is found in the packing arrangements which are illustrated in Figs. 2 and 3, respectively.

A list of thermal parameters and observed and calculated structure factors are available from the author.

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