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Further refinement of the structure of *o*-nitrobenzoic acid.*

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The structure of *o*-nitrobenzoic acid has been refined further by including hydrogen atoms and anisotropic temperature factors; the final *R* for 694 general observed reflexions is 0·104 and the average e.s.d. in bond lengths is 0·013 Å.

In order to obtain a better comparison with the structures of *p*- and *m*-nitrobenzoic acids (Tavale & Pant, 1971; Dhaneshwar, Tavale & Pant, 1974), the structure of *o*-nitrobenzoic acid reported earlier (Sakore, Tavale & Pant, 1967) has been subjected to five more cycles of least-squares refinement with the inclusion of hydrogen atoms, anisotropic temperature factors and Cruickshank's weighting scheme; 694 general observed structure factors listed by Sakore, Tavale & Pant (1967) were used for the refinement. *R* decreased from 0·142 to 0·104 and was nearly constant for the last three cycles of refinement. Final atomic and thermal parameters together with the e.s.d.'s (obtained from the diagonal terms of the least-squares inverse matrices) are given in Table 1.

The intramolecular bond lengths and angles (average e.s.d.'s, 0·013 Å and 0·9° respectively) are shown in Fig. 1. The corrections to C(7)–O(1), C(7)–O(2), N–O(3) and N–O(4) bond distances due to librational effects were evaluated in the usual way (Takwale & Pant, 1971) and amounted to 0·015, 0·012, 0·016 and 0·011 Å respectively; the details of the atomic thermal vibration ellipsoids are omitted for the sake of brevity. The carboxyl and nitro groups make angles of 24·1 and 54·3° (earlier values, 23·4 and 54·7°) respectively with the aromatic plane. The observed r.m.s. displacements of atoms O(1), O(2), C(7) and of N, O(3) and O(4) imply r.m.s. amplitudes of torsional oscillation of the carboxyl group about the C(1)–C(7) bond and of the nitro group about the C(2)–N bond of about 10° (each); however, unequal values for the amplitude of torsional oscillation obtained from O(3) (11°) and O(4) (10°) probably imply that NO_2 is not a perfectly rigid group.

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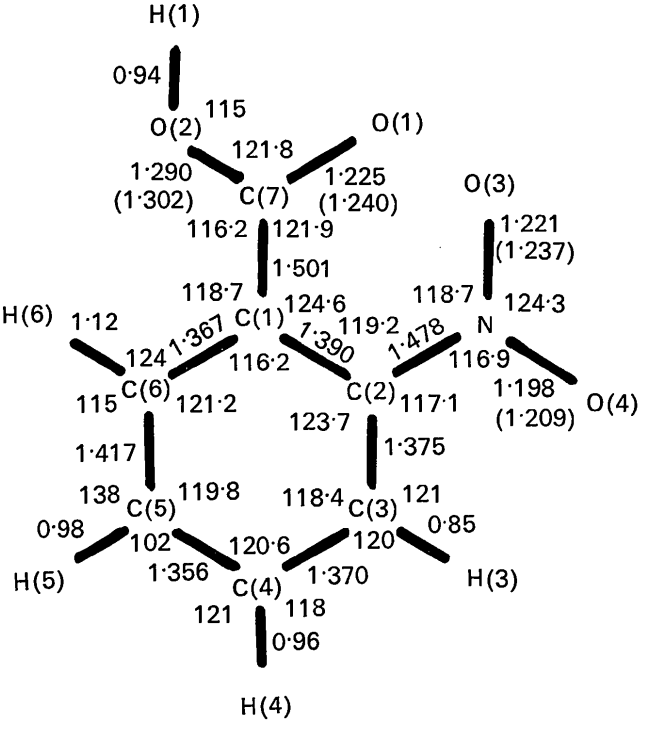

Fig. 1. Bond lengths (Å) and angles (°); bond lengths in parentheses have been corrected for thermal motion.

Table 1. *Final atomic and thermal parameters and their standard deviations (in parentheses)*

Anisotropic thermal parameters are of the form $T=\exp[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+2b_{12}hk+2b_{23}kl+2b_{13}hl)]$.

(*a*) Non-hydrogen atoms ($\times 10^4$)

	x	*y*	*z*	b_{11}	b_{22}	b_{33}	b_{12}	b_{23}	b_{13}
C(1)	−2829 (12)	−605 (20)	1936 (7)	166 (21)	448 (58)	61 (8)	148 (27)	101 (19)	42 (9)
C(2)	−2313 (11)	−951 (20)	2937 (7)	127 (19)	481 (57)	50 (7)	123 (25)	97 (18)	26 (8)
C(3)	−3622 (13)	−1891 (24)	3506 (8)	177 (23)	757 (75)	72 (9)	142 (32)	152 (23)	40 (10)
C(4)	−5511 (13)	−2314 (25)	3125 (9)	149 (21)	714 (73)	95 (10)	118 (32)	145 (23)	69 (11)
C(5)	−6088 (13)	−1919 (24)	2179 (9)	134 (21)	732 (77)	82 (9)	156 (31)	126 (23)	30 (10)
C(6)	−4732 (12)	−1088 (23)	1570 (8)	118 (19)	702 (71)	79 (9)	141 (30)	151 (22)	33 (10)
C(7)	−1550 (11)	−231 (19)	1128 (7)	136 (19)	448 (57)	50 (7)	65 (25)	98 (18)	16 (8)
O(1)	−192 (9)	−1088 (17)	1044 (6)	183 (16)	930 (56)	84 (6)	228 (24)	204 (17)	70 (8)
O(2)	−1992 (9)	963 (17)	500 (6)	227 (18)	965 (59)	96 (7)	256 (26)	242 (19)	76 (9)
O(3)	1082 (9)	2746 (17)	3904 (7)	123 (14)	726 (52)	126 (8)	37 (21)	189 (18)	10 (8)
O(4)	39 (9)	−2324 (16)	3513 (6)	187 (17)	709 (52)	111 (8)	195 (23)	174 (18)	35 (8)
N	−232 (10)	−136 (18)	3475 (6)	193 (19)	567 (52)	55 (6)	128 (24)	126 (17)	36 (8)

Table 1 (*cont.*)

(*b*) Hydrogen atoms ($\times 10^3$)

	x	*y*	*z*
H(1)	−114 (14)	130 (22)	4 (10)
H(3)	−327 (15)	−218 (28)	409 (11)
H(4)	−644 (15)	−305 (28)	350 (11)
H(5)	−743 (15)	−218 (29)	217 (11)
H(6)	−541 (15)	−126 (27)	68 (11)

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The structure of *N*,*N*-dimethylanthranilic acid.*

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The crystals of *N*,*N*-dimethylanthranilic acid, $N(CH_3)_2C_6H_4COOH$, are monoclinic, space group $P2_1/n$ with $a=7{\cdot}66$, $b=15{\cdot}74$, $c=7{\cdot}58$ Å; $\beta=100{\cdot}0°$; ϱ_c for $Z=4$, 1·218 g cm^{-3}. The structure was solved by direct methods with visually estimated data and refined by the method of least squares, resulting in an *R* value of 0·113 for 1037 observed reflexions; the e.s.d.'s in bond lengths not involving hydrogen atoms are 0·006–0·009 Å and in bond angles about 0·5°. The molecule exists in the crystal in the form of a zwitterion. There is a strong intramolecular NH···O hydrogen bond in which the N–O distance is 2·497 Å and O–H distance, 1·42 Å; the angle ONH is 16°.

This work is a continuation of our earlier study of the structure of *N*-methylanthranilic acid (Dhaneshwar & Pant, 1972). The crystals grown from solution in carbon tetrachloride are monoclinic, space group $P2_1/n$ with $a=7{\cdot}66$, $b=15{\cdot}74$, $c=7{\cdot}58$ Å; $\beta=100{\cdot}0°$; ϱ_c for $Z=4$, 1·218 g cm^{-3}. The axial lengths were measured from high-angle reflexions ($\theta\sim75°$) on zero-layer Weissenberg photographs with the films mounted in the Straumanis arrangement; β was determined by the method of triangulation (Jeffery, 1971). Owing to paucity of high-angle reflexions, errors could not be estimated but are expected to be within 0·02 Å in axial lengths and 0·2° in β. The density could not be measured as the crystals are soluble in all common solvents. Data were collected with unfiltered Cu radiation from zero to fourth-layer Weissenberg photographs about the '*a*' axis and from zero to fifth layer photographs about the '*c*' axis; the crystals used for the two sets of photographs had cross-sections $0{\cdot}5\times0{\cdot}6$ mm^2 and $0{\cdot}6\times1{\cdot}2$ mm^2 respectively. The data were processed in the usual way; absorption was neglected.

The normalized values of 157 structure factors were used in the Sayre's-equation program written by Long (1965); signs of 156 *F*'s were obtained and the three-dimensional Fourier map obtained from these revealed all the non-hydrogen atoms. The structure was first refined isotropically with unit weight for all reflexions; later hydrogen atoms and anisotropic temperature factors were included and the refinement continued using Cruickshank's weighting scheme. The final *R* is 0·113 for 1037 observed reflexions. The final atomic and thermal parameters along with their

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Table 1. *Final atomic and thermal parameters and their estimated standard deviations (in parentheses)*

Anisotropic thermal parameters are of the form $T=\exp[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+2b_{12}hk+2b_{23}kl+2b_{13}hl)]$.

(*a*) Non-hydrogen atoms ($\times 10^4$)

	x	*y*	*z*	b_{11}	b_{22}	b_{33}	b_{12}	b_{23}	b_{13}
C(1)	5638 (7)	1227 (3)	3938 (8)	147 (9)	44 (2)	203 (12)	12 (4)	15 (4)	34 (9)
C(2)	7313 (6)	1132 (3)	3455 (7)	141 (9)	47 (3)	133 (10)	7 (4)	3 (4)	26 (8)
C(3)	8792 (7)	1526 (4)	4358 (8)	161 (10)	56 (3)	242 (15)	−9 (4)	−20 (5)	39 (10)
C(4)	8615 (9)	2051 (4)	5824 (9)	244 (14)	62 (3)	233 (14)	−2 (5)	−37 (6)	18 (11)
C(5)	6977 (9)	2155 (4)	6292 (9)	295 (15)	53 (3)	248 (15)	9 (5)	−28 (6)	47 (12)
C(6)	5529 (8)	1748 (4)	5380 (8)	236 (12)	53 (3)	200 (12)	30 (5)	−3 (5)	76 (10)
C(7)	3990 (7)	797 (4)	2896 (8)	116 (9)	56 (3)	261 (15)	6 (4)	20 (5)	48 (9)
C(8)	8457 (8)	−202 (4)	2484 (9)	204 (12)	51 (3)	255 (15)	15 (5)	−14 (5)	59 (11)
C(9)	8202 (10)	1044 (5)	475 (10)	291 (16)	69 (4)	232 (16)	−33 (6)	2 (6)	114 (14)
O(1)	2586 (5)	898 (3)	3482 (6)	155 (7)	87 (3)	315 (12)	0 (3)	2 (5)	92 (8)
O(2)	4167 (5)	398 (3)	1515 (6)	155 (7)	89 (3)	279 (11)	−19 (4)	−57 (5)	28 (7)
N	7449 (5)	588 (3)	1919 (5)	143 (7)	49 (2)	153 (9)	−5 (3)	−13 (4)	51 (6)