1-Naphthaleneacetic Acid*

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Abstract. $C_{12}H_{10}O_2$ is monoclinic, $P2_1/c$ with $a = 12.705$ (6), $b = 5.146$ (2), $c = 15.015$ (7) Å, $\beta = 92.00$ (10)°, $V = 981.7$ Å^3, $Z = 4$, $D_m = 1.263$, $D_c = 1.262$ g cm^{-3}, $M_r = 186$. The structure was solved by direct methods and refined to an R factor of 0.129 for 776 observed reflections. The structure is stabilized by O–H$\cdots$O hydrogen bonds. The molecules exist as dimers.

Introduction. The lattice parameters of the title compound were determined with rotation and Weissenberg photographs, and later refined, with the 2θ values for 32 reflections obtained with the Picker diffractometer available in this laboratory, by use of the least-squares program of Main & Woolfson (1963). Three-dimensional intensity data for 776 reflections were collected by the multiple-film equi-inclination Weissenberg technique with Cu $K\alpha$ radiation and were estimated visually by comparison with a standard set of spots prepared with the same crystal. These were then corrected for Lorentz, polarization and spot-shape factors (Phillips, 1954). The data collected about the two axes (a and b) were correlated (Rollett & Sparks, 1960). As $\mu t \simeq 0.28$, no absorption correction was applied.

The structure was solved by direct methods with the multisolution technique of Germain, Main & Woolfson (1971), with the computer program available on the IBM 370/155 at the Indian Institute of Technology, Madras. All the non-hydrogen atoms of the molecule were located in the subsequent Fourier map. The initial R factor with all the non-hydrogen atoms was 0.46. Successive block-diagonal least-squares refinement of these atoms (Shiono, 1968) with isotropic temperature factors, followed by three cycles of anisotropic refinement, reduced the R factor to 0.15. A difference Fourier map revealed all ten H atoms. Two cycles of full-matrix refinement (Gantzel, Sparks & Trueblood, 1961) of the non-hydrogen atoms, with H atoms included only in the structure factor calculation, reduced the R factor to 0.129. The weighting scheme of Cruickshank, Bujosa, Lovell & Truter (1961), *i.e.* $w = (a + |F_o| + b|F_o|^2)^{-1}$ with $a = 2.0$ and $b = 0.033$, was

used. The scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The final positional parameters of the non-hydrogen atoms are given in Table 1,† while Table 2 gives the positions of the H atoms located from the difference Fourier map.

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33169 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional atomic coordinates* $(\times 10^4)$ *of non-hydrogen atoms*

Standard deviations given in parentheses refer to the least significant digit.

	x	y	z
C(1)	7214 (7)	1104 (14)	6935 (5)
C(2)	8051 (7)	−414 (15)	7274 (5)
C(3)	8456 (7)	−67 (20)	8149 (6)
C(4)	7969 (10)	1741 (24)	8697 (6)
C(5)	7187 (10)	3253 (19)	8388 (7)
C(6)	6783 (8)	3040 (16)	7514 (6)
C(7)	5917 (10)	4567 (22)	7171 (9)
C(8)	5543 (10)	4191 (27)	6317 (10)
C(9)	5962 (10)	2346 (25)	5741 (8)
C(10)	6771 (9)	752 (19)	6043 (6)
C(11)	8540 (7)	−2483 (14)	6670 (5)
C(12)	9170 (7)	−1310 (15)	5941 (5)
O(1)	9550 (5)	846 (12)	5994 (4)
O(2)	9304 (5)	−2829 (11)	5289 (4)

Table 2. *Positional* $(\times 10^3)$ *and thermal parameters of hydrogen atoms as obtained from the difference electron density map*

	Bonded to	x	y	z	$B(\text{Å}^2)$
H(1)	C(3)	946	−28	833	6.7
H(2)	C(4)	825	167	925	6.3
H(3)	C(5)	661	483	872	6.2
H(4)	C(7)	553	633	764	5.5
H(5)	C(8)	475	567	625	6.7
H(6)	C(9)	539	83	570	7.7
H(7)	C(10)	716	−95	563	5.9
H(8)	C(11)	786	−367	642	4.8
H(9)	C(11)	960	−317	700	4.8
H(10)	O(2)	981	−150	497	5.9

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Table 3. *Hydrogen-bond lengths* (Å) *and angles* (°)

Symmetry code: (i) $2 - x, 1 - y, 1 - z$.

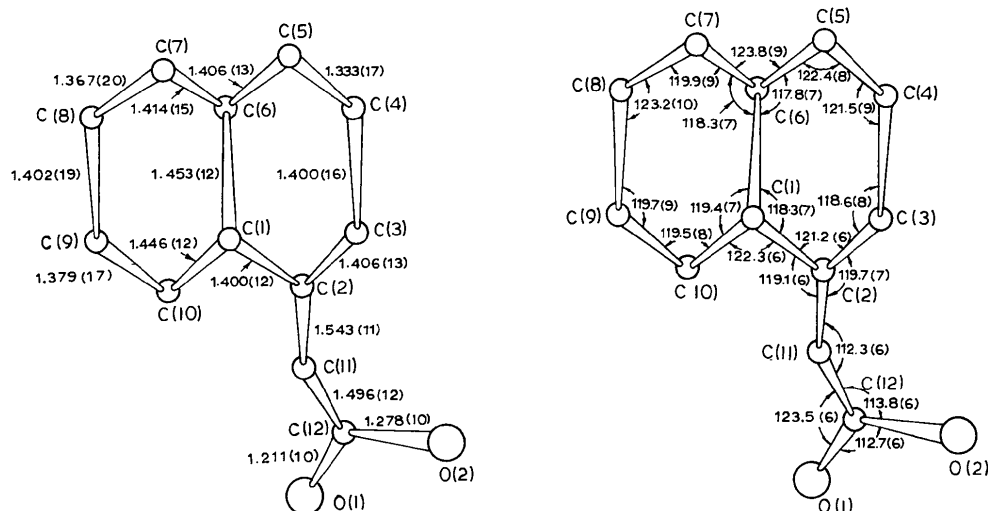| Donor (D)	Acceptor (A)	$D\cdots A$	$H\cdots A$	$D-H\cdots A$	$C-D\cdots A$	$H-D\cdots A$
C(12)—O(2)—H(10)	O(1^i)	2·65	1·71	146·1	114·8	21·0

Fig. 1. Bond lengths (Å) and bond angles (°) observed in 1-naphthaleneacetic acid. Estimated standard deviations are given in parentheses for the last digit quoted.
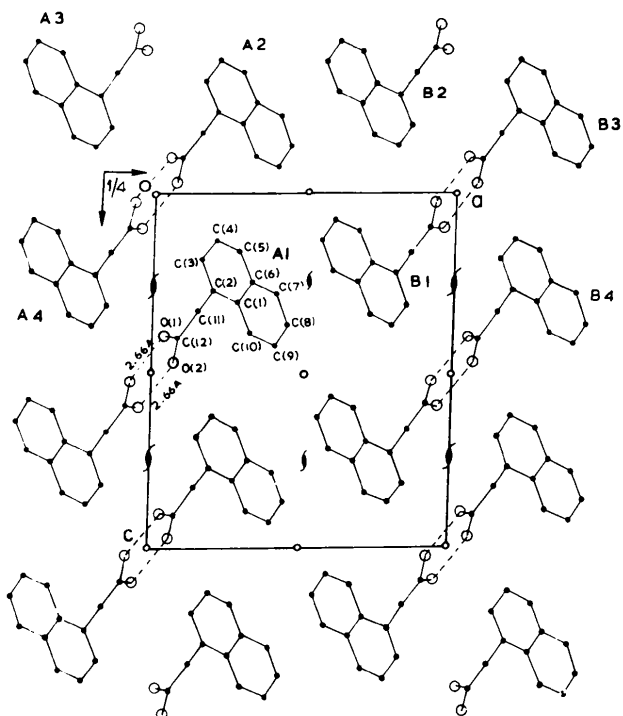

Fig. 2. Packing of the molecules viewed down **b**.

Table 4. *Least-squares planes and deviations* (Å) *of atoms from them*

Equation of the plane: $AX + BY + CZ = D$ with respect to the crystallographic axes a, b and c where X, Y, Z and D are in Å.

Naphthalene group: $0·6609X + 0·6800Y - 0·3172Z = 2·8938$

C(1)	0·008	C(8)	0·002
C(2)	0·008	C(9)	0·002
C(3)	0·022	C(10)	−0·031
C(4)	−0·033	C(11)	0·003
C(5)	−0·033	C(12)*	1·311
C(6)	0·013	O(1)*	2·361
C(7)	0·011	O(2)*	1·229

Carboxyl group: $-0·8180X + 0·3667Y - 0·4430Z = -13·4898$

C(11)	−0·003	O(1)	0·004
C(12)	0·012	O(2)	0·004

* Atom not included in plane calculation.

Discussion. The bond lengths and angles obtained for the molecule are as shown in Fig. 1 and are close to the normal values. O(1^i) accepts a proton from O(2) and forms a hydrogen bond. The hydrogen bond lengths and angles obtained are in Table 3.

The least-squares planes passing through (1) the atoms in the naphthalene ring and C(11) and (2) the

atoms in the carboxyl plane, are given in Table 4 along with the deviations of individual atoms from the planes. The angle between the two planes is found to be $97.8°$.

The molecular packing seen down **b** is shown in Fig. 2. The molecules, as in other plant-growth hormones (Swaminathan, Vijayalakshmi & Srinivasan, 1976; Karle, Britts & Gum, 1964), exist as dimers. Hydrogen bonds between the acid groups form across the centre of symmetry and are in parallel planes.

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2-Methyl-2,2'-spirobi(1,3,2-benzodioxaarsole)

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Abstract. $(C_6H_4O_2)_2AsCH_3$, orthorhombic, $Pca2_1$; $a = 18.086$ (2), $b = 8.294$ (1), $c = 8.229$ (1) Å. $M_r = 306.1$, $Z = 4$, $D_x = 1.65$ g cm^{-3}, μ(Mo $K\alpha$) = 29.1 cm^{-1}. The structure was refined to $R = 0.05$. The geometry of the pentacoordinate As atom can be described as a 74% rectangular pyramid with the methyl group in apical position. The molecule contains *trans*-basal angles O–As–O of 158.6 and 143.1° and apical-basal angles C–As–O of 108.6, 108.3, 100.2 and 101.1°.

Introduction. The compound was synthesized by Wieber, Eichhorn & Götz (1973) and recrystallized from CS_2. Photographs showed systematic absences $0kl$ for l odd and $h0l$ for h odd indicative of the space groups *Pcam* (conventional setting: *Pbcm*) and $Pca2_1$. A comparison with the volume of the molecule of the corresponding phosphorane (Wunderlich, 1974) yielded $Z = 4$. The assumption of no crystallographic symmetry of the molecule reduces the alternative to the noncentrosymmetric space group $Pca2_1$ which was later supported by the statistics of the E values. The

intensities of all 1528 symmetry-independent reflections up to $2\theta = 55°$ were collected on a computer-controlled diffractometer (Syntex $P2_1$) in a $\theta:2\theta$ scan mode with Mo $K\alpha$ radiation and a crystal monochromator. 1141 reflections were classified as observed ($F_o \geq 3\sigma_F$). The structure was solved by the heavy-atom method. All H atoms except those of the methyl group could be located from a difference synthesis. The final refinement with fixed (H) and anisotropic (As, O and C) temperature factors converged at $R = 0.050$ (0.074) and $R_w = 0.046$ (0.049) for the observed (all) reflections. Weights were derived from counter statistics by $1/w = \sigma_F^2 + (0.02F_o)^2$; scattering factors were taken from Cromer & Waber (1974). The final atomic parameters are given in Tables 1 and 2.* All calculations were carried out on a Nova 1200 computer with the program system *XTL* (Syntex).