1988). This distance is similar to the values for other TPPO adducts with phenoxy acids, for example, 2.58 (1), 2.61 (1) Å in [(TPPO)(2,4-D)] (Lynch *et al.*, 1992*a*) and 2.54 (1) Å in [(TPPO)(2,4,5-T)] (Lynch *et al.*, 1992*b*). A comparison of ring conformation for molecule *A* in the title compound with the TPPO molecules in other phenoxyacetic acid adducts is made in Table 4.

The authors thank the Australian Research Council, The University of Queensland and the Queensland University of Technology for financial support.

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Structure of 4-[4′-(Dimethylamino)phenylazo]phenyl Acrylate (1) and 4-[4′-(Dimethylamino)phenylazo]phenyl Methacrylate (2)

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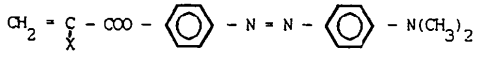
(*Received* 24 *March* 1992; *accepted* 29 *June* 1992)

Abstract. Compound (1): $C_{17}H_{17}N_3O_2$, $M_r = 295.34$, orthorhombic, $Fdd2$, $a = 23.345$ (2), $b = 40.671$ (4), $c = 6.561$ (1) Å, $V = 6229.4$ (12) Å^3, $Z = 16$, $D_x = 1.260$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0.71073 Å, $\mu = 0.8$ cm^{-1}, $F(000) = 2496$, $T = 295$ K, $R_F = 0.086$ for 726 unique observed reflections with $I \geq 1.5\sigma(I)$ and 201 parameters. Compound (2): $C_{18}H_{19}N_3O_2$, $M_r = 309.37$, monoclinic, $C2/c$, $a = 23.949$ (5), $b = 6.804$ (1), $c = 22.893$ (5) Å, $\beta = 117.66$ (2)°, $V = 3304.1$ (13) Å^3, $Z = 8$, $D_x = 1.224$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.8$ cm^{-1}, $F(000) = 1312$, $T = 295$ K, $R_F = 0.086$ for 1294 unique observed reflections with $I \geq 1.5\sigma(I)$ and 265 parameters. Both (1) and (2) have an *E* (*trans*) configuration with the phenyl rings inclined at angles of 13.9 (8) and 10.10 (2)°, respectively. None of the rings is coplanar with the C—N=N—C plane.

Introduction. The crystal structure of the parent compound azobenzene, $C_6H_5N{=}NC_6H_5$, is well known in both its *E* (*trans*) form (Bouwstra, Schouten & Kroon, 1983) and its *Z* (*cis*) form (Mostad & Rømming, 1971), which can be interchanged by irradiation at a particular wavelength. Usually the *E* form of azobenzenes is the more stable. The *Z* form always returns to the *E* configuration on standing, even in the dark, the rate of this back-isomerization being dependent on a number of factors, such as phenyl-ring substitution, solvent polarity and temperature. Because of this instability, azo compounds generally adopt the *E* configuration under normal conditions. The crystal structures of a number of substituted azobenzenes have been described in the literature, but not that of the following polymerizable monomers:



(1) $X = H$
(2) $X = CH_3$

Experimental. Compound (1). To a stirred solution of 4 g (0.0166 mol) of 4-(4-hydroxyphenylazo)-*N*,*N*-

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dimethylaniline (3) and 1.85 g (0.0183 mol) of triethylamine as HCl scavenger in 150 ml THF, blanketed with N_2 and cooled to 273 K, was added dropwise a solution of 1.67 g (0.0183 mol) of acryloyl chloride in 50 ml THF over a period of 3 h. After 6 h the reaction mixture was filtered and extracted with a solution of three parts chloroform to two parts distilled water. The orange-coloured $CHCl_3$ layer was separated and dried on Na_2SO_4, filtered, the solvent evaporated, and the remaining solid dried at 313 K *in vacuo*. It was recrystallized by dissolution in 25 ml $CHCl_3$ at 313 K and diluted with 100 ml *n*-hexane. On cooling, dark brown crystals of unreacted (3) separated, which were removed by filtration. The residual solution was again heated to 313 K, diluted with another 200 ml *n*-hexane and kept standing at 258 K for 15 h. Long orange needles of monomer (1) developed, were separated, washed with cold *n*-hexane and dried at 313 K. The yield was 69%; m.p. 386 K.

Compound (2). The procedure was identical to that for (1) except for the use of methacryloyl chloride instead of acryloyl chloride. The yield of purified material was 75%; m.p. 396 K.

Although the scattering power of the studied crystals was very weak and the single-crystal fragments were small, a successful structure analysis was carried out. The crystals selected for characterization and data collection were orange-coloured fragments of dimensions 0.08 × 0.12 × 0.38 mm and 0.07 × 0.12 × 0.13 mm for (1) and (2), respectively, cleaved from a bunch of needles. The crystal was glued on top of a glass fibre and transferred to an Enraf–Nonius CAD-4*F* diffractometer interfaced to a VAX-11/730 computer. Graphite-monochromated Mo $K\bar{\alpha}$ radiation was used to obtain 1478 reflections (h 0 to 22, k 0 to 48, l 0 to 7); $1.0 < \theta < 25.0°$; $\omega/2\theta$ scan, $\Delta\omega = (0.60 + 0.34 \tan\theta)°$, in the case of (1); and 3248 reflections (h −28 to 24, k 0 to 8, l 0 to 27); $1.05 < \theta < 25°$; $\omega/2\theta$ scan, $\Delta\omega = (0.85 + 0.34 \tan\theta)°$, for (2). Unit-cell parameters with e.s.d.'s were determined from a least-squares treatment of the setting angles of 22 reflections in the range $7.25 < \theta < 12.42°$ in four alternative settings (de Boer & Duisenberg, 1984) for (1) and 15 reflections with $9.65 < \theta < 19.29°$ for (2). The unit cell of (1) was identified as orthorhombic in space group *Fdd*2; the *E* statistics showed a non-centrosymmetric space group (Snow & Teikink, 1988). This choice was confirmed by the solution and successful refinement in this space group. The space group *C*2/*c* of (2) was derived from the observed systematic absences. Reduced cell calculations did not indicate any higher metrical lattice symmetry (Spek, 1988) and examination of the final atomic coordinates of the structure did not yield extra metric symmetry elements (Le Page, 1987). Crystal and/or instrumental instability was monitored by measurement of the intensities of three reference reflections collected every 3 h of X-ray exposure time; there was no indication of crystal decomposition. A 360° ψ scan for the reflection close to axial ($\bar{1}\bar{3}3$) of (1) showed an intensity variation of up to 8% about the mean value. The net intensities of the data were corrected for scale variation, Lorentz and polarization effects, but not for absorption. Standard deviation $\sigma(I)$ in the intensities was increased according to analysis of the excess variance of the reference reflection in the case of (1) and of the three reference reflections in the case of (2): $\sigma^2(I) = \sigma^2_{cs}(I) + (0.0093I)^2$. The variance was calculated based on counting statistics and the term (P^2I^2), where $P = 0.019$ is the instability constant (McCandlish, Stout & Andrews, 1975) derived from the excess variance in the reference reflections. This results in 726 reflections for (1) satisfying the $I \geq 1.5\sigma(I)$ criterion of observability. To provide an adequate observation-to-parameter ratio, only reflections where F_o was less than $1.5\sigma(F)$ were considered unobserved and were not included in the refinement, although this low cutoff leads to the higher *R* factors.

The structures were solved by direct methods with *SHELXS*86 (Sheldrick, 1990) using the 'brute force approach'. The positional and anisotropic thermal displacement parameters for non-H atoms were refined with block-diagonal least-squares procedures (*CRYLSQ*; Olthof-Hazekamp, 1990) minimizing the function $Q = \sum_h [w(|F_o| - k|F_c|)^2]$. The positions for some of the H atoms in (1) were provided in subsequent difference Fourier maps and the remainder were calculated at idealized geometric positions, whereby the located H atoms served to determine the conformation. The H atoms, except H(16), the positional parameters of which were left free, were included in the final refinements with their positions calculated using sp^2 or sp^3 hybridization at the C atom as appropriate and a fixed C—H distance of 0.98 Å, refined in the riding mode on their carrier atoms with an isotropic thermal displacement parameter common to all. The polarity of the structure was tested by refinement with if'' and $-if''$ values, respectively, giving only marginally different *R* values, probably due to twinning in this respect. In (2), all H atoms located on subsequent difference Fourier maps were included in the final refinement with one overall temperature factor.

Weights were introduced in the final refinement cycles. Refinement on *F* was carried out by full-matrix least-squares techniques with anisotropic thermal displacement parameters for the non-H atoms and one overall isotropic thermal displacement parameter for the H atoms, and converged at $R_F = 0.086$ and 0.086 [$wR = 0.056$ and 0.038; $w = 1/\sigma^2(F)$]; $S = 1.860$ and 1.784; average

Table 1. *Final fractional atomic coordinates for atoms of the asymmetric unit and equivalent isotropic thermal displacement parameters* (Å^2) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Compound (1)				
O(1)	0.4535 (4)	0.0384 (3)	−0.60510 (–)	0.082 (5)
O(2)	0.5265 (4)	0.0687 (2)	−0.506 (2)	0.080 (5)
N(1)	0.1837 (5)	0.0784 (3)	0.894 (3)	0.068 (6)
N(2)	0.3145 (5)	0.0720 (3)	0.185 (2)	0.057 (5)
N(3)	0.3275 (4)	0.0436 (3)	0.118 (2)	0.046 (4)
C(1)	0.2150 (6)	0.0759 (4)	0.715 (3)	0.058 (6)
C(2)	0.2276 (5)	0.0451 (4)	0.632 (3)	0.063 (6)
C(3)	0.2598 (5)	0.0439 (3)	0.461 (3)	0.042 (5)
C(4)	0.2800 (6)	0.0714 (4)	0.358 (3)	0.053 (7)
C(5)	0.2653 (6)	0.1019 (4)	0.441 (3)	0.065 (7)
C(6)	0.2336 (6)	0.1039 (4)	0.619 (3)	0.058 (6)
C(7)	0.3610 (6)	0.0448 (4)	−0.057 (4)	0.065 (8)
C(8)	0.3858 (6)	0.0152 (4)	−0.117 (3)	0.067 (7)
C(9)	0.4176 (6)	0.0135 (4)	−0.295 (3)	0.061 (7)
C(10)	0.4263 (6)	0.0408 (5)	−0.408 (2)	0.060 (7)
C(11)	0.4029 (6)	0.0710 (4)	−0.361 (3)	0.065 (7)
C(12)	0.3709 (6)	0.0720 (4)	−0.180 (3)	0.063 (7)
C(13)	0.1680 (5)	0.0492 (3)	1.002 (3)	0.079 (7)
C(14)	0.1716 (6)	0.1089 (3)	0.993 (3)	0.089 (7)
C(15)	0.5015 (7)	0.0550 (4)	−0.631 (4)	0.080 (9)
C(16)	0.5173 (9)	0.0544 (4)	−0.856 (4)	0.0981 (1)
C(17)	0.5559 (7)	0.0694 (4)	−0.933 (3)	0.1211 (1)
Compound (2)				
O(1)	0.08972 (13)	0.6808 (4)	−0.00929 (13)	0.0720 (14)
O(2)	0.02893 (12)	0.5297 (5)	−0.10515 (13)	0.0770 (12)
N(1)	0.37653 (16)	−0.7702 (5)	0.22553 (17)	0.0679 (17)
N(2)	0.24513 (15)	−0.0909 (5)	0.09856 (15)	0.0604 (17)
N(3)	0.21260 (14)	−0.0039 (6)	0.12089 (16)	0.0620 (17)
C(1)	0.34412 (18)	−0.6057 (6)	0.1943 (2)	0.0568 (19)
C(2)	0.30978 (18)	−0.4999 (7)	0.21930 (19)	0.600 (19)
C(3)	0.27732 (18)	−0.3308 (7)	0.19024 (19)	0.0553 (19)
C(4)	0.27679 (18)	−0.2586 (6)	0.13322 (19)	0.0539 (19)
C(5)	0.31038 (19)	−0.3607 (7)	0.1080 (2)	0.065 (2)
C(6)	0.34300 (19)	−0.5299 (7)	0.1363 (2)	0.063 (2)
C(7)	0.18210 (18)	0.1665 (7)	0.08444 (19)	0.0577 (19)
C(8)	0.1411 (2)	0.2589 (7)	0.1022 (2)	0.074 (2)
C(9)	0.10777 (19)	0.4271 (7)	0.0687 (2)	0.071 (2)
C(10)	0.11922 (18)	0.5002 (7)	0.0194 (2)	0.0616 (19)
C(11)	0.16066 (19)	0.4143 (7)	0.0027 (2)	0.072 (3)
C(12)	0.19151 (19)	0.2471 (7)	0.0346 (2)	0.069 (2)
C(13)	0.3704 (2)	−0.8577 (8)	0.2793 (2)	0.090 (3)
C(14)	0.4091 (2)	−0.8838 (7)	0.1983 (2)	0.089 (3)
C(15)	0.04585 (18)	0.6758 (7)	−0.07372 (19)	0.056 (2)
C(16)	0.02299 (17)	0.8767 (6)	−0.9951 (19)	0.058 (2)
C(17)	−0.0136 (2)	0.8888 (7)	−0.1716 (2)	0.095 (3)
C(18)	0.0354 (2)	1.0276 (8)	−0.0598 (2)	0.097 (3)

$\Delta/\sigma = 0.149$ and 0.048; max. $\Delta/\sigma = 0.948$ and 0.257, for (1) and (2), respectively. Final difference Fourier maps did not show residual peaks outside the range ± 0.33 e Å^{-3}. Scattering factors were taken from Cromer & Mann (1968). Anomalous-dispersion factors given by Cromer & Liberman (1970) were included in F_c. All calculations were carried out on the CDC-Cyber 962-31 computer at the University of Groningen using the program packages *XTAL* (Hall & Stewart, 1990) and *PLATON* (Spek, 1990) for calculation of geometric data, and a locally modified version (Meetsma, 1991) of the *PLUTO* program (Motherwell & Clegg, 1978) for the preparation of illustrations.

Discussion. The final fractional atomic coordinates and equivalent isotropic thermal displacement parameters for non-H atoms are given in Table 1.* Bond lengths, angles and selected torsion angles are listed in Table 2.

The configuration and the atom-labelling scheme of (1) are shown in the *PLUTO* drawing in Fig. 1 and those of (2) in Fig. 2. The packing of the molecules in the unit cell of (1) and of (2) are shown in Figs. 3 and 4, respectively. Each asymmetric unit contains one complete molecule with no atom at a special position. The orthorhombic unit cell of (1) contains 16 discrete molecules whereas the monoclinic unit cell of (2) contains eight. In both cells, the molecules are separated by normal van der Waals distances.

Both compounds have an *E* configuration. In (1), the N(2)—C(4) and N(3)—C(7) bond lengths of 1.39 (3) Å, which are very similar to that of N(1)—C(1), are short compared with the average value of 1.437 Å in other *E* azo compounds (Mostad & Rømming, 1971). However, the N=N length of 1.273 (17) Å is quite normal as are the C—N=N angles of 113.8 (12) and 112.8 (12)°. The dimethyl-

* Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55609 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1013]

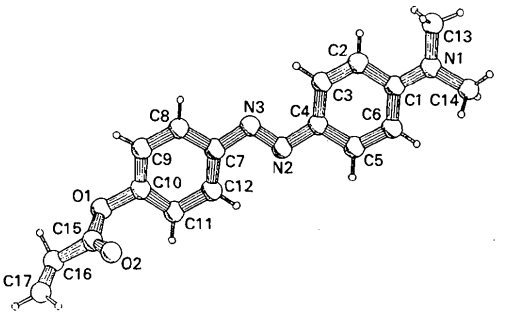

Fig. 1. *PLUTO* drawing of molecule (1) illustrating the conformation and showing the atom-numbering scheme.

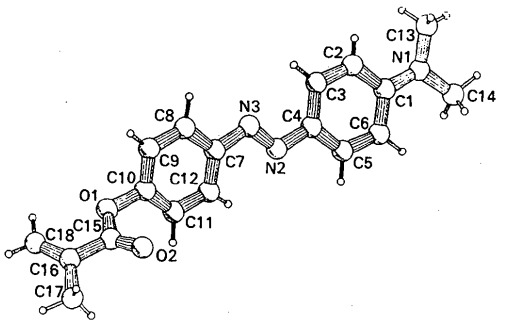

Fig. 2. *PLUTO* drawing of molecule (2) illustrating the conformation and showing the adopted numbering scheme.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

	Compound (1)	Compound (2)		Compound (1)	Compound (2)
O(1)—C(10)	1.444 (14)	1.418 (5)	C(3)—C(4)	1.39 (2)	1.389 (6)
O(1)—C(15)	1.319 (19)	1.357 (5)	C(4)—C(5)	1.40 (2)	1.376 (7)
O(2)—C(15)	1.15 (2)	1.183 (6)	C(5)—C(6)	1.38 (3)	1.373 (7)
N(1)—C(1)	1.39 (3)	1.361 (5)	C(7)—C(8)	1.39 (2)	1.377 (7)
N(1)—C(13)	1.43 (2)	1.435 (6)	C(7)—C(12)	1.39 (3)	1.376 (6)
N(1)—C(14)	1.43 (2)	1.430 (6)	C(8)—C(9)	1.39 (3)	1.402 (7)
N(2)—N(3)	1.273 (17)	1.260 (5)	C(9)—C(10)	1.35 (3)	1.372 (6)
N(2)—C(4)	1.39 (2)	1.396 (5)	C(10)—C(11)	1.38 (3)	1.351 (7)
N(3)—C(7)	1.39 (3)	1.417 (6)	C(11)—C(12)	1.40 (3)	1.368 (7)
C(1)—C(2)	1.40 (2)	1.400 (7)	C(15)—C(16)	1.52 (4)	1.489 (6)
C(1)—C(6)	1.37 (2)	1.413 (6)	C(16)—C(17)	1.20 (3)	1.468 (6)
C(2)—C(3)	1.35 (2)	1.375 (7)	C(16)—C(18)		1.311 (6)

	Compound (1)	Compound (2)
C(10)—O(1)—C(15)	117.0 (14)	116.7 (3)
C(1)—N(1)—C(13)	119.6 (13)	122.1 (4)
C(1)—N(1)—C(14)	123.6 (14)	120.6 (4)
C(13)—N(1)—C(14)	116.4 (16)	116.3 (4)
N(3)—N(2)—C(4)	113.8 (12)	115.4 (3)
N(2)—N(3)—C(7)	112.8 (12)	113.1 (3)
N(1)—C(1)—C(2)	120.4 (15)	120.6 (4)
N(1)—C(1)—C(6)	119.6 (15)	123.5 (4)
C(2)—C(1)—C(6)	119.9 (17)	116.0 (4)
C(1)—C(2)—C(3)	118.2 (15)	122.7 (4)
C(2)—C(3)—C(4)	124.3 (14)	120.5 (4)
N(2)—C(4)—C(3)	127.4 (15)	126.1 (4)
N(2)—C(4)—C(5)	116.4 (15)	116.3 (4)
C(3)—C(4)—C(5)	116.2 (16)	117.6 (4)
C(4)—C(5)—C(6)	120.8 (16)	122.7 (4)
C(1)—C(6)—C(5)	120.5 (16)	120.5 (4)
N(3)—C(7)—C(8)	115.9 (16)	116.3 (4)
N(3)—C(7)—C(12)	127.0 (15)	125.2 (4)
C(8)—C(7)—C(12)	117.1 (19)	118.5 (4)
C(7)—C(8)—C(9)	120.3 (17)	120.9 (4)
C(8)—C(9)—C(10)	120.2 (15)	117.7 (4)
O(1)—C(10)—C(9)	120.1 (16)	116.6 (4)
O(1)—C(10)—C(11)	115.8 (15)	121.2 (4)
C(9)—C(10)—C(11)	123.4 (14)	122.0 (4)
C(10)—C(11)—C(12)	115.2 (15)	119.6 (4)
C(7)—C(12)—C(11)	123.9 (16)	121.2 (5)
O(1)—C(15)—O(2)	126 (2)	123.8 (4)
O(1)—C(15)—C(16)	108.8 (17)	111.4 (4)
O(2)—C(15)—C(16)	125.2 (17)	124.8 (4)
C(15)—C(16)—C(17)	126 (2)	114.5 (4)
C(15)—C(16)—C(18)		121.4 (4)
C(17)—C(16)—C(18)		124.1 (4)
C(4)—N(2)—N(3)—C(7)	178.8 (13)	179.3 (4)
C(2)—C(1)—N(1)—C(13)	5 (2)	8.7 (6)
C(2)—C(1)—N(1)—C(14)	176.8 (14)	176.9 (4)
C(6)—C(1)—N(1)—C(13)	−175.4 (14)	−171.9 (4)
C(6)—C(1)—N(1)—C(14)	−3 (2)	−3.6 (7)
N(1)—C(1)—C(2)—C(3)	−177.9 (14)	178.7 (4)
N(1)—C(1)—C(6)—C(5)	179.5 (15)	−178.6 (4)
C(2)—C(3)—C(4)—N(2)	178.1 (15)	179.1 (4)
C(3)—C(4)—N(2)—N(3)	1 (2)	−2.7 (6)
C(5)—C(4)—N(2)—N(3)	178.7 (13)	177.3 (4)
N(2)—C(4)—C(5)—C(6)	−176.7 (14)	−179.0 (4)
C(8)—C(7)—N(3)—N(2)	168.3 (14)	174.2 (4)
C(12)—C(7)—N(3)—N(2)	−14 (2)	−7.4 (6)
N(3)—C(7)—C(8)—C(9)	176.8 (15)	−179.1 (4)
N(3)—C(7)—C(12)—C(11)	−176.5 (16)	−178.9 (4)
C(8)—C(9)—C(10)—O(1)	−172.6 (13)	−174.0 (4)
C(9)—C(10)—O(1)—C(15)	−120.5 (17)	−114.7 (5)
C(11)—C(10)—O(1)—C(15)	69.0 (17)	70.8 (6)
O(1)—C(10)—C(11)—C(12)	172.9 (13)	175.5 (4)
O(2)—C(15)—O(1)—C(10)	8 (2)	4.4 (7)
C(16)—C(15)—O(1)—C(10)	−169.8 (13)	−174.6 (4)
O(1)—C(15)—C(16)—C(17)	173.5 (18)	167.1 (4)
O(2)—C(15)—C(16)—C(17)	−4 (3)	−11.9 (7)
O(1)—C(15)—C(16)—C(18)		−12.8 (6)
O(2)—C(15)—C(16)—C(18)		168.2 (5)

amino skeleton is nearly coplanar with the C(1)–C(6) ring, whereas the acryloyl moiety makes roughly an angle of 70° with the C(7)–C(12) ring. The planar phenyl rings appear to be inclined at an angle of 13.9 (8)° with respect to each other. This can be seen in Fig. 3.

Although (2) has an α CH_3 group instead of an α H atom, its structure differs only slightly from its acrylic counterpart. The inclination between the phenyl rings is somewhat less at 10.10 (2)°. Such relatively large deviations from coplanarity were also found in other *E* azo compounds such as 4-(4-ethoxyphenylazo)phenyl hexanoate with an angle of 10.16° between the rings (Prasad, 1979), the heptanoate derivative with an angle of 15.02° (Shaikh, Shivaprakash, Abdoh & Prasad, 1984) and methyl orange monohydrate monoethanolate with an angle of 10° (Hanson, 1973).

This work was carried out within the framework of the Innovation-Oriented Research Program on

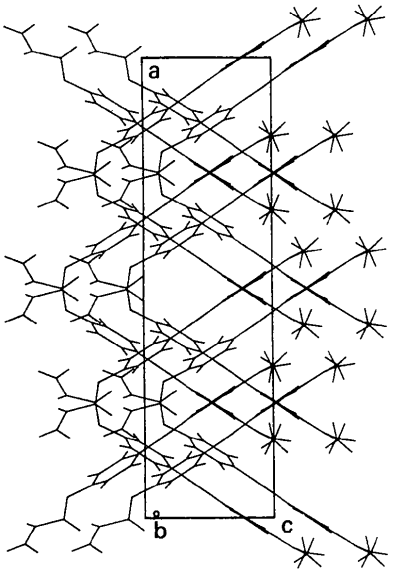

Fig. 3. Projection of the crystal structure of compound (1) down [010].

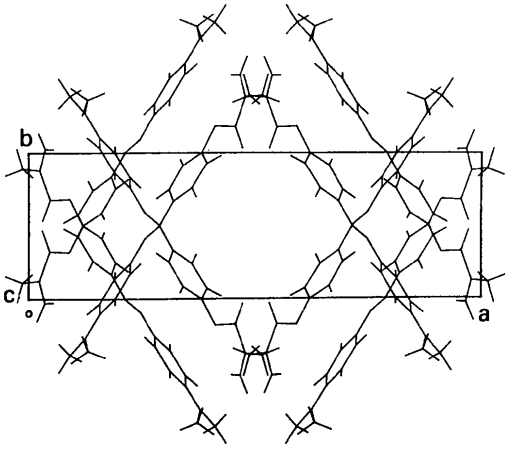

Fig. 4. Projection of compound (2) down [001].

Polymer Composites and Specialty Polymers (IOP-PCBP), Project No. BP-106, with financial aid from the Netherlands Ministry of Economic Affairs.

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Structure of (4-Bromophenyl)propiolic Acid and its Unusual Hydrogen-Bonding Pattern

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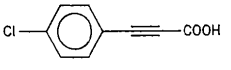
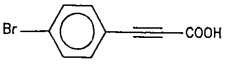
(*Received* 17 *December* 1991; *accepted* 20 *July* 1992)

Abstract. $C_9H_5BrO_2$, $M_r = 225.04$, monoclinic, $P2_1/c$, $a = 3.913$ (20), $b = 6.141$ (2), $c = 34.444$ (27) Å, $\beta = 91.79$ (5)°, $V = 827.2$ (8) Å^3, $Z = 4$, $D_x = 1.81$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 4.870$ mm^{-1}, $F(000) = 440$, $T = 153$ K, $R = 0.030$, $wR = 0.030$ for 1469 independent reflections. The title compound exhibits unusual catemeric hydrogen bonding with both *syn* and *anti* arrangements of the carboxyl group. Such hydrogen bonding is also found in the corresponding chloro acid but the pattern of halogen–halogen contacts is different.

Introduction. We have previously reported that molecules of (4-chlorophenyl)propiolic acid (1) do not pack in the crystal according to the expected carboxylic dimer motif but in an unusual catemeric arrangment (Desiraju, Murty & Kishan, 1990). A possible reason could be the inability of the molecules to form C—H···O hydrogen bonds. Such bonds exist in other carboxylic acids which have the expected carboxylic dimer motif (Leiserowitz, 1976). There is not much crystallographic data on phenylpropiolic acids. The unsubstituted acid (Rollett, 1955) and several alkoxy derivatives examined by us (Desiraju & Kishan, 1989) adopt the dimer motif and it was not clear if the hydrogen-bond pattern exhibited by the chloro acid (1) is an uncommon variant or whether it would be observed in related derivatives. Accordingly, a crystallographic study of the title compound (2) was initiated.



(1)



(2)

Experimental. Acid (2) (m.p. 468–469 K) was prepared from 4-bromobenzaldehyde by literature procedures (Desiraju & Kishan, 1989) and recrystallized from 1:3 acetone–chloroform. Intensity data were collected on an irregularly shaped block 0.4 × 0.4 × 0.3 mm on a Nicolet *R*3*m* diffractometer. Unit-cell parameters were obtained from 25 reflections in the range $5 \le 2\theta \le 30°$. The ω-scan method was used with a scan width of 1.20° and scan speed between 0.30 and 3.32° min^{-1}. 1974 reflections (1886 unique) were collected with $4 < 2\theta < 55°$ and in the range $-5 \le h \le 5$, $0 \le k \le 8$, $0 \le l \le 45$. Of these,

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0108-2701/93/020292-03$06.00 © 1993 International Union of Crystallography