the favorable interaction between the acidic C(2) proton of the thiazolium ring and the π electrons in the pyrimidine ring (Turano, Pletcher, Furey & Sax, 1982). In this context, the present conformation is a rather unexpected one since amprolium apparently does not have an acidic proton in the pyridine ring and thus could adopt the less observed *S* form ($\varphi_T \simeq$ 110 and $\varphi_P \simeq \pm 180°$) of thiamin in which H(6′) sits on top of the thiazolium ring.

It has been pointed out that the relative orientation of the two rings in the thiamin-related molecules is determined mainly by the interactions between the substituents at the four positions [C(2), C(6), C(4′) and C(6′) in amprolium] which are *ortho* to the methylene bridge C atom (Shin & Kim, 1986). Since amprolium and pyrithiamin have the same substituents at these positions, we expect that the conformational property of pyrithiamin may be similar to that of amprolium. Together with the presence of the pyridine ring, which is positively charged like the thiazolium ring but lacking the active site, conformational similarity may be the major factor for the inhibitory power of amprolium and pyrithiamin.

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Molecular Co-crystals of Carboxylic Acids. 10.* Structure of the 1:1 Adduct of Triphenylphosphine Oxide with (Pentachlorophenoxy)acetic Acid

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Abstract. $C_{18}H_{15}OP.C_8H_3Cl_5O_3$, $M_r = 602.7$, triclinic, $P\bar{1}$, $a = 10.185$ (6), $b = 12.036$ (5), $c = 12.194$ (6) Å, $\alpha = 92.21$ (3), $\beta = 108.48$ (3), $\gamma = 106.10$ (3)°, $V = 1349$ (1) Å^3, $Z = 2$, $D_x = 1.483$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.63$ mm^{-1}, $F(000) = 612$, $T = 295$ K, $R = 0.041$ for 3317 observed reflections. The compound was prepared by interacting a 1:1 molar ratio of triphenylphosphine oxide and (pentachlorophenoxy)acetic acid in toluene/ethanol and allowing the mixture to evaporate to dryness. The two organic molecules associate through a single directed hydrogen bond between the carboxylic acid group and the phosphoryl O atom [OH···O 2.541 (4) Å].

Introduction. Triphenylphosphine oxide (TPPO) has been reported as a useful crystallizing aid (Etter & Baures, 1988). This is possible because the molecule is a good acceptor, available for hydrogen-bonding interactions through the phosphoryl O atom, with examples of both O—H and N—H donor-molecule adducts already reported. The conformational flexibility of the TPPO molecule *via* rotation of the three phenyl rings about the C—P bonds makes it ideal for the stabilization of crystal lattices. Interesting comparisons can therefore be made both with the parent molecule (Spek, 1987; Ruban & Zabel, 1976;

* Part 9: Byriel, Kennard, Lynch, Smith & Thompson (1992).
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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
P(1*A*)	0.18948 (9)	0.05650 (7)	0.81704 (7)	51.6 (5)
O(1*A*)	0.2730 (3)	0.0856 (2)	0.7361 (2)	76 (2)
C(11*A*)	0.2582 (3)	0.1631 (3)	0.9452 (3)	48 (2)
C(12*A*)	0.2728 (4)	0.1328 (3)	1.0564 (3)	58 (2)
C(13*A*)	0.3214 (4)	0.2185 (3)	1.1508 (3)	67 (2)
C(14*A*)	0.3555 (3)	0.3342 (3)	1.1372 (3)	62 (2)
C(15*A*)	0.3429 (4)	0.3647 (3)	1.0277 (3)	64 (2)
C(16*A*)	0.2955 (3)	0.2810 (3)	0.9327 (3)	57 (2)
C(21*A*)	0.0017 (3)	0.0472 (2)	0.7483 (2)	49 (2)
C(22*A*)	−0.0751 (4)	0.0931 (2)	0.8033 (3)	56 (2)
C(23*A*)	−0.2194 (4)	0.0831 (3)	0.7478 (4)	74 (3)
C(24*A*)	−0.2886 (5)	0.0285 (4)	0.6361 (4)	84 (3)
C(25*A*)	−0.2153 (6)	−0.0182 (5)	0.5801 (4)	87 (3)
C(26*A*)	−0.0708 (6)	−0.0084 (4)	0.6350 (3)	76 (3)
C(31*A*)	0.1920 (3)	−0.0816 (3)	0.8684 (3)	51 (2)
C(32*A*)	0.0711 (4)	−0.1585 (3)	0.8828 (3)	55 (2)
C(33*A*)	0.0811 (4)	−0.2593 (3)	0.9317 (3)	62 (2)
C(34*A*)	0.2104 (5)	−0.2830 (3)	0.9675 (3)	74 (3)
C(35*A*)	0.3300 (5)	−0.2086 (4)	0.9516 (5)	86 (3)
C(36*A*)	0.3214 (4)	−0.1085 (3)	0.9013 (4)	74 (2)
C(1*B*)	−0.1378 (3)	0.5238 (3)	0.6796 (3)	49 (2)
C(2*B*)	−0.0044 (3)	0.5065 (3)	0.7238 (3)	53 (2)
Cl(2*B*)	0.0204 (1)	0.4139 (1)	0.8273 (1)	84 (1)
C(3*B*)	0.1101 (4)	0.5637 (3)	0.6884 (3)	66 (2)
Cl(3*B*)	0.2751 (1)	0.5403 (1)	0.7436 (1)	117 (1)
C(4*B*)	0.0886 (5)	0.6399 (3)	0.6077 (4)	79 (3)
Cl(4*B*)	0.2310 (2)	0.7182 (1)	0.5685 (1)	152 (1)
C(5*B*)	−0.0455 (6)	0.6556 (3)	0.5601 (3)	74 (3)
Cl(5*B*)	−0.0775 (2)	0.7437 (1)	0.4531 (1)	137 (1)
C(6*B*)	−0.1605 (4)	0.5977 (3)	0.5969 (3)	62 (2)
Cl(6*B*)	−0.3277 (2)	0.6180 (1)	0.5444 (1)	111 (1)
O(7*B*)	−0.2460 (2)	0.4739 (2)	0.7231 (2)	60 (1)
C(8*B*)	−0.3325 (4)	0.3578 (3)	0.6672 (3)	58 (2)
C(9*B*)	−0.4434 (3)	0.3122 (3)	0.7231 (3)	52 (2)
O(10*B*)	−0.4670 (3)	0.3678 (2)	0.7939 (2)	86 (2)
O(11*B*)	−0.5131 (3)	0.2020 (2)	0.6813 (2)	70 (2)

Table 2. *Bond distances* (Å) *and angles* (°)

O(1*A*)—P(1*A*)	1.486 (2)	C(11*A*)—P(1*A*)	1.797 (3)
C(21*A*)—P(1*A*)	1.798 (3)	C(31*A*)—P(1*A*)	1.802 (3)
C(12*A*)—C(11*A*)	1.390 (4)	C(16*A*)—C(11*A*)	1.392 (4)
C(13*A*)—C(12*A*)	1.377 (5)	C(14*A*)—C(13*A*)	1.369 (5)
C(15*A*)—C(14*A*)	1.373 (5)	C(16*A*)—C(15*A*)	1.370 (5)
C(22*A*)—C(21*A*)	1.380 (5)	C(26*A*)—C(21*A*)	1.383 (5)
C(23*A*)—C(22*A*)	1.378 (5)	C(24*A*)—C(23*A*)	1.361 (6)
C(25*A*)—C(24*A*)	1.364 (7)	C(26*A*)—C(25*A*)	1.379 (7)
C(32*A*)—C(31*A*)	1.385 (4)	C(36*A*)—C(31*A*)	1.383 (5)
C(33*A*)—C(32*A*)	1.387 (4)	C(34*A*)—C(33*A*)	1.364 (5)
C(35*A*)—C(34*A*)	1.371 (6)	C(36*A*)—C(35*A*)	1.384 (6)
C(2*B*)—C(1*B*)	1.372 (4)	C(6*B*)—C(1*B*)	1.377 (4)
O(7*B*)—C(1*B*)	1.372 (3)	Cl(2*B*)—C(2*B*)	1.719 (3)
C(3*B*)—C(2*B*)	1.384 (4)	Cl(3*B*)—C(3*B*)	1.707 (4)
C(4*B*)—C(3*B*)	1.378 (6)	Cl(4*B*)—C(4*B*)	1.713 (3)
C(5*B*)—C(4*B*)	1.374 (6)	Cl(5*B*)—C(5*B*)	1.720 (4)
C(6*B*)—C(5*B*)	1.400 (5)	Cl(6*B*)—C(6*B*)	1.709 (4)
C(8*B*)—O(7*B*)	1.435 (4)	C(9*B*)—C(8*B*)	1.487 (4)
O(10*B*)—C(9*B*)	1.193 (4)	O(11*B*)—C(9*B*)	1.311 (4)

C(11*A*)—P(1*A*)—O(1*A*)	112.9 (1)	C(21*A*)—P(1*A*)—O(1*A*)	111.8 (1)
C(21*A*)—P(1*A*)—C(11*A*)	106.4 (1)	C(31*A*)—P(1*A*)—O(1*A*)	112.4 (1)
C(31*A*)—P(1*A*)—C(11*A*)	106.2 (1)	C(31*A*)—P(1*A*)—C(21*A*)	106.7 (1)
C(12*A*)—C(11*A*)—P(1*A*)	122.8 (2)	C(16*A*)—C(11*A*)—P(1*A*)	118.8 (2)
C(16*A*)—C(11*A*)—C(12*A*)	118.4 (3)	C(13*A*)—C(12*A*)—C(11*A*)	120.1 (3)
C(14*A*)—C(13*A*)—C(12*A*)	121.0 (4)	C(15*A*)—C(14*A*)—C(13*A*)	119.2 (3)
C(16*A*)—C(15*A*)—C(14*A*)	120.9 (3)	C(15*A*)—C(16*A*)—C(11*A*)	120.5 (3)
C(22*A*)—C(21*A*)—P(1*A*)	123.2 (2)	C(26*A*)—C(21*A*)—P(1*A*)	119.2 (3)
C(26*A*)—C(21*A*)—C(22*A*)	117.7 (4)	C(23*A*)—C(22*A*)—C(21*A*)	121.4 (3)
C(24*A*)—C(23*A*)—C(22*A*)	120.0 (5)	C(25*A*)—C(24*A*)—C(23*A*)	119.8 (5)
C(26*A*)—C(25*A*)—C(24*A*)	120.5 (4)	C(25*A*)—C(26*A*)—C(21*A*)	120.6 (5)
C(32*A*)—C(31*A*)—P(1*A*)	122.2 (2)	C(36*A*)—C(31*A*)—P(1*A*)	118.8 (2)
C(36*A*)—C(31*A*)—C(32*A*)	118.8 (3)	C(33*A*)—C(32*A*)—C(31*A*)	120.4 (3)
C(34*A*)—C(33*A*)—C(32*A*)	120.3 (4)	C(35*A*)—C(34*A*)—C(33*A*)	119.8 (4)
C(36*A*)—C(35*A*)—C(34*A*)	120.6 (4)	C(35*A*)—C(36*A*)—C(31*A*)	120.0 (4)
C(6*B*)—C(1*B*)—C(2*B*)	120.0 (3)	O(7*B*)—C(1*B*)—C(2*B*)	120.2 (3)
O(7*B*)—C(1*B*)—C(6*B*)	119.6 (3)	Cl(2*B*)—C(2*B*)—C(1*B*)	118.8 (2)
C(3*B*)—C(2*B*)—C(1*B*)	121.2 (3)	C(3*B*)—C(2*B*)—Cl(2*B*)	120.0 (3)
Cl(3*B*)—C(3*B*)—C(2*B*)	120.6 (3)	C(4*B*)—C(3*B*)—C(2*B*)	118.8 (3)
C(4*B*)—C(3*B*)—Cl(3*B*)	120.6 (3)	Cl(4*B*)—C(4*B*)—C(3*B*)	119.9 (4)
C(5*B*)—C(4*B*)—C(3*B*)	120.6 (3)	C(5*B*)—C(4*B*)—Cl(4*B*)	119.5 (4)
Cl(5*B*)—C(5*B*)—C(4*B*)	121.4 (3)	C(6*B*)—C(5*B*)—C(4*B*)	120.1 (3)
C(6*B*)—C(5*B*)—Cl(5*B*)	118.5 (4)	C(5*B*)—C(6*B*)—C(1*B*)	119.2 (3)
Cl(6*B*)—C(6*B*)—C(1*B*)	118.8 (3)	Cl(6*B*)—C(6*B*)—C(5*B*)	122.0 (3)
C(8*B*)—O(7*B*)—C(1*B*)	113.9 (2)	C(9*B*)—C(8*B*)—O(7*B*)	109.0 (3)
O(10*B*)—C(9*B*)—C(8*B*)	125.3 (3)	O(11*B*)—C(9*B*)—C(8*B*)	109.6 (3)
O(11*B*)—C(9*B*)—O(10*B*)	125.1 (3)		

Bandoli, Bortolozzo, Clemente, Croatto & Panattoni, 1970) and with other known TPPO adducts. (Pentachlorophenoxy)acetic acid (PCPA) is an inactive member of the phenoxyalkanoic acid series which includes a number of commercial herbicides (Spencer, 1973). Because of uncompromising crystal morphology, the structure of (pentachlorophenoxy)acetic acid has not previously been determined. However, structural comparisons can be made with the other known phenoxyacetic acid having Cl atoms in both the *ortho* ring positions, (2,4,6-trichlorophenoxy)acetic acid [2,4,6-T (Smith, Kennard & White, 1977)]. The structures of two TPPO adducts with chloro-substituted phenoxyacetic acids are also known. These are with (2,4-dichlorophenoxy)acetic acid {[(TPPO)(2,4-D)] (Lynch, Smith, Byriel & Kennard, 1992*a*)} and with (2,4,5-trichlorophenoxy)acetic acid {[(TPPO)(2,4,5-T)] (Lynch, Smith, Byriel & Kennard, 1992*b*)}.

Experimental. The title compound was prepared by refluxing equimolar amounts of triphenylphosphine oxide (TPPO) and (2,3,4,5,6-pentachlorophenoxy)-acetic acid (PCPA) in 50% ethanol/toluene for 20 min at 423 K. Colourless plates (m.p. 404–406 K) were obtained after evaporation to dryness at room temperature. PCPA was synthesized, using the general procedure of Synerholm & Zimmerman (1945), from pentachlorophenol and ethyl bromoacetate followed by base hydrolysis. Pentachlorophenol is a commercial pesticide with both insecticidal and herbicidal properties (Spencer, 1973). A crystal with dimensions 0.28 × 0.10 × 0.48 mm was used to collect data at 295 K on an Enraf–Nonius CAD-4 four-circle diffractometer using graphite-monochromatized Mo $K\alpha$ radiation. Cell parameters were obtained using angle data from 25 reflections with $2\theta < 50°$. Data collection details: $2\theta/\omega$ collection mode, variable scanning rate; $2\theta_{max} = 50°$; collection range h 0 to 14, k −14 to 14, l −14 to 14; total data 5023; unique 4732 ($R_{int} = 0.011$); unobserved data 1670; standards variation ($11\bar{7}$, $06\bar{3}$, $\bar{1}7\bar{1}$) −1.9%; 3317 reflections with $I > 2.5\sigma(I)$ used in structure refinement. Data were corrected for Lorentz and polarization effects as well as for absorption (empirical; max. and min. transmission factors 1.00 and 0.96, respectively). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986). Blocked-matrix least-squares refinement (on F) with anisotropic thermal parameters for all non-H atoms gave $R = 0.041$ and $wR = 0.049$ $\{w = 1.0/[\sigma^2(F_o) + 6.3 \times 10^{-4}(F_o)^2]$; $S = 1.72\}$ (*SHELX*76;

Sheldrick, 1976). H atoms were located by difference methods and their positional and isotropic thermal parameters refined. Max. (Δ/σ) in the last cycle was 0.01 for all non-H atoms. Max. and min. difference peaks were 0.30 and -0.57 e Å^{-3}. Neutral-atom scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol, IV). Atom positional and thermal parameters are listed in Table 1* while bond distances and angles are given in Table 2. The atom-numbering scheme is shown in Fig. 1.

Discussion. The structure of the title compound comprises a 1:1 adduct between triphenylphosphine oxide (TPPO, denoted molecule *A*) and (pentachlorophenoxy)acetic acid (PCPA, denoted molecule *B*) linked by a single directed hydrogen bond [graph set *D* (Etter, 1990)] (Fig. 2). The phenoxyalkanoic acid in this structure (PCPA) is conformationally different from the other phenoxy acids in adducts with TPPO. Torsion angles for the oxyacetic acid side chain are compared in Table 3. PCPA is also structurally different from 2,4,6-T, having the oxyacetic acid group perpendicular to the benzene ring [ring to

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55460 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1007]

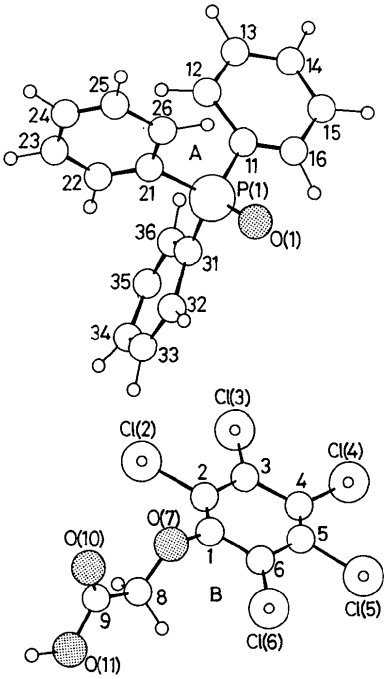

Fig. 1. Molecular conformation and atom-numbering scheme for the adduct pair. Unless othewise indicated, atoms are C. The molecule *A* is TPPO while the molecule *B* is PCPA.

Table 3. *Comparison of torsion angles for PCPA in [(TPPO)(PCPA)] with other TPPO–phenoxyalkanoic acid adducts*

	(PCPA)[a]	(2,4-D)[b] Molecule 1	(2,4-D)[b] Molecule 2	(2,4,5-T)[c]	2,4,6-T*[d]
C(2)—C(1)—O(7)—C(8)	−87.5 (2)	175.4	−176.6	158.7	75.3
C(1)—O(7)—C(8)—C(9)	178.8 (2)	77.2	−85.4	−60.5	−152.3
O(7)—C(8)—C(9)—O(11)	−173.4 (2)	−175.4	176.3	172.1	−151.1

References: (*a*) this work; (*b*) Lynch *et al.* (1992*a*); (*c*) Lynch *et al.* (1992*b*); (*d*) Smith *et al.* (1977).

* Free acid.

Table 4. *Comparative torsion angles for TPPO in adducts with phenoxyalkanoic acids*

	(PCPA)	(2,4-D) Molecule 1	(2,4-D) Molecule 2	(2,4,5-T)
O(1)—P(1)—C(11)—C(12)	−137.6 (2)	116.6	123.1	−47.5
O(1)—P(1)—C(21)—C(22)	−137.8 (2)	−150.5	−27.5	−30.3
O(1)—P(1)—C(31)—C(32)	−141.2 (2)	15.5	156.6	112.0

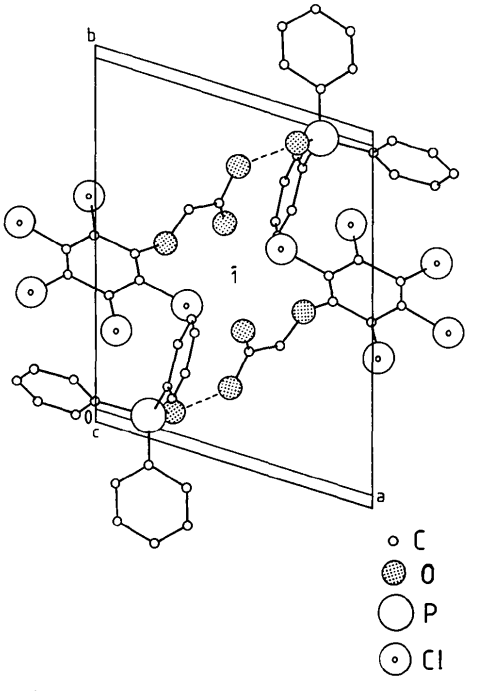

Fig. 2. Packing in the unit cell viewed down **c**.

carboxylic acid dihedral angle 93.2 (1) *cf* 32.0 (1)° for 2,4,6-T]. However, the intramolecular O(ether)···O(carboxyl) distance [O(7*B*)···O(10*B*) 2.676 (4) Å] remains relatively constant, an observation for phenoxyalkanoic acids generally, irrespective of ring substitution or side-chain conformation (Kennard, Smith & White, 1982). PCPA adopts the common *syn–syn* side-chain conformation with the carboxyl H atom located on O(11*B*). The directed hydrogen bond therefore exists between this acid group and the phosphoryl O atom O(1*A*) [2.541 (4) Å; $1+x, y, z$], which is the expected mode of interaction for TPPO with donor molecules in co-crystals (Etter & Baures,

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.

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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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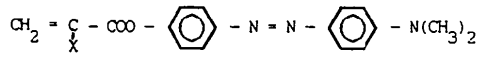
Abstract. Compound (1): $C_{17}H_{17}N_3O_2$, $M_r = 295.34$, orthorhombic, *Fdd*2, $a = 23.345$ (2), $b = 40.671$ (4), $c = 6.561$ (1) Å, $V = 6229.4$ (12) Å^3, $Z = 16$, $D_x = 1.260$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0.71073 Å, $\mu = 0.8$ cm^{-1}, $F(000) = 2496$, $T = 295$ K, $R_F = 0.086$ for 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, *C*2/*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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