mizing $\sum w(F_o - F_c)^2$ with a 6:1 reflection to parameter ratio, showed a max. shift/e.s.d. of 0.11. Max. peak on final difference map was $0.19\ e\ Å^{-3}$, $R = 0.054$, $wR = 0.045$ and $S = 2.40$ with weights based on counting statistics. Scattering factors were taken from Cromer & Waber (1974) and anomalous-dispersion corrections for the non-H atoms were from Cromer (1974).

Discussion. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* The bond distances and angles are listed in Table 2. The structure and labeling of the title compound are shown in Fig. 1 and a stereo packing diagram is given in Fig. 2.

The ureido moiety is planar ($\chi^2 = 3.696$). The plane of the aromatic ring is defined by atoms C(1), C(2), C(3), C(4), C(5), C(6) and makes a dihedral angle of 58.92 (16)° with the plane of the ureido system. This geometry suggests that the lone pair on N(1) is delocalized mainly through the N(1)—C(2′) bond of the ureido group. It would consequently be less conjugated to the aromatic ring [N(1)—C(1) 1.414 (5), N(1)—C(2′) 1.356 (6) Å].

The distance between N(4) and C(2′) is 2.975 (4) Å and the bond angles around N(4) are C(2)—N(4)—H 113.19 and H(1)—N(4)—H 111.90°. These values indicate a pyramidal geometry for N(4).

In the crystal lattice (Fig. 2), the ureido moiety of one molecule is oriented antiparallel to that of another molecule, and the intermolecular distances O(2′)—N(3) and O(2′)—N(1) are 3.009 (5) and 2.855 (5) Å, respectively.

The geometry of the title compound in the solid is similar to that of one of its major conformations in solution, as confirmed by NMR spectroscopy results (Jean-Claude & Just, 1992).

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Structure of Tris(2,4,6-trimethoxyphenyl)phosphine Oxide Hydrate

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Abstract. $C_{27}H_{33}O_{10}P.H_2O$, $M_r = 566.54$, triclinic, $P\bar{1}$, $a = 11.429$ (1), $b = 14.390$ (4), $c = 19.459$ (3) Å, $\alpha = 70.49$ (2), $\beta = 78.14$ (1), $\gamma = 69.69$ (2)°, $V = 2814.5$ Å^3, $Z = 4$, $D_x = 1.337$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.49$ cm^{-1}, $F(000) = 1200$, $T = 293$ K, $R = 0.068$ and $wR = 0.088$ for 6333 observed reflections with $|F^2| > 2\sigma(F^2)$. The asymmetric unit contains two molecules, differing in the orientation of one methoxy group. Water molecules in the crystal are hydrogen bonded to each other and to the O atom of the P═O group [P═O 1.467(3), 1.475 (3) Å, for molecules *A* and *B*, respectively].

Introduction. There has been considerable interest in the use of the basic, hindered phosphine, tris(2,4,6-trimethoxyphenyl)phosphine (TMPP) (Wada

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& Higashizaki, 1984), as a ligand in organometallic chemistry, and the structures of a number of complexes have been determined (Dunbar, Haefner & Pence, 1989; Chen & Dunbar, 1990; Dunbar, Haefner & Burzynski, 1990; Dunbar, Haefner & Quillevéré, 1990; Chen & Dunbar, 1991; Haefner, Dunbar & Bender, 1991). We have been interested in the possible use of phosphine oxides as materials for second harmonic generation (SHG), and we now report the structure of TMPP oxide. The structure of a different polymorph of this oxide has been determined by Dunbar (1992).

Experimental. TMPP was prepared as previously described (Wada & Higashizaki, 1984). TMPPO was prepared by oxidation of the phosphine with H_2O_2 in chloroform (Senear, Valient & Wirth, 1960). Good quality crystals were obtained by recrystallization from a dichloromethane–hexane mixture. A crystal of dimensions 0.30 × 0.40 × 0.13 mm was used for data collection. Unit-cell parameters by least-squares fit of 25 reflections in the range $8 < 2\theta < 12°$, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, θ–2θ scan, $\Delta\theta = (0.8 + 0.35\tan\theta)°$, maximum scan time 1 min, 9898 unique measured reflections for $2 < \theta < 25°$ and h 0 to 13, k −17 to 17, l −23 to 23, 6333 observed reflections with $|F^2| > 2\sigma(F^2)$, $\sigma(F^2) = [\sigma^2(I) + (0.04I)^2]^{1/2}/\mathrm{Lp}$. Two standard reflections measured every hour showed a 0.9% decrease in intensity and no correction was applied to the data. Lorentz and polarization corrections, but no absorption or extinction corrections. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985), refinement by full-matrix least squares on F, non-H atoms with anisotropic thermal parameters. H atoms were held fixed at calculated positions with $U_{\mathrm{iso}} = 1.3U_{\mathrm{eq}}$ for the parent atom. Weighting scheme of $w = 1/\sigma^2(F)$, $\sum w(|F_o| - |F_c|)^2$ minimized, the final residuals were $R = 0.068$, $wR = 0.088$ for 6333 observed reflections, 698 variables, $S = 1.4$, $(\Delta/\sigma)_{\max} = 0.01$, $(\Delta\rho)_{\max,\min} = 0.98$, -0.10 e Å^{-3} on a final difference map. Atomic parameters are given in Table 1* and selected bond distances and angles in Table 2. Fig. 1 shows the molecular structure and the numbering scheme of the two molecules in the asymmetric unit.

Programs from the Enraf–Nonius *SDP-Plus* package (Frenz, 1985) were run on a MicroVAX II computer. Atomic scattering factors from *Interna-*

Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
P1	3188.0 (10)	213.1 (8)	2790.3 (6)	33 (1)
O1	4016 (3)	622 (2)	2167 (2)	41 (3)
O2	6008 (3)	−782 (2)	2923 (2)	53 (3)
O3	5765 (3)	−3187 (3)	5228 (2)	68 (4)
O4	2034 (3)	−995 (3)	4057 (2)	55 (3)
O5	1388 (3)	2080 (2)	2122 (2)	46 (3)
O6	421 (4)	3908 (3)	3865 (2)	76 (3)
O7	3326 (3)	486 (3)	4295 (2)	59 (3)
O8	3906 (3)	−1266 (2)	2007 (2)	55 (3)
O9	−187 (3)	−890 (3)	1432 (2)	78 (3)
O10	246 (3)	640 (2)	3121 (2)	46 (3)
C1	3989 (4)	−885 (3)	3488 (2)	34 (3)
C2	5313 (4)	−1240 (3)	3507 (2)	35 (3)
C3	5847 (4)	−2003 (3)	4095 (2)	41 (4)
C4	5124 (4)	−2455 (3)	4673 (2)	42 (4)
C5	3823 (4)	−2169 (3)	4673 (3)	44 (4)
C6	3309 (4)	−1385 (3)	4086 (2)	39 (4)
C7	7331 (5)	−1159 (5)	2906 (4)	77 (6)
C8	5091 (6)	−3628 (5)	5865 (3)	81 (7)
C9	1229 (5)	−1373 (6)	4640 (4)	85 (7)
C10	2315 (4)	1242 (3)	3216 (2)	34 (3)
C11	1492 (4)	2123 (3)	2788 (2)	37 (4)
C12	842 (4)	2982 (3)	3028 (3)	46 (4)
C13	1066 (5)	2998 (3)	3690 (3)	51 (4)
C14	1878 (5)	2175 (4)	4130 (2)	50 (4)
C15	2486 (4)	1304 (3)	3882 (2)	40 (4)
C16	436 (5)	2869 (4)	1701 (3)	59 (5)
C17	794 (8)	4088 (5)	4451 (4)	115 (7)
C18	3270 (7)	367 (5)	5044 (3)	87 (7)
C19	2082 (4)	−227 (3)	2509 (2)	34 (3)
C20	2606 (4)	−878 (3)	2051 (2)	40 (4)
C21	1913 (4)	−1136 (3)	1689 (3)	46 (4)
C22	621 (4)	−721 (4)	1775 (3)	52 (4)
C23	53 (4)	−122 (3)	2248 (2)	45 (4)
C24	777 (4)	103 (3)	2622 (2)	36 (4)
C25	4564 (6)	−1728 (4)	1447 (3)	69 (6)
C26	327 (6)	−1442 (5)	898 (3)	96 (6)
C27	−1066 (5)	1139 (4)	3170 (3)	63 (5)
P1*a*	6685.5 (10)	4292.8 (8)	2039.4 (6)	30 (1)
O1*a*	7373 (3)	3797 (2)	2696 (2)	39 (2)
O2*a*	8581 (3)	5318 (2)	2142 (2)	48 (3)
O3*a*	7747 (3)	8130 (2)	31 (2)	59 (3)
O4*a*	5705 (3)	5489 (2)	667 (2)	44 (2)
O5*a*	5800 (3)	2543 (2)	2631 (2)	46 (3)
O6*a*	7213 (3)	1072 (2)	604 (2)	67 (3)
O7*a*	8266 (3)	3960 (2)	609 (2)	47 (3)
O8*a*	5762 (3)	5764 (2)	2809 (2)	47 (3)
O9*a*	1314 (3)	6406 (3)	2901 (2)	76 (4)
O10*a*	4221 (3)	4133 (2)	1631 (2)	47 (3)
C1*a*	7130 (4)	5392 (3)	1400 (2)	33 (3)
C2*a*	7961 (4)	5829 (3)	1534 (2)	34 (3)
C3*a*	8120 (4)	6742 (3)	1072 (2)	39 (4)
C4*a*	7483 (4)	7251 (3)	458 (2)	39 (4)
C5*a*	6654 (4)	6853 (3)	301 (2)	41 (4)
C6*a*	6493 (4)	5943 (3)	778 (2)	35 (3)
C7*a*	9194 (5)	5854 (4)	2371 (3)	72 (5)
C8*a*	7092 (6)	8710 (4)	−595 (3)	74 (6)
C9*a*	4661 (5)	6133 (4)	263 (3)	56 (4)
C10*a*	6912 (4)	3308 (3)	1590 (2)	32 (3)
C11*a*	6391 (4)	2510 (3)	1957 (2)	36 (3)
C12*a*	6466 (4)	1728 (3)	1660 (3)	44 (4)
C13*a*	7132 (4)	1740 (3)	985 (2)	45 (4)
C14*a*	7763 (4)	2453 (3)	631 (2)	44 (4)
C15*a*	7650 (4)	3239 (3)	931 (2)	37 (4)
C16*a*	5243 (5)	1767 (4)	3053 (3)	65 (5)
C17*a*	6616 (6)	295 (4)	924 (3)	74 (5)
C18*a*	9002 (6)	3940 (5)	−69 (3)	70 (5)
C19*a*	5016 (4)	4918 (3)	2248 (2)	32 (3)
C20*a*	4739 (4)	5623 (3)	2655 (2)	34 (3)
C21*a*	3530 (4)	6142 (3)	2887 (3)	43 (4)
C22*a*	2550 (4)	5947 (4)	2712 (3)	52 (5)
C23*a*	2759 (4)	5271 (4)	2291 (3)	52 (4)
C24*a*	3978 (4)	4766 (3)	2066 (2)	37 (4)
C25*a*	5609 (5)	6375 (4)	3283 (3)	64 (4)
C26*a*	1028 (6)	7018 (5)	3393 (4)	82 (6)
C27*a*	3266 (5)	3706 (4)	1625 (3)	75 (5)
O1*s*	2187 (4)	−3869 (4)	6031 (2)	96 (5)
O2*s*	2602 (13)	5274 (10)	4941 (8)	136 (5)

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55870 (63 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1027]

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

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
P1—O1	1.467 (3)	1.475 (3)	P1—C1	1.804 (4)	1.814 (4)
P1—C10	1.824 (5)	1.822 (5)	P1—C19	1.835 (6)	1.830 (4)
O2—C2	1.360 (5)	1.357 (5)	O2—C7	1.416 (6)	1.421 (8)
O3—C4	1.357 (5)	1.356 (5)	O3—C8	1.397 (7)	1.415 (7)
O4—C6	1.375 (5)	1.366 (7)	O4—C9	1.391 (7)	1.426 (6)
O5—C11	1.348 (6)	1.355 (5)	O5—C16	1.426 (5)	1.412 (6)
O6—C13	1.378 (6)	1.367 (7)	O6—C17	1.422 (10)	1.411 (7)
O7—C15	1.361 (5)	1.362 (6)	O7—C18	1.399 (7)	1.415 (6)
O8—C20	1.389 (5)	1.359 (6)	O8—C25	1.412 (7)	1.424 (7)
O9—C22	1.369 (8)	1.367 (5)	O9—C26	1.407 (8)	1.426 (9)
O10—C24	1.358 (6)	1.366 (6)	O10—C27	1.419 (5)	1.430 (8)
C1—C2	1.424 (6)	1.412 (7)	C1—C6	1.390 (6)	1.395 (6)
C2—C3	1.367 (5)	1.370 (6)	C3—C4	1.369 (6)	1.377 (6)
C4—C5	1.398 (7)	1.391 (8)	C5—C6	1.370 (6)	1.377 (6)
C10—C11	1.404 (5)	1.392 (6)	C10—C15	1.387 (7)	1.395 (6)
C11—C12	1.379 (7)	1.398 (8)	C12—C13	1.372 (8)	1.374 (6)
C13—C14	1.372 (6)	1.373 (7)	C14—C15	1.396 (7)	1.394 (8)
C19—C20	1.408 (7)	1.403 (7)	C19—C24	1.393 (6)	1.409 (7)
C20—C21	1.367 (8)	1.387 (6)	C21—C22	1.384 (6)	1.373 (9)
C22—C23	1.381 (7)	1.403 (9)	C23—C24	1.381 (8)	1.386 (6)
O1—P1—C1	114.6 (2)	114.5 (2)	O1—P1—C10	107.9 (2)	107.9 (2)
O1—P1—C19	112.1 (2)	113.0 (2)	C1—P1—C10	107.7 (2)	110.6 (2)
C1—P1—C19	105.1 (2)	99.9 (2)	C10—P1—C19	109.3 (2)	111.0 (2)
C2—O2—C7	118.4 (4)	117.6 (3)	C4—O3—C8	118.7 (4)	118.2 (5)
C6—O4—C9	120.2 (4)	118.2 (4)	C11—O5—C16	119.1 (4)	119.5 (4)
C13—O6—C17	118.2 (4)	118.9 (4)	C15—O7—C18	119.5 (4)	119.0 (5)
C20—O8—C25	119.6 (5)	119.6 (3)	C22—O9—C26	118.0 (4)	117.6 (5)
C24—O10—C27	119.5 (4)	118.1 (4)	P1—C1—C2	123.8 (3)	124.4 (3)
P1—C1—C6	120.3 (3)	118.9 (4)	C2—C1—C6	115.7 (3)	116.1 (4)
O2—C2—C1	117.1 (3)	117.4 (3)	O2—C2—C3	122.2 (4)	121.6 (5)
C1—C2—C3	120.7 (4)	121.0 (4)	C2—C3—C4	120.9 (4)	120.8 (5)
O3—C4—C3	115.1 (4)	115.4 (5)	O3—C4—C5	123.8 (4)	124.1 (4)
C3—C4—C5	121.1 (4)	120.5 (4)	C4—C5—C6	116.8 (4)	117.9 (4)
O4—C6—C1	113.7 (3)	114.1 (4)	O4—C6—C5	121.5 (4)	122.2 (4)
C1—C6—C5	124.8 (4)	123.7 (5)	P1—C10—C11	116.8 (4)	117.7 (3)
P1—C10—C15	126.7 (3)	125.4 (4)	C11—C10—C15	116.0 (4)	116.7 (4)
O5—C11—C10	115.6 (4)	114.8 (4)	O5—C11—C12	122.0 (4)	122.0 (4)
C10—C11—C12	122.4 (5)	123.1 (4)	C11—C12—C13	118.6 (4)	117.5 (4)
O6—C13—C12	114.4 (4)	124.1 (5)	O6—C13—C14	123.5 (5)	114.3 (4)
C12—C13—C14	122.2 (5)	121.6 (5)	C13—C14—C15	117.7 (5)	119.7 (4)
O7—C15—C10	117.8 (4)	116.7 (5)	O7—C15—C14	119.2 (5)	122.3 (4)
C10—C15—C14	123.0 (4)	121.0 (4)	P1—C19—C20	115.9 (3)	115.5 (4)
P1—C19—C24	127.7 (4)	128.4 (4)	C20—C19—C24	116.0 (5)	116.1 (4)
O8—C20—C19	114.2 (5)	114.4 (3)	O8—C20—C21	122.0 (4)	121.9 (5)
C19—C20—C21	123.8 (4)	123.7 (5)	C20—C21—C22	117.8 (5)	118.1 (5)
O9—C22—C21	124.1 (5)	124.3 (5)	O9—C22—C23	115.0 (4)	114.5 (5)
C21—C22—C23	120.9 (6)	121.2 (4)	C22—C23—C24	120.0 (4)	119.3 (5)
O10—C24—C19	117.4 (5)	117.2 (3)	O10—C24—C23	121.3 (4)	121.2 (5)
C19—C24—C23	121.3 (4)	121.6 (5)			

tional Tables for X-ray Crystallography (1974, Vol. IV). Drawings by *ORTEP* (Johnson, 1976), showing 20% thermal ellipsoids.

Discussion. The unit cell contains two crystallographically distinct TMPP molecules which differ only in the orientation of one methoxy group with respect to the rotation about the C13—O6 bond. Examination of the structures of other complexes of phosphine suggests that this variability is not unusual. There are two water molecule sites in the asymmetric unit (O1*s*, O2*s*); O1*s* is hydrogen bonded to the phosphine oxide [O1*a*···O1*s*, 2.668 (6) Å], and there is also a hydrogen bond between the two water molecules [O1*s*···O2*s*, 2.678 (18) Å]. Water molecule O2*s* has a partial occupancy of 0.44 and could only be refined isotropically. There may be weak interactions between the lattice water molecule and two of the methoxy groups [O3···O2*s* 3.020 (14), O6···O1*s* 2.965 (7) Å], but since the atoms of the solvent in the lattice were not located, it is difficult to be certain of this. The origin of the water in the lattice is not clear and no attempt was made to crystallize the material under strictly anhydrous conditions. The structure of a polymorph has recently been determined (Dunbar, 1992). This is also triclinic, but with $Z = 2$, and two molecules of water hydrogen bonded to each P=O unit. This crystal was grown by slow evaporation of benzene, and the water is again adventitious. The P=O distance [1.497 (2) Å] is a little elongated presumably as a result of the more extensive hydrogen bonding. It is clear that this phosphine oxide is extremely susceptible to acquiring water.

It is interesting to compare this structure with that of TMPPSe and tris(2,6-dimethoxyphenyl)phosphine selenide (Allen, Bell, March & Nowell, 1990). The P—C bond lengths are slightly longer than are seen

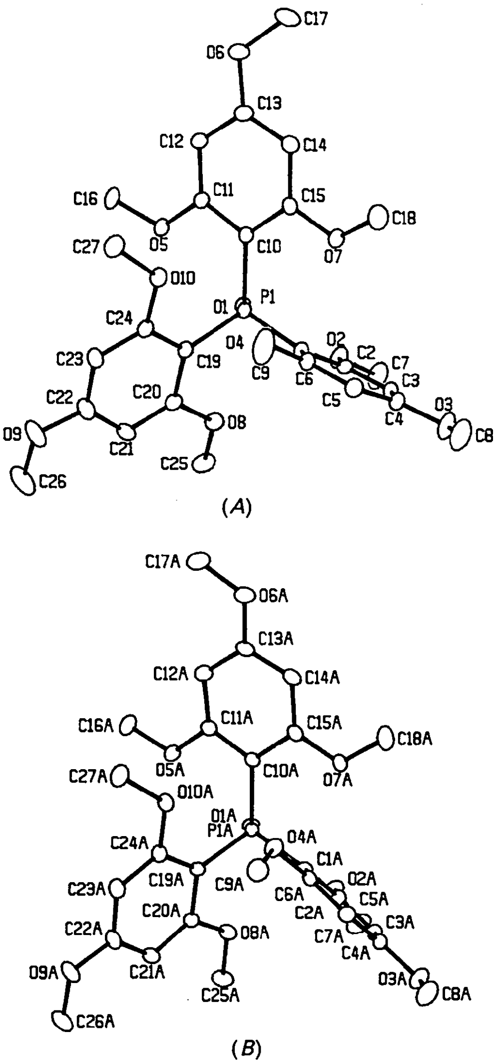

Fig. 1. Molecular structure and numbering scheme of the two TMPPO molecules, (*A*) and (*B*), of the asymmetric unit.

in the title compound. In both these structures there are said to be weak bonding interactions between phosphorus and the O atoms of the *ortho*-methoxy groups with contact distances varying between 2.819 and 2.996 Å. These contacts are attributed to an interaction between the lone pair at the O atom and a dipolar resonance form of the P=Se double bond. Such an interaction was also invoked in the description of the structure of $[Ph_2P(CH_2Ph)(C_6H_4OMe)]Br$, in which the P—O distance is 2.88 Å (Wood, Wilkholm & McEwan, 1977). It might reasonably be expected that such effects would be at least as pronounced in the title compound. In each of the crystallographically distinct molecules there are three shorter [P1—O4 = 2.869 (3), P1—O5 = 2.841 (3) and P1—O8 = 2.823 (4); P1*a*—O4*a* = 2.858 (3), P1*a*—O5*a* = 2.836 (4) and P1*a*—O8*a* = 2.784 (4) Å] and three longer [P1—O2 = 3.066 (3), P1—O7 = 3.124 (4) and P1—O10 = 3.168 (3); P1*a*—O2*a* = 3.082 (4), P1*a*—O7*a* = 3.075 (3) and P1*a*—O10*a* = 3.179 (4) Å] P···O contacts. The shorter set of distances are comparable with the previous data in which some dipolar interaction is invoked.

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Thiophene Chalcone

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Abstract. 3-(4-Methylphenyl)-1-(3-thienyl)-2-propen-1-one, $C_{14}H_{12}OS$, $M_r = 228.31$, orthorhombic, *Fdd*2, $a = 18.787$ (7), $b = 43.46$ (2), $c = 5.828$ (2) Å, $V = 4758$ (3) Å^3, $Z = 16$, $D_x = 1.27$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073, $\mu = 2.35$ cm^{-1}, $F(000) = 1920$, $T = 296$ K, final $R = 0.041$ for 767 observed reflections with $I > 3\sigma(I)$. The torsion angle is 4° for O—C(3)—C(2)—C(1) of the central C_2H_2CO group. The dihedral angle between the phenyl ring and thiophene is 7.85°.

Introduction. The chalcone derivatives are newly developed organic crystals with nonlinear optical coefficients (Fichou, Watanabe, Takeda, Miyata, Goto & Nakayama, 1988). In order to explore the relationship between their structure and nonlinear optical properties, we synthesized a series of substituted chalcones. The title compound is one of them. The second harmonic generation (SHG) efficiencies of the compound were measured by Kurtz & Perry (1968) powder technique; the intensity is three times larger than that of KDP ($d_{36} = 0.63$ pm V^{-1}) standard. The effect is not particularly strong. The crystal structure is determined and discussed.

Experimental. The title compound was prepared by acyloin condensation of *p*-methylbenzaldehyde with 3-acetylthiophene at room temperature (Migrdichian, 1957). The crystals were obtained from ethanol solution by evaporation. A colourless transparent prismatic crystal with approximate dimen-

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