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# *p*-Chloro-, *p*-bromo- and two polymorphs of *p*-iodoacetophenone

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The title *p*-haloacetophenones, $C_8H_7XO$ (*X* = Cl, Br and I), have different packing modes. The chloro compound contains H···O and H···Cl contacts, but no Cl···O contacts. The bromo compound and one polymorph (*A*) of the iodo compound are isomorphous, with significant *X*···O contacts [Br···O = 3.320 (4) Å and I···O = 3.374 (5) Å]. In the other polymorph (*B*) of the iodo compound, the I···O distance is 3.082 (4) Å. Both polymorphs contain C—H···$\pi$ contacts; these contacts are shorter in *A* than in *B*.

## Comment

The structure determinations of *p*-chloro-, *p*-bromo (Britton, 1994) and *p*-iodobenzaldehyde (Britton & Young, 1997) were undertaken in a search for examples of O···*X* interactions. The iodo compound exhibits such an interaction, with O···I distances of 3.07 Å, but the chloro and bromo compounds have *X*···*X* contacts at the halogens and weak C—H···O hydrogen bonds at the aldehyde group. We report here the structures of the corresponding acetophenones. This work was undertaken with the expectation that replacement of the aldehyde H atom with a methyl group would make the O···*X* interaction more likely in the chloro and bromo compounds. The structure of the bromo compound has been reported previously (Lipkowski & Tabaszewska, 1992) but has been repeated here so that all the structure determinations are at the same temperature.
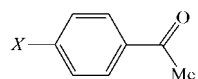
Fig. 1 shows the atom-labeling scheme and anisotropic displacement ellipsoids for the chloro, bromo and two polymorphs of the iodo compound (ClM, BrM, IM-*A* and IM-*B*, respectively). All of the bond distances and angles are normal and, with the exception of the C4—*X* distances, are the same, within experimental error, in all four compounds. The exocyclic C2—C1—C7 angles [mean 119.6 (4)°] are signifi-
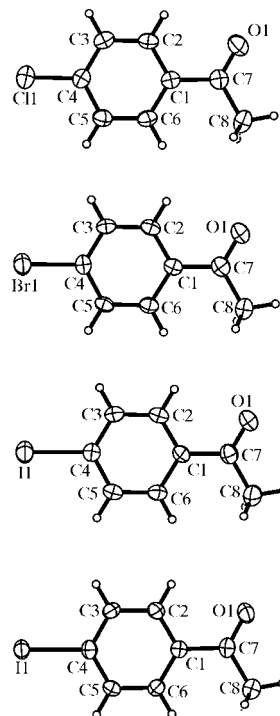


**Figure 1**
Views of the molecules (top to bottom) of ClM, BrM, IM-*A* and IM-*B*.

cantly smaller than the C6—C1—C7 angles [mean 121.9 (4)°]. The dihedral angle between the plane of the ring and the acetyl group is 4.2 (2)° in ClM, 9.4 (3)° in BrM, 10.6 (4)° in IM-*A* and 2.9 (3)° in IM-*B*.

ClM forms $\pi$ stacks parallel to the *a* axis (see Fig. 2), in which the rings are 3.473 (2) Å apart. The molecules are held together by C—H···O and C—H···Cl interactions. All of the C—H···O interactions shorter than 2.8 Å and all of the C—H···Cl interactions shorter than 3.2 Å are shown as dotted lines in Fig. 2. There are no short O···Cl or Cl···Cl contacts. The Cl atom appears to be a weaker Lewis acid than the CH group in this compound, which is somewhat surprising. The metric data for the interactions are given in Table 1; the intramolecular distances are given for comparison.
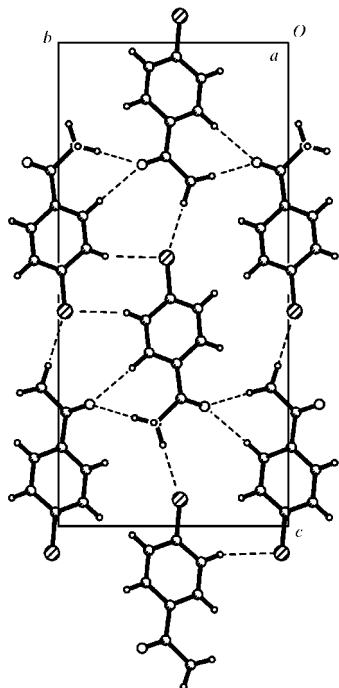
The packing in the remaining compounds is significantly different. The C—H···Cl interactions in ClM are replaced by C—*X*···O—C interactions in BrM, IM-*A* and IM-*B*. These interactions are shown in Fig. 3 and the geometric data are given in Table 2, along with comparison data from three other compounds with short I···O contacts.

BrM and IM-*A* are isomorphous, containing chains of molecules parallel to the [10$\bar{1}$] direction held together by *X*···O interactions. IM-*B* contains similar chains parallel to the [001] direction. Successive molecules in the chains are rotated with respect to each other by 44.9 (1)° in BrM, 44.0 (1)° in IM-*A* and 71.8 (1)° in IM-*B*. Geometric details of intermolecular interactions involving CH groups are included in Table 1. Since the BrM and IM-*A* structures are isomorphous, further discussion will be confined to the two polymorphs of IM. The description of the packing for BrM is the same as that for IM-*A*.

DOI: 10.1107/S0108270104013824

Both IM-*A* and IM-*B* contain C—H···$\pi$ interactions. In IM-*A*, these are from atoms H2 and H5 to the centers of adjoining C$_6$ rings. The two interactions are virtually identical in distance and direction. In IM-*A*, the molecules pack in such a way as to form layers of molecules, with individual molecules perpendicular to the plane of the layer.

In Fig. 4, the packing in IM-*A* is shown perpendicular and parallel to one of the layers. As can be seen, the layers are such that one face contains the I atoms, separated by slightly more than the van der Waals distance from the other face, which contains CH$_3$ groups and O atoms. It can also be seen that the C—H···$\pi$ contacts are almost centrosymmetric with respect to the benzene rings. IM-*B* also contains C—H···ring contacts, but these are to individual ring C atoms and at longer distances than those in IM-*A*. For general discussions of C—H···$\pi$ contacts, see Malone *et al.* (1997), Nishio *et al.* (1998) and Desiraju & Steiner (1999).

The most surprising difference between the two polymorphs of IM is the large difference in the two I···O distances (see Table 2). The distance in IM-*B* is similar to the two independent distances found in *p*-iodobenzaldehyde (Britton & Young, 1997) and is toward the low end of the range of I···O distances reported for other compounds, although by no means the lowest. A search of the Cambridge Structural Database (CSD; Allen, 2002) did not disclose any other examples of two polymorphs with short but differing I···O distances. However, two examples were found where crystallographically independent I···O distances in the same crystal differ by more than 0.1 Å. In ethyl (*E*)-4-[*N*-(2′-iodophenyl)-*N*-methylamino]-4-oxabut-2-enoate (Horne *et al.*, 1991), two crystallographically independent molecules form a dimer held together by two I···O interactions; these dimers have approximately twofold symmetry except for the terminal ethyl groups, but the two distances are 2.987 (4) and 3.110 (4) Å. In (*R*,*S*)-2-methoxytetrahydrofuran-3*R*-ylmethyl 3,4,5-tri-iodobenzoate (Franssen *et al.*, 1996), two crystallographically
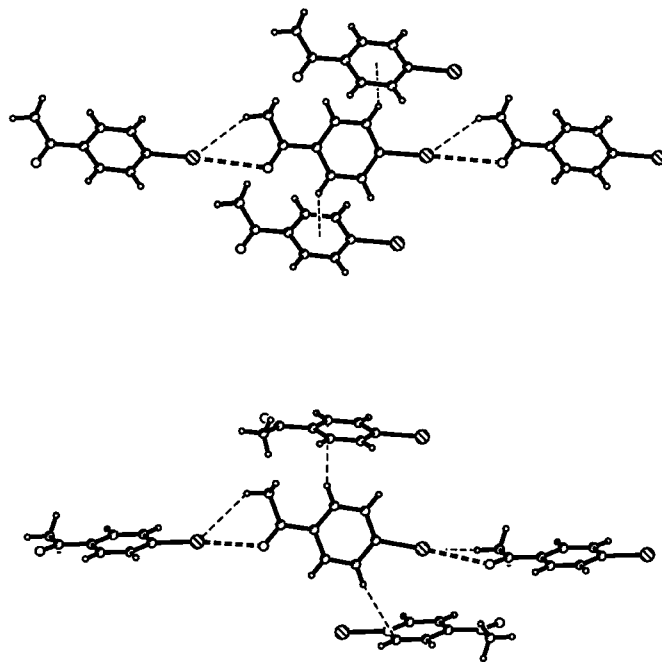


**Figure 3**
The intermolecular contacts in IM-*A* (top) and IM-*B* (bottom), viewed normal to the plane of the central molecule. The *X*···O contacts are shown as double-dashed lines, and the H···*X* and H···$\pi$ contacts as single dashed lines. The top drawing also describes BrM. The top view is approximately normal to (0$\bar{4}$1) and the bottom view normal to (110).



**Figure 2**
The packing of ClM, viewed along *a*. The C—H···O and C—H···Cl contacts are shown as dashed lines.
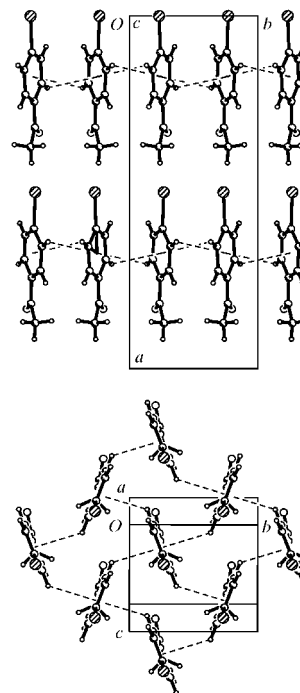


**Figure 4**
The H···$\pi$ contacts in IM-*A*, viewed along *c* (top) and perpendicular to the top view (bottom). Only one layer is shown in the bottom view. The H···$\pi$ contacts are shown as dashed lines.

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independent molecules again form a dimer, with local approximate twofold symmetry. In this case, the two I···O=C contacts in the dimer are similar, *viz.* 3.229 (7) and 3.347 (7) Å, but two other contacts to O atoms in C/O/C groups are significantly different, *viz.* 3.092 (7) and 3.507 (7) Å. Overall, although the I···O interaction is relatively strong, it is weak enough to be significantly affected by the overall packing.

If the change in the I···O distances were the only difference between the two structures, the volume of IM-*B* might be expected to be 3.16 (3)% smaller than that in IM-*A* because of the shortening of the chain length in the I···I direction. The experimental difference is that IM-*B* is larger by 0.76 (6)%; the decrease in the length of the chain is more than offset by increases in the other dimensions. Overall, the packings in the two polymorphs are about equally efficient.

There are no short $X \cdots X$ interactions in either structure. The BrM and IM-*A* structures contain planes of Br or I atoms that are separated by slightly more than the usual $X \cdots X$ van der Waals distances. Although these contacts are not unusually short they contribute significantly to the packing energy. On the other hand, the IM-*B* structure contains no short I···I distances at all.

## Experimental

All three title compounds were obtained from Aldrich. ClM was prepared by sublimation and the crystal of BrM was found in the original sample. Crystals of IM-*A* and IM-*B* were prepared by crystallization from acetone and benzene, respectively; both polymorphs had the same melting point (357–359 K), with no solid-state transformation apparent in either.

### ClM

*Crystal data*

$C_8H_7ClO$
$M_r = 154.59$
Orthorhombic, $P2_12_12_1$
$a = 4.0079 (10)$ Å
$b = 9.366 (2)$ Å
$c = 19.787 (5)$ Å
$V = 742.8 (3)$ Å$^3$
$Z = 4$
$D_x = 1.382$ Mg m$^{-3}$

Mo $K\alpha$ radiation
Cell parameters from 1949 reflections
$\theta = 2.4$–$25.6°$
$\mu = 0.44$ mm$^{-1}$
$T = 173 (2)$ K
Irregular prism, colorless
$0.50 \times 0.40 \times 0.30$ mm

*Data collection*

Siemens SMART area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996; Blessing, 1995)
$T_{min} = 0.81$, $T_{max} = 0.88$
6382 measured reflections

1674 independent reflections
1434 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 27.5°$
$h = -5 \rightarrow 5$
$k = -12 \rightarrow 11$
$l = -25 \rightarrow 23$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.080$
$S = 1.03$
1674 reflections
91 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 0.061P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.17$ e Å$^{-3}$
$\Delta\rho_{min} = -0.14$ e Å$^{-3}$
Absolute structure: Flack (1983)
Flack parameter = 0.11 (8)

### BrM

*Crystal data*

$C_8H_7BrO$
$M_r = 199.05$
Monoclinic, $Cc$
$a = 18.635 (5)$ Å
$b = 6.9554 (17)$ Å
$c = 5.9035 (15)$ Å
$\beta = 96.36 (1)°$
$V = 760.5 (3)$ Å$^3$
$Z = 4$
$D_x = 1.739$ Mg m$^{-3}$

Mo $K\alpha$ radiation
Cell parameters from 2078 reflections
$\theta = 2.2$–$25.7°$
$\mu = 5.33$ mm$^{-1}$
$T = 173 (2)$ K
Plate, colorless
$0.45 \times 0.15 \times 0.03$ mm

*Data collection*

Siemens SMART area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS* including thin plate correction; Sheldrick, 1996; Blessing, 1995)
$T_{min} = 0.39$, $T_{max} = 0.85$

3840 measured reflections
1691 independent reflections
1569 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 27.5°$
$h = -23 \rightarrow 23$
$k = -8 \rightarrow 9$
$l = -7 \rightarrow 7$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.077$
$S = 1.05$
1691 reflections
91 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.036P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.75$ e Å$^{-3}$
$\Delta\rho_{min} = -0.27$ e Å$^{-3}$
Absolute structure: Flack (1983)
Flack parameter = $-0.004$ (14)

### Polymorph IM-*A*

*Crystal data*

$C_8H_7IO$
$M_r = 246.04$
Monoclinic, $Cc$
$a = 19.425 (5)$ Å
$b = 7.049 (2)$ Å
$c = 5.8769 (15)$ Å
$\beta = 94.41 (1)°$
$V = 802.3 (4)$ Å$^3$
$Z = 4$
$D_x = 2.037$ Mg m$^{-3}$

Mo $K\alpha$ radiation
Cell parameters from 3302 reflections
$\theta = 3.1$–$27.5°$
$\mu = 3.92$ mm$^{-1}$
$T = 173 (2)$ K
Plate, pale yellow
$0.25 \times 0.20 \times 0.10$ mm

*Data collection*

Siemens SMART area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996; Blessing, 1995)
$T_{min} = 0.38$, $T_{max} = 0.68$
4483 measured reflections

1821 independent reflections
1768 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.5°$
$h = -25 \rightarrow 25$
$k = -9 \rightarrow 9$
$l = -7 \rightarrow 7$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.060$
$S = 1.05$
1821 reflections
91 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 0.031P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.63$ e Å$^{-3}$
$\Delta\rho_{min} = -0.31$ e Å$^{-3}$
Absolute structure: Flack (1983)
Flack parameter = $-0.01$ (3)

## Table 1
Distances and angles ($\text{Å}$, °) in the C—H$\cdots$X contacts.

Entries preceded by an asterisk (*) are intramolecular distances included for comparison. $\pi$ refers to the center of the $C_6$ ring.

| H | X | C—H | C—H$\cdots$X | H$\cdots$X | H$\cdots$X—C | C$\cdots$X |
|---|---|---|---|---|---|---|
| **ClM** | | | | | | |
| *H2 | O1 | 0.95 | 98 | 2.49 | 83 | 2.788 (2) |
| *H8A | O1 | 0.98 | 73 | 2.46 | 56 | 2.370 (2) |
| H6 | O1$^i$ | 0.95 | 167 | 2.57 | 116 | 3.499 (3) |
| H8B | O1$^{ii}$ | 0.95 | 166 | 2.46 | 151 | 3.421 (3) |
| *H3 | Cl1 | 0.95 | 74 | 2.80 | 46 | 2.697 (2) |
| *H5 | Cl1 | 0.95 | 74 | 2.81 | 46 | 2.700 (2) |
| H5 | Cl1$^{iii}$ | 0.95 | 163 | 3.02 | 87 | 3.936 (3) |
| H8A | Cl1$^{iv}$ | 0.95 | 158 | 3.03 | 121 | 3.951 (3) |
| H8A | Cl1$^v$ | 0.98 | 121 | 3.13 | 148 | 3.736 (3) |
| | | | | | | |
| **BrM** | | | | | | |
| *H2 | O1 | 0.95 | 98 | 2.51 | 82 | 2.799 (6) |
| *H8A | O1 | 0.98 | 73 | 2.47 | 56 | 2.377 (6) |
| H8B | O1$^{vi}$ | 0.98 | 131 | 2.70 | 106 | 3.430 (6) |
| *H3 | Br1 | 0.95 | 76 | 2.94 | 44 | 2.854 (4) |
| *H5 | Br1 | 0.95 | 77 | 2.90 | 45 | 2.835 (4) |
| H3 | Br1$^{vii}$ | 0.95 | 133 | 3.32 | 66 | 4.024 (4) |
| H8A | Br1$^{viii}$ | 0.98 | 148 | 2.96 | 137 | 3.822 (4) |
| H8B | Br1$^{ix}$ | 0.98 | 114 | 3.18 | 120 | 3.697 (4) |
| H2 | $\pi^{vii}$ | 0.95 | 125 | 2.81 | 82† | 3.437 (4) |
| H5 | $\pi^x$ | 0.95 | 125 | 2.80 | 82† | 3.436 (4) |
| | | | | | | |
| **IM-A** | | | | | | |
| *H2 | O1 | 0.95 | 98 | 2.52 | 82 | 2.809 (7) |
| *H8A | O1 | 0.98 | 73 | 2.46 | 56 | 2.374 (7) |
| H8A | O1$^{vi}$ | 0.98 | 135 | 1.74 | 105 | 3.494 (7) |
| *H3 | I1 | 0.95 | 78 | 3.08 | 41 | 3.035 (5) |
| *H5 | I1 | 0.95 | 79 | 3.05 | 42 | 3.016 (5) |
| H3 | I1$^{vii}$ | 0.95 | 135 | 3.42 | 62 | 4.152 (5) |
| H8A | I1$^{viii}$ | 0.98 | 148 | 3.04 | 138 | 3.907 (5) |
| H8B | I1$^{ix}$ | 0.98 | 108 | 3.38 | 124 | 3.814 (5) |
| H2 | $\pi^{vii}$ | 0.95 | 121 | 2.92 | 83† | 3.504 (5) |
| H5 | $\pi^x$ | 0.95 | 122 | 2.90 | 80† | 3.494 (5) |
| | | | | | | |
| **IM-B** | | | | | | |
| *H2 | O1 | 0.95 | 98 | 2.49 | 83 | 2.792 (5) |
| *H8A | O1 | 0.98 | 73 | 2.46 | 56 | 2.368 (5) |
| H8C | O1$^{xi}$ | 0.98 | 111 | 2.78 | 121 | 3.262 (5) |
| *H3 | I1 | 0.95 | 79 | 3.07 | 41 | 3.036 (4) |
| *H5 | I1 | 0.95 | 79 | 3.09 | 41 | 3.047 (4) |
| H5 | I1$^{xii}$ | 0.95 | 155 | 3.53 | 63 | 4.406 (4) |
| H8A | I1$^{xiii}$ | 0.98 | 135 | 3.22 | 139 | 3.969 (4) |
| H3 | C5$^i$ | 0.95 | 161 | 2.97 | 66† | 3.880 (5) |
| H6 | C2$^{xii}$ | 0.95 | 156 | 2.97 | 82† | 3.860 (5) |

† The angle between the H$\cdots$X bond and the plane of the $C_6$ ring. Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $-x, \frac{1}{2} + y, \frac{3}{2} - z$; (iii) $\frac{1}{2} + x, \frac{3}{2} - y, 1 - z$; (iv) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$; (v) $\frac{3}{2} - x, 1 - y, \frac{1}{2} + z$; (vi) $x, -y, \frac{1}{2} + z$; (vii) $x, -y, -\frac{1}{2} + z$; (viii) $\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$; (ix) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (x) $x, 1 - y, -\frac{1}{2} + z$; (xi) $1 - x, -y, 2 - z$; (xii) $\frac{3}{2} - x, -\frac{1}{2} + y, z$; (xiii) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

## Table 2
Distances and angles ($\text{Å}$, °) in the C—X$\cdots$O=C contacts.

| Compound | X | O | C—X$\cdots$O | X$\cdots$O | X$\cdots$O=C |
|---|---|---|---|---|---|
| BrM† | Br1 | O1$^{xiv}$ | 177.4 (4) | 3.320 (4) | 115.2 (3) |
| IM-A† | I1 | O1$^{xiv}$ | 177.5 (4) | 3.374 (5) | 116.3 (3) |
| IM-B† | I1 | O1$^{xv}$ | 174.7 (4) | 3.082 (4) | 124.0 (3) |
| $IC_6H_4CHO$‡ | I2 | O1 | 174.5 (4) | 3.068 (4) | 119.2 (3) |
| $IC_6H_4CHO$‡ | I2 | O2 | 174.0 (4) | 3.074 (4) | 118.5 (3) |
| $C_{13}H_{14}INO_3$§ | I1 | O4 | 170.9 (4) | 2.987 (4) | 119.5 (3) |
| $C_{13}H_{14}INO_3$§ | I2 | O1 | 166.4 (4) | 3.110 (4) | 120.8 (3) |
| $C_{13}H_{13}I_3O_4$¶ | I1 | O5 | 159.8 (7) | 3.229 (7) | 143.3 (6) |
| $C_{13}H_{13}I_3O_4$¶ | I2 | O4 | 176.1 (7) | 3.092 (7) | 113.1 (6) |
| $C_{13}H_{13}I_3O_4$¶ | I4 | O1 | 158.2 (7) | 3.347 (7) | 143.6 (6) |
| $C_{13}H_{13}I_3O_4$¶ | I5 | O7 | 146.4 (7) | 3.507 (7) | 106.8 (6) |

† This work. ‡ Britton & Young (1997), CSD refcode RIWTOG. § Horne *et al.* (1991), CSD refcode KOLWIR. ¶ Franssen *et al.* (1996), CSD refcode TATDEX. Symmetry codes: (xiv) $-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (xv) $x, \frac{1}{2} - y, -\frac{1}{2} + z$.

## Polymorph IM-B

*Crystal data*

| | |
|---|---|
| $C_8H_7IO$ | Mo $K\alpha$ radiation |
| $M_r = 246.04$ | Cell parameters from 2276 |
| Orthorhombic, $Pbca$ | reflections |
| $a = 9.777$ (2) $\text{Å}$ | $\theta = 2.9–27.4°$ |
| $b = 8.232$ (2) $\text{Å}$ | $\mu = 3.89$ mm$^{-1}$ |
| $c = 20.089$ (5) $\text{Å}$ | $T = 173$ (2) K |
| $V = 1616.8$ (7) $\text{Å}^3$ | Plate, colorless |
| $Z = 8$ | $0.25 \times 0.20 \times 0.01$ mm |
| $D_x = 2.021$ Mg m$^{-3}$ | |

*Data collection*

| | |
|---|---|
| Siemens SMART area-detector diffractometer | 1855 independent reflections |
| | 1236 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int} = 0.052$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996; Blessing, 1995) | $\theta_{max} = 27.5°$ |
| | $h = -12 \rightarrow 12$ |
| | $k = -10 \rightarrow 10$ |
| $T_{min} = 0.41$, $T_{max} = 0.96$ | $l = -26 \rightarrow 26$ |
| 15 576 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.019P)^2 + 2.68P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.026$ | |
| $wR(F^2) = 0.070$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.09$ | $(\Delta/\sigma)_{max} = 0.002$ |
| 1855 reflections | $\Delta\rho_{max} = 0.56$ e $\text{Å}^{-3}$ |
| 91 parameters | $\Delta\rho_{min} = -0.45$ e $\text{Å}^{-3}$ |
| H-atom parameters constrained | |

For each of the four structures, a complete sphere of data was collected and all possible Friedel pairs were measured. For ClM, 638 (62%) of the 1036 pairs contained information about which enantiomer was present; refinement as a racemic twin led to a minor twin fraction of 0.11 (8). For BrM [827 (96%) of 864 pairs] and IM-A [897 (97%) of 924 pairs], the values of the Flack (1983) parameters indicate that the reported polarities are correct. In the refinement of BrM and IM-A, the origin restraints on $x$ and $z$ were made using the method of Flack & Schwarzenbach (1988). After the refinements were complete, the origin was shifted, in each case, to $x = \frac{1}{2}$ and $z = \frac{1}{2}$, to facilitate comparison between the two structures. In the final refinement of each of the four structures, the methyl H atoms were included with idealized distances and angles, since this treatment seems appropriate in the presence of the heavy atoms. However, in each case, the H atoms were also found from a difference Fourier map and the positions and $U_{iso}$ values were refined; these results agreed with the group refinements within experimental error in every case.

For all compounds, data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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