Isostructural polymorphs of triiodophloroglucinol and triiodoresorcinol†

Naba K. Nath, Binoy K. Saha and Ashwini Nangia*

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Triiodoresorcinol (TIR, 2,4,6-triiodoresorcinol) and triiodophloroglucinol (TIG, 2,4,6-triiodophloroglucinol) crystallized as orthorhombic (TIR-O and TIG-O in $P2_12_12_1$) and monoclinic (TIR-M and TIG-M in $P2_1/n$) polymorphs mediated *via* inter-halogen I···I interactions. The orthorhombic polymorphs are isostructural and in turn similar to the crystal structure of 1,3,5-triiodobenzene (TIB). The isostructural monoclinic polymorphs are similar to the structure of 1,3,5-trifluoro-2,4,6-triiodobenzene (TIF). Triiodophenol (TIP) crystallized in a single orthorhombic form only. The monoclinic structures have tandem O–H···O hydrogen bonds in addition to inter-iodine interactions. The similarity of crystal structures was confirmed by the formation of 1 : 1 binary solid-solutions, TIP + TIR-O and TIR + TIG-O, in orthorhombic space group $P2_12_12_1$. Dimorphs of TIR and TIG establish a structural link in the triiodobenzene series TIB ($P2_12_12_1$) → TIP ($P2_12_12_1$) → TIR ($P2_12_12_1$ and $P2_1/n$) → TIG ($P2_12_12_1$ and $P2_1/n$) → TIF ($P2_1/n$). The search for new polymorphs was initiated by isostructurality relationship in the series of compounds.

Introduction

Polymorphism is the existence of the same chemical substance in more than one crystalline modification.[1] When different molecules, usually within a family of compounds, adopt the same (similar) crystal structure it is termed isostructurality.[2] The two phenomena typify almost opposite molecule to crystal structure behavior in the organic solid-state.[3] Polymorphs show how the same molecule can crystallize in different hydrogen bonding and close packing arrangements. The identity of space group and unit cell parameters of the crystal structures being compared is called isomorphism. When closely related molecules (*e.g.* stereoisomers, *cis*/*trans* isomers, homologues, phenylogues, *etc.*) have the same or very similar crystal structure, the phenomenon is termed morphotropism.[4] Polymorphism and isostructurality are important in crystal engineering, materials science and pharmaceutical chemistry. One may want to know all possible crystal forms of a drug[5] or replace one molecule with another without disturbing the crystalline order.[6] There are examples of molecules that are polymorphic and those that exhibit isostructurality.[7] We show a rare case of the title molecules that have two polymorphs and these forms are in turn isomorphous and isostructural.[8]

Triiodophloroglucinol (TIG) and triiodoresorcinol (TIR) crystallized as orthorhombic (O) and monoclinic (M) polymorphs that make isomorphous and isostructural pairs (TIG-O = TIR-O and TIG-M = TIR-M).[9] The high degree of structural similarity is confirmed by the formation of binary solid-solutions.

Results

TIR and TIG were synthesized[10] by the iodination of resorcinol and phloroglucinol. Triiodophenol (TIP) was purchased (Aldrich) and used as such for crystallization. Preparation of compounds, crystallization conditions and single-crystal X-ray diffraction are detailed in the Experimental section. The nature of inter-halogen interactions is discussed in recent papers.[11] Halogen···halogen interactions are of two main types: in type I geometry $\theta_1 \cong \theta_2$ and type II approach is characterized by $\theta_1 \cong 180^\circ$ and $\theta_2 \cong 90^\circ$ (Fig. 1). The heavy halogens are polarized positively in the polar region (along the C–X vector) and negatively in the equatorial region (perpendicular to the C–X vector). The atomic size, radial anisotropy and polarization increase in the order Cl < Br < I whereas electronegativity changes in the reverse direction Cl > Br > I. Type II geometry is believed to be a result of specific inter-halogen interactions and hence favored for iodine because of stabilization from the dual polarization in $C–I^{(\delta+)}\cdots I^{(\delta-)}–C$. On the other hand, electronegative chlorine atom is more often present in type I geometry, a result largely of close packing consideration.

Polymorphism

Crystallization of TIG from EtOAc, $CHCl_3$ or mesitylene afforded good quality single crystals. Crystals obtained from $CHCl_3$ were solved and refined in the non-centrosymmetric orthorhombic space group $P2_12_12_1$ are designated as TIG-O. A second form was obtained when a hot solution of TIG in mesitylene was poured over the filter paper. Quick precipitation gave single crystals for X-ray diffraction in the centrosymmetric space group $P2_1/n$, or TIG-M form. The H atoms

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School of Chemistry, University of Hyderabad, Hyderabad, 500 046, India. E-mail: ashwini.nangia@gmail.com; Tel: +91 40 2313 4854

† Electronic supplementary information (ESI) available: Packing diagrams (Fig. S1 and S2) and crystallographic data (Table S1) of iodohydroxy-benzenes extracted from the literature. CCDC reference numbers 687008–687014. For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/b804905j

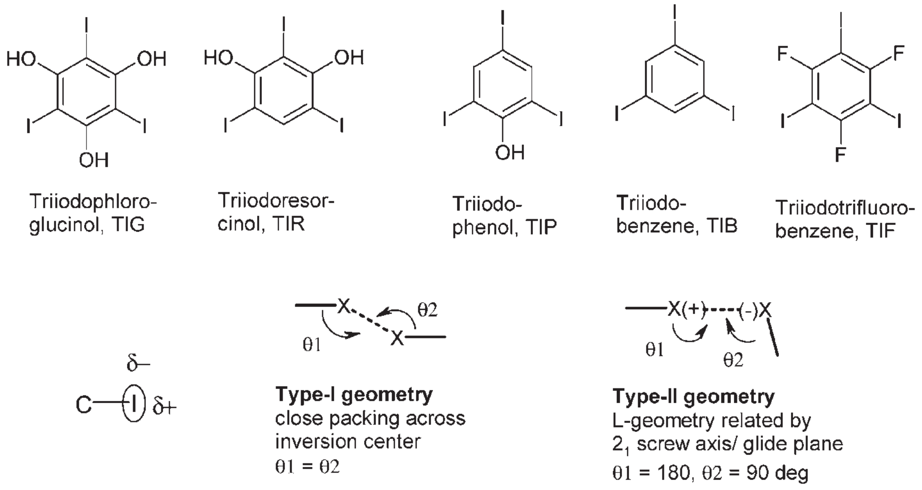


Fig. 1 Triiodo-hydroxybenzene compounds studied and two types of inter-halogen interactions. Note the electrostatic stabilization in type-II L-geometry for iodo···iodo interaction.

of OH groups could not be located in difference electron density maps of X-ray radiation because of the flanking heavy iodine atoms. The molecular packing is discussed *via* inter-halogen interactions listed in Table 1.

In the crystal structure of TIG-O, the three OH groups are flanked by iodine atoms and do not engage in significant hydrogen bonding for steric reasons. The structure is directed by type-II iodo interactions. The molecules form 1D zigzag

Table 1 Inter-halogen interactions (d/Å, θ/°) in crystal structures

			C–I···I–C		
Compound	Interaction	d(I···I)	θ_1	θ_2	Symmetry code
TIB[17a]	I2···I1	3.985(1)	153.4(1)	112.0(1)	$3/2 - x, 1 - y, -1/2 + z$
	I1···I1	3.944(1)	153.1(1)	108.6(1)	$-1/2 + x, 1/2 - y, 1 - z$
	I2···I1	4.052(1)	147.8(1)	93.5(1)	$1 - x, 1/2 + y, 1/2 - z$
	I3···I2	4.048(1)	150.1(1)	77.9(1)	$1/2 + x, 1/2 - y, -z$
TIP	I2···I2	3.996(1)	153.1(2)	108.0(2)	$-1/2 + x, -1/2 - y, -z$
	I1···I3	4.035(1)	143.3(2)	84.7(1)	$-1/2 + x, 1/2 - y, -z$
	I3···I2	4.022(1)	157.1(2)	112.1(2)	$1 - x, 1/2 + y, 1/2 - z$
TIR-M	I1···I1	3.736(1)	175.5(2)	112.4(2)	$1/2 - x, -1/2 + y, 1/2 - z$
	I2···I3	3.907(1)	152.5(2)	79.9(1)	$3/2 - x, 1/2 + y, 1/2 - z$
	I3···I1	3.921(1)	160.5(2)	110.0(2)	$1/2 + x, 3/2 - y, 1/2 + z$
TIR-O	I3···I2	3.812(2)	162.1(5)	106.6(1)	$1 - x, 1/2 + y, 1/2 - z$
	I1···I3	3.919(2)	147.2(5)	81.8(2)	$-1/2 + x, 1/2 - y, -z$
TIG-O	I3···I2	3.771(1)	164.2(2)	103.7(2)	$1 - x, 1/2 + y, 1/2 - z$
	I1···I3	3.888(1)	146.0(2)	82.2(1)	$-1/2 + x, 1/2 - y, -z$
TIG-M	I1···I1	3.699(2)	175.7(3)	107.2(3)	$1/2 - x, -1/2 + y, 1/2 - z$
	I3···I1	4.021(2)	159.6(3)	109.9(3)	$1/2 + x, 3/2 - y, 1/2 + z$
	I2···I3	4.003(2)	145.4(3)	82.8(1)	$3/2 - x, 1/2 + y, 1/2 - z$
	I2··· I2[a]	3.990(2)	131.1(3)	131.1(3)	$1 - x, 3 - y, -z$
TIF[14b]	F1···F1[a]	2.766(4)	157.7(2)	157.7(2)	$-x, -y, 1 - z$
	I3···I2	3.912(1)	147.1(1)	79.7(1)	$-1/2 - x, -1/2 + y, 3/2 - z$
	I2···I1	3.994(1)	157.7(1)	105.9(1)	$-1/2 + x, -1/2 - y, -1/2 + z$
	I1···I1	3.736(1)	171.3(1)	100.4(1)	$1/2 - x, 1/2 + y, 3/2 - z$
	I3···I3[a]	4.048(1)	136.7(1)	136.7(1)	$-x, -2 - y, 2 - z$
	F2···I3	3.455(3)	164.9(2)	73.0(2)	$-1/2 - x, -1/2 + y, 3/2 - z$
TIP + TIR-O	I3···I2	3.935(1)	159.1(2)	109.4(2)	$1 - x, 1/2 + y, 1/2 - z$
	I1···I3	3.959(1)	146.3(2)	82.1(1)	$-1/2 + x, 1/2 - y, -z$
TIR + TIG-O	I1···I3	3.896(1)	146.9(2)	82.1(1)	$-1/2 + x, 1/2 - y, -z$
	I3···I2	3.768(1)	164.5(2)	104.9(2)	$1 - x, 1/2 + y, 1/2 - z$

[a] Inter-halogen type-I interaction. All other inter-halogen interactions are type-II.

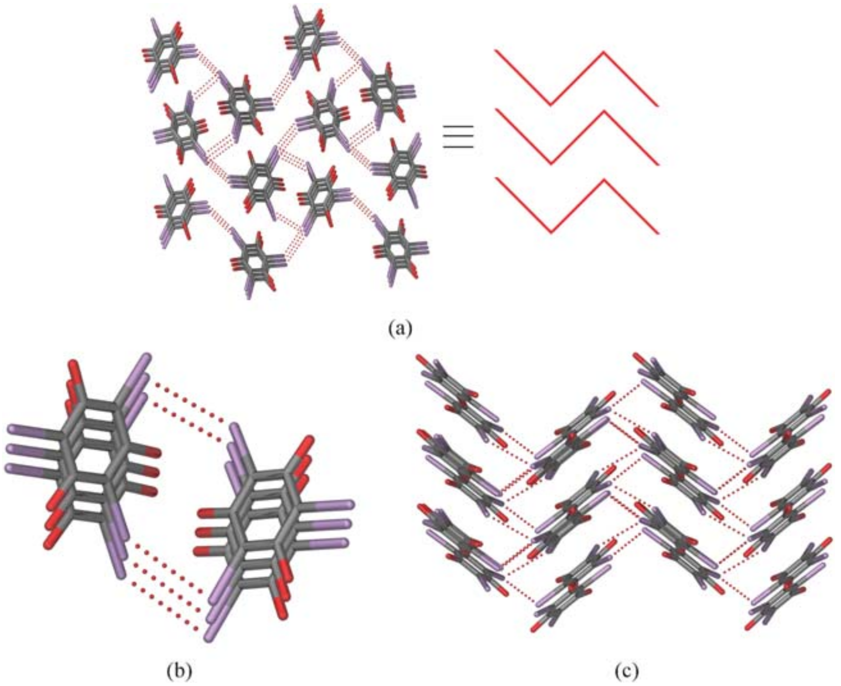


Fig. 2 Molecular packing in TIG-O. (a) Zigzag tapes mediated by type-II I···I interaction are shown as a line diagram, (b) a left-handed helix down [100], and (c) corrugated sheet parallel to the (100) plane.

tapes *via* I3···I2 interaction (3.77 Å; 164.0°, 103.7°) along the [010] direction. These tapes extend into the third dimension *via* I1···I3 interaction (3.89 Å; 145.9°, 82.2°) forming a left-handed helix in the [100] direction. The molecules form a corrugated sheet-like structure parallel to the (100) plane (Fig. 2).

In the TIG-M form, molecules extend along [100] *via* I1···I1 (3.70 Å; 175.7°, 107.2°) and I2···I3 (4.00 Å; 145.4°, 82.8°) type-II interaction to form tapes which extend in the third dimension *via* I3···I1 (4.02 Å; 159.6°, 109.9°) and type-I I2···I2 interaction (3.99 Å; 131.1°). I2···I3 interactions make left- and right-handed helices in the [010] direction. Zigzag chains of I1···I1 interactions along [010] and corrugated sheet-like structures in (010) are present (Fig. 3).

When TIR was crystallized from solvents such as $CHCl_3$, mesitylene and CCl_4, reflections (crystal from $CHCl_3$ was used for X-ray diffraction) were solved in the monoclinic space group $P2_1/n$ (TIR-M). A second orthorhombic form was obtained when 20 mg of TIR in $CHCl_3$–MeOH was seeded with 2–3 crystals (~10 by wt%) of TIP and the solution kept for slow evaporation. X-Ray diffraction showed that it crystallized in the orthorhombic space group $P2_12_12_1$ (TIR-O). The oxygen atoms are disordered so as to mimic a 1,3,5-trihydroxybenzene molecule in both crystal structures. The partial site occupancy factor (s.o.f.) of resorcinol O atoms is given in Table 2.

In the TIR-M form, molecules form a tape along [100] *via* I1···I1 (3.74 Å; 175.5°, 112.5°) and I2···I3 (3.91 Å 152.5°, 79.9°). The tapes extend *via* I3···I1 (3.92 Å, 160.5°, 110.0°) interaction. I2···I3 interactions make left- and right-handed helices in the [010] direction. Type-II I1···I1 interactions make zigzag chain along [010] and the molecules form a corrugated sheet-like structure in the (010) plane (Fig. 4).

In TIR-O, molecules form a zigzag tape along [010] *via* I3···I2 (3.81 Å; 162.1°, 106.6°) type-II interaction. These tapes extend in the third dimensions *via* I1···I3 type-II interaction (3.92 Å; 147.2°, 81.8°) forming a left-handed helix along [100]. Molecules form corrugated sheets in the (100) plane (Fig. 5). Iodo···iodo interactions direct the crystal packing and OH groups are mostly silent. There are no short O···O contacts.

TIP (crystallized from $CHCl_3$, space group $P2_12_12_1$) extends in [010] direction through a zigzag tape of type-II I3···I2 interaction (4.02 Å; 157.1°, 112.1°). The packing is completed *via* I2···I2 (3.99 Å; 153.1°, 108.0°) and I1···I3 (4.03 Å; 143.4°, 84.7°) type-II interactions. A zigzag chain of I2···I2 interactions and left-handed helix of I1···I3 runs along the [100] direction. The molecules form a corrugated sheet in the (100) plane (Fig. 6). A monoclinic form of this compound or another polymorph could not be obtained.

Isostructurality and solid-solution

The unit cell similarity index $(\Pi)^2$ of two crystal structures being compared may be calculated using the equation

$$\Pi = \left|\frac{a+b+c}{a'+b'+c'}\right| - 1$$

where a, b, c and a', b', c' are the orthogonalized lattice parameters of related crystals. Π is close to zero for isostructural crystals. Π values of crystal structures in this paper (Table 3) show that the monoclinic and orthorhombic structures are very similar in each crystal system.

Given the excellent isomorphous and isostructural nature among TIR and TIG polymorphs, we attempted solid-solution formation[12] with TIP. Dissolution of equimolar amounts of TIP and TIR in $CHCl_3$ gave 1 : 1 solid-solution

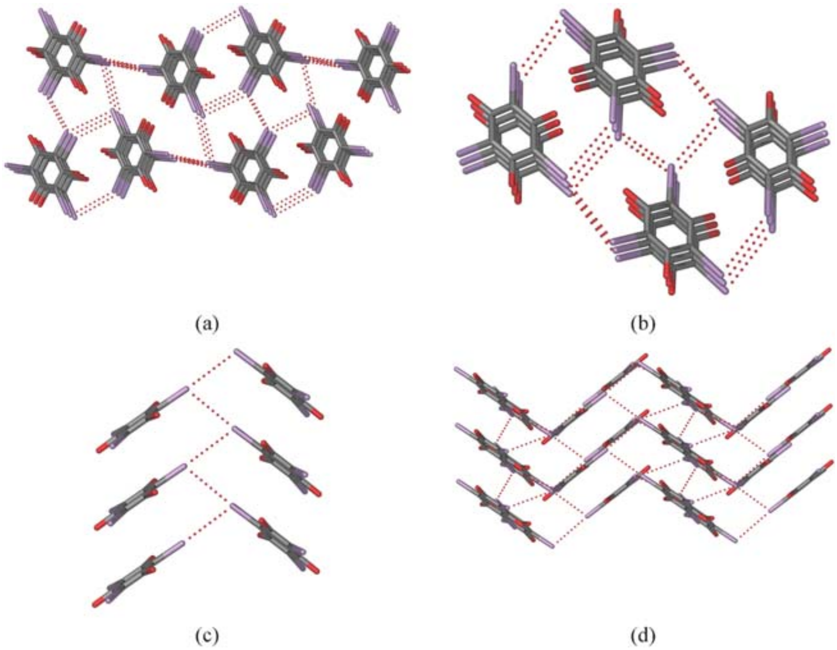


Fig. 3 TIG-M. (a) Tapes along [100] complete the 3D packing *via* formation of I···I zigzag chains and helices, (b) right- and left-handed helices are connected by type-I I···I interaction, (c) type II I···I zigzag chain along [010], and (d) corrugated sheet parallel to the (010) plane.

Table 2 Partial s.o.f. of oxygen atoms in disordered and solid-solution crystal structures

	O1	O2	O3
TIR-M	0.88	0.86	0.27
TIR-O	0.62	0.70	0.68
TIP + TIR-O	0.65	0.47	0.41
TIR + TIG-O	0.76	0.92	0.84

single crystals that solved in the orthorhombic space group $P2_12_12_1$. Similarly, TIR and TIG also gave 1 : 1 solid-solution crystal in space group $P2_12_12_1$. The stoichiometry was derived from s.o.f. of O atoms given in Table 2. Molecular packing in solid-solution crystal structures is shown in Fig. S1 and S2, see ESI†.

Discussion

There are examples of polymorphic crystal structures exhibiting isostructurality.[13] To our knowledge, triiodoresorcinol and triiodophloroglucinol represent a rare case wherein polymorphic

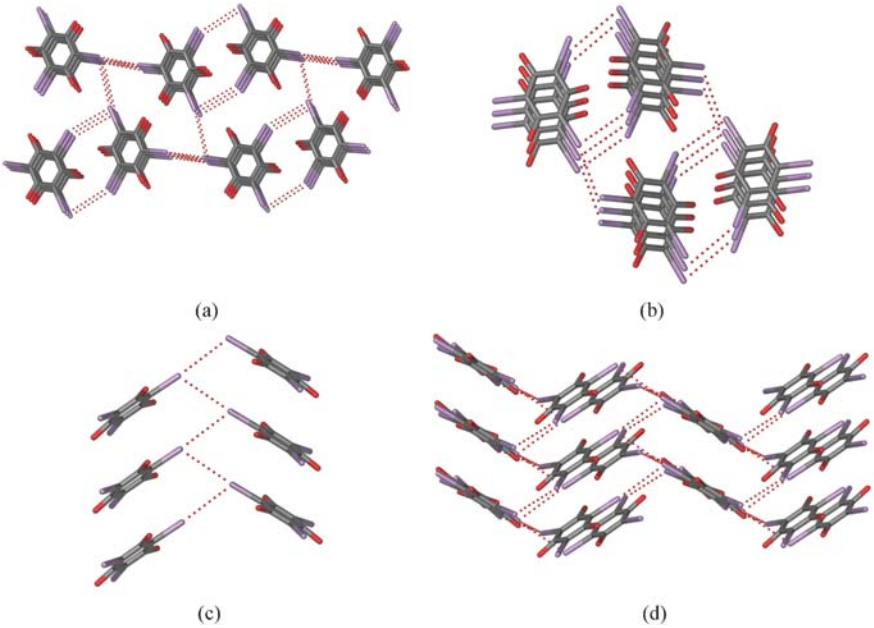


Fig. 4 TIR-M. (a) Tapes along [100] complete the 3D packing *via* formation of helix and zigzag chain, (b) right- and left-handed helix, and (c) iodo interaction-mediated zigzag chain, (d) corrugated sheet parallel to (010).

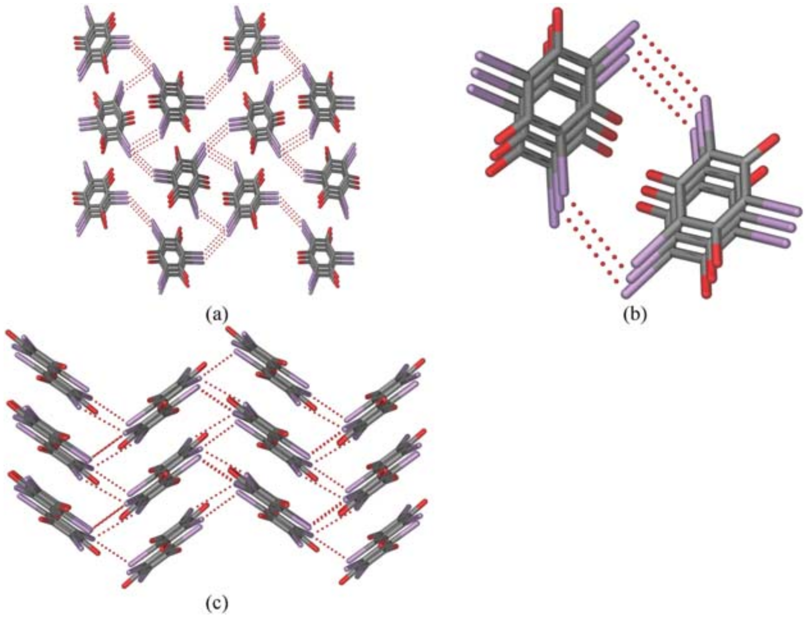


Fig. 5 TIR-O. (a) Zigzag tapes along [010], (b) left handed helix along [100], and (c) corrugated sheet in the (100) plane.

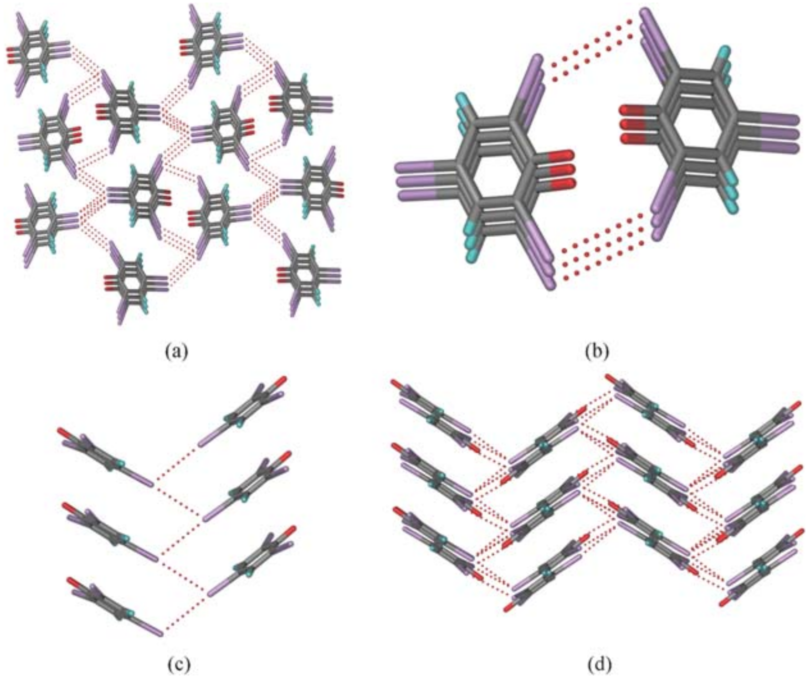


Fig. 6 TIP. (a) Zigzag tapes along [010] extend the packing *via* zigzag chains and helices, (b) left-handed helix along [100], (c) zigzag chain along [100], (d) corrugated sheet parallel to (100).

crystal structures of different molecules make an isostructural pair (Fig. 7). Secondly, the orthorhombic form is similar to the crystal structure of triiodobenzene (and in turn TIB is similar to tribromo- and trichloro-benzene) whereas the monoclinic form resembles the crystal structure of trifluorotriiodobenzene (TIF)[14] in terms of unit cell parameters and molecular arrangement in the crystal lattice. Thus, the dimorphic crystal structures of TIR and TIG serve as a bridge between the monomorphic orthorhombic crystal structures of TIB and TIP and the monoclinic cell of TIF. TIB and TIP crystallize in the $P2_12_12_1$ space group (Table 4) and the molecules are ordered in both crystal structures. TIG crystallized in the $P2_12_12_1$ space group (TIG-O) and the structure is fully ordered. However, the middle member, TIR, crystallized in the $P2_1/n$ space group (TIR-M) but the molecules are disordered so as to resemble TIG. As the terminal members (TIP and TIG) crystallize in the $P2_12_12_1$ space group, it was expected that the

Table 3 Unit cell similarity Π values

Crystal pair	Π
TIB, TIP	0.0054
TIB, TIR-O	0.0127
TIB, TIG-O	0.0134
TIP, TIR-O	0.0073
TIR-O, TIG-O	0.0007
TIP, TIG-O	0.0079
TIF, TIR-M	0.0227
TIR-M, TIG-M	0.0048
TIF, TIG-M	0.0178

middle member TIR too should be able to adopt the same space group. The orthorhombic form TIR-O was obtained when crystallized from $CHCl_3$–CH_3OH solvent mixture using TIP crystals as seed. Similarly, the monoclinic structure of TIR suggested that there should be a monoclinic form of TIG too. Thus the concept of isostructurality led to the discovery of new crystal forms of TIR and TIG. The degree of isostructurality and isomorphous behavior in the family of crystals was confirmed by the formation of solid-solutions. Solid solutions of TIP + TIR and TIR + TIG crystallized in the orthorhombic space group although the monoclinic setting is also a possibility. The solid-solution crystal structures resemble the structures of the pure components in $P2_12_12_1$ space group. A reason for dimorphism in TIR and TIG may be found in the less significant OH groups in this family of compounds. The OH groups are buried between the flanking iodine atoms and the structures do not make conventional O–H···O hydrogen bonds despite having one, two and three phenol groups. We note that the OH groups are silent in the orthorhombic crystal structure whereas O···O distances are <3.0 Å in the monoclinic forms. When H atoms on the OH groups that participate in hydrogen bonding were placed in putative positions using the HFIX command (see Experimental section) they participate in tandem hydrogen bonds[15] in the monoclinic structures (TIG-M: H···O 2.48 Å, O···O 2.78 Å; ∠O–H···O 102.6°; TIR-M: 2.75 Å, 2.73 Å; 80.3°). The energy of tandem hydrogen bonds is estimated as -4.67 kcal mol^{-1}, weaker than linear (-5.19 kcal mol^{-1}) but stronger than bifurcated H bonds (-2.87 kcal mol^{-1}).[16] O–H···O hydrogen bonds are much longer in the orthorhombic polymorphs TIG-O and TIR-O (O···O 3.20, 3.16 Å). The role of tandem H bonding in monoclinic crystal structures explains why trihalobenzenes and triiodophenol adopt orthorhombic form only (Table 4) because O–H···O interactions are not possible to optimise in these structures. The OH disorder in TIR mimics the TIG structure *via* tandem H bonds. Incidentally, the tandem OH arrangement in TIR-M and TIG-M is replaced by F···F contacts in the monoclinic structure of TIF. TIF has a corrugated layer structure[14] sustained by type-II I···I and other inter-halogen contacts (Table 1). The completely different crystal structures of TIB (orthorhombic) and TIF (monoclinic) by H → F replacement is not surprising but now a link is established in the series through TIR and TIG dimorphs (Fig. 8). A full list of closely related crystal structures archived from the literature[17] is given in Table S1 (ESI†).

Conclusions and future studies

Triiodoresorcinol and triiodophloroglucinol exhibit dimorphism in contrast to several other members which are monomorphic. Whereas crystal structures of trihalobenzenes in the orthorhombic crystal setting are traditionally well known, a monoclinic structure of TIF was very recently reported.[14] The present study shows alternative crystal packing arrangements for trihalobenzenes in the monoclinic cell setting. The empty slots in Table 4 suggest that there is scope to discover new crystalline forms in these compounds.

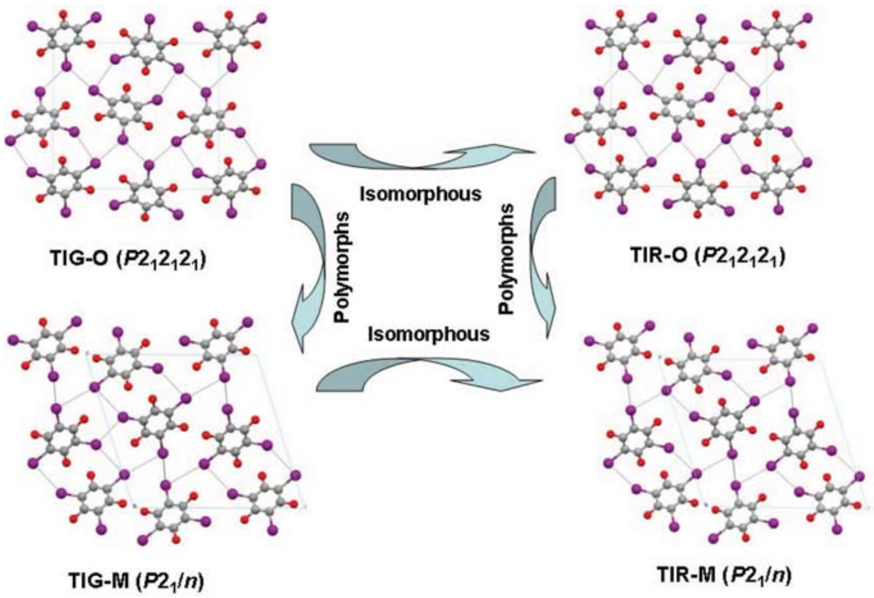
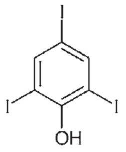
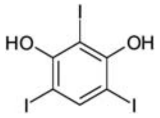
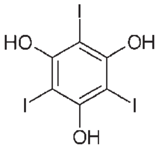
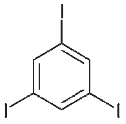
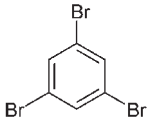
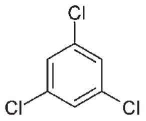
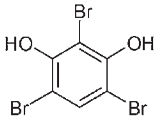
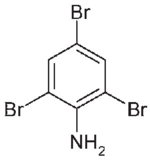
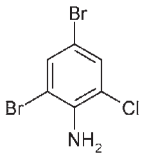
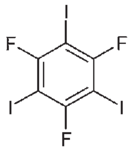
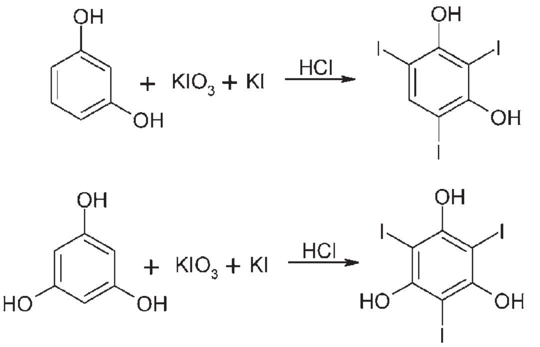


Fig. 7 Isomorphous orthorhombic and monoclinic pairs of TIG and TIR polymorphs.

Table 4 Crystal systems of some trihalobenzenes

Compound	Structure	$P2_12_12_1$	$P2_1/n$
TIP		√[a]	—
TIR		√[a]	√[a]
TIG		√[a]	√[a]
TIB[17a]		√[b]	—
TBB[17b]		√[b]	—
TCB[17b]		√[b]	—
TBR[17c]		√[b]	—
TBA[17d]		√[b]	—
BCA[17e]		√[b]	—
TIF[14b]		—	√[b]

[a] This study. [b] Cambridge structural database.



Scheme 1 Preparation of triiodoresorcinol and triiodophloroglucinol.

Experimental

Synthesis

All compounds were synthesized and characterized by IR and ^{1}H NMR. NMR spectra ($CDCl_3$ solution, δ scale) were recorded on a Bruker Avance at 400 MHz. FT-IR spectra (KBr pellet, ν cm^{-1}) were recorded on a Jasco 5300 spectrophotometer. Melting points were recorded on Fisher-Johns apparatus. Crystal structures were secured by single-crystal X-ray diffraction.

Triiodoresorcinol (TIR) and triiodophloroglucinol (TIG) were synthesized by poly-iodination of the starting phenol (Scheme 1).[10]

TIR was synthesized by the dropwise addition of a solution of KI (3.22 g, 20 mmol) and KIO_3 (2.14 g, 10 mmol) in 50 mL water to a solution of resorcinol (1.10 g, 10 mmol) in 10 mL water with 5–6 drops of 50% HCl added. The solution was stirred at room temperature for 6 h to obtain the desired product as a precipitate which was purified by crystallization from $CHCl_3$.

Mp 152 °C (154–157 °C).[10] ^{1}H NMR: 5.89 (2H, s), 7.96 (1H, s). IR: 588, 627, 3430 cm^{-1}.

TIG was synthesized by the same procedure starting from phloroglucinol and the product was crystallized from $CHCl_3$.

Mp (decomp.) 170 °C (171–172 °C).[10] ^{1}H NMR: 5.98 (3H, s). IR: 534, 638, 3443 cm^{-1}.

X-Ray diffraction

Reflections were collected on a Bruker Smart Apex CCD diffractometer at 100 K using Mo-Kα incident X-radiation (λ = 0.71073 Å). SAINT software[18] was used for data reduction. Structures were solved using the direct methods in SHELX-97.[19] Semi-empirical and multi-scan absorption correction SADABS[20] was applied. Crystallographic details are summarized in Table 5. All non-hydrogen atoms were refined anisotropically and C–H hydrogens in the case of TIP were fixed. OH hydrogen atoms could not be located in difference electron density maps due to the large electron density of flanking iodine atoms. The OH hydrogen for the group engaged in short O···O contact in the monoclinic structure was generated using HFIX command for Fig. 8. The stoichiometry of solid-solution structures was derived from the partial site occupancies of O atoms listed in Table 2.

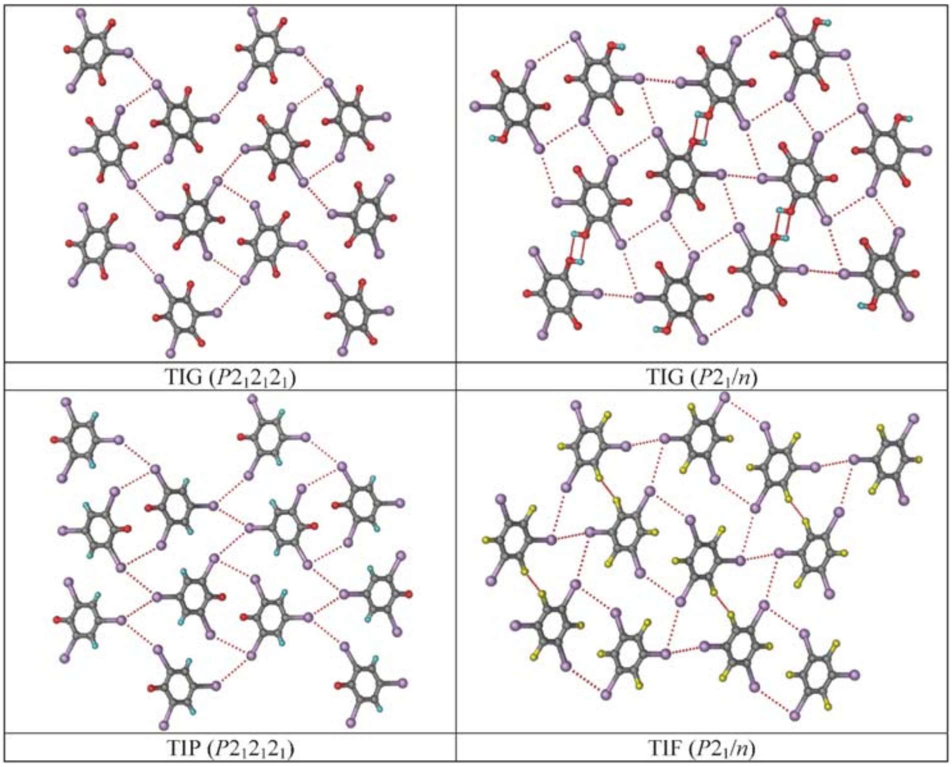


Fig. 8 Tandem hydrogen bonds in the monoclinic crystal structure of TIG are replaced by F···F interaction in TIF. There are no significant O–H···O hydrogen bonds in the orthorhombic structures of TIG and TIP. The hydrogen of the OH group in TIG-M was generated in calculated position using HFIX command.

Table 5 X-Ray crystal data of triiodobenzene derivatives determined in this study

	TIP	TIR-M	TIR-O	TIG-O	TIG-M	TIP + TIR-O	TIR + TIG-O
Chemical formula	$C_6H_3I_3O$	$C_6H_3I_3O_2$	$C_6H_3I_3O_2$	$C_6H_3I_3O_3$	$C_6H_3I_3O_3$	$C_6H_3I_3O_{1.50}$	$C_6H_3I_3O_{2.50}$
M_r	471.78	486.78	486.78	503.78	503.78	479.78	495.78
T/K	100	100	100	100	100	100	100
Crystal system	Orthorhombic	Monoclinic	Orthorhombic	Orthorhombic	Monoclinic	Orthorhombic	Orthorhombic
Space group	$P2_12_12_1$	$P2_1/n$	$P2_12_12_1$	$P2_12_12_1$	$P2_1/n$	$P2_12_12_1$	$P2_12_12_1$
a/Å	4.3695(5)	14.8819(10)	4.4949(7)	4.6376(4)	14.582(4)	4.4419(3)	4.5607(3)
b/Å	14.6940(15)	4.3317(3)	13.958(2)	13.5291(11)	4.5004(13)	14.3683(9)	13.6484(8)
c/Å	14.1839(15)	15.5848(11)	15.036(2)	15.3446(12)	15.507(5)	14.5730(9)	15.3485(9)
β/°	90.00	108.4950(10)	90.00	90.00	107.569(4)	90	90
V/Å^3	910.68(17)	952.77(11)	943.3(3)	962.76(14)	970.2(5)	930.09(10)	955.39(10)
Z	4	4	4	4	4	4	4
D_c/g cm^{-3}	3.441	3.394	3.427	3.476	3.449	3.416	3.447
$2\theta_{max}$/°	52.0	52.1	52.0	51.0	52.2	52.0	52.1
μ/mm^3	10.231	9.793	9.890	9.705	9.631	10.024	9.773
F(000)	824	852	852	888	888	834	872
N (total)	4958	8909	5135	5921	8806	9609	9686
N (indep.)	1788	1853	1800	1800	1719	1834	1884
N (used)	1797	1878	1861	1805	1859	1846	1889
GoF	1.126	1.187	1.256	1.108	1.187	1.197	1.178
R_{int}	0.0251	0.0314	0.0558	0.0281	0.0463	0.0242	0.0223
R_1	0.0290	0.0277	0.0582	0.0241	0.0488	0.0215	0.0225
R_w	0.0700	0.0696	0.1346	0.0602	0.1115	0.0535	0.0544

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