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Structural investigations on four heterocyclic Disperse Red azo dyes having the same benzothiazole/azo/benzene skeleton

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ABSTRACT

Four heterocyclic Disperse Red azo dyes having the same benzothiazole/azo/benzene skeleton, namely three classical C.I. Disperse Red 145, 152 and 153 (**1**–**3**) and another C.I. Disperse Red 153 substitute (**4**), have been structurally characterized and compared in this paper. X-ray single-crystal diffraction analyses reveal that they have similar planar molecular conformation between azo and aromatic units but dissimilar crystal packing fashions. In addition, different ratios of isomers have been found in the co-crystals of **3** and **4**. To the best of our knowledge, this is the first structural report about the azo dyes having the benzothiazole/azo/benzene skeleton as well as co-crystal structures.

1. Introduction

Disperse azo dyes bearing aromatic moieties linked together by azo chromophores have been widely used in textile coloration such as polyester, nylon, acetate, cellulose and acrylic, and so on [1,2]. The optical properties of azo dyes depend on not only their spectral properties, but also their molecular arrangements in the solid state [3,4]. So the single-crystal X-ray structural determination of dye molecules is of great significance because it reveals their molecular conformation, packing fashion and supramolecular interactions, which will help to understand the interactions between dyes and fibers, the colouring mechanism and other related technical performance [5–8].

Investigations on disperse azo dyes having aromatic heterocycles are very important because they have shown brilliant color and chromophoric strength, excellent light, color, washing and sublimation fastness as well as high level-dying property in the dyestuff industry. Among them, benzothiazole diazo components are most popular in heterocyclic Disperse Red dyes, but only several of them have been commercialized up till now. C.I. Disperse Red 145 (**1**), C.I. Disperse Red 152 (**2**) and C.I. Disperse Red 153 (**3**) are most classical and economic commercialized heterocyclic Disperse Red azo dyes after many years of development and elimination through competition (Scheme 1). However, this kind of benzothiazole azo dyes, which have been broadly used for polyester coloration and other high-tech applications, is faced with the limitation of availability of insufficient structural data. To date, there have been merely several structural studies on thiazole/azo/indole and benzothiazole/azo/indole dye compounds reported by Hökelek et al. [9–11], even if the crystal structures of some other types of Disperse Red dyes have been documented [12–18].

In our previous work, several Disperse Yellow azo dyes crystallizing in the hydrazone form in the solid state and their azo-hydrazone tautomerisms have been reported [19–22]. As an extensive study in this area, we report herein the single-crystal structures of **1**–**3** and another C.I. Disperse Red 153 substitute (**4**) with higher thermal stability due to the presence of an additional benzyl group in its molecular structure. They have either nitro (**1**) or dichlorine (**2**–**4**) substituents as electron-withdrawing groups and *p*-positioned *N*-ethyl-*N*′-cyanoethyl (**1**–**3**) or *N*-ethyl-*N*′-benzyl (**4**) substituents as common electron-donating units in their molecular structures. Compounds **1** and **2** are mono-constituent Disperse Red azo dyes, while compounds **3** and **4** are double-constituent ones co-crystallized with different ratios of isomers (50:50 in **3** and 38:62 in **4**) in the solid-state structures. By checking the latest version of CCDC databases (CSD version of 5.31 updated

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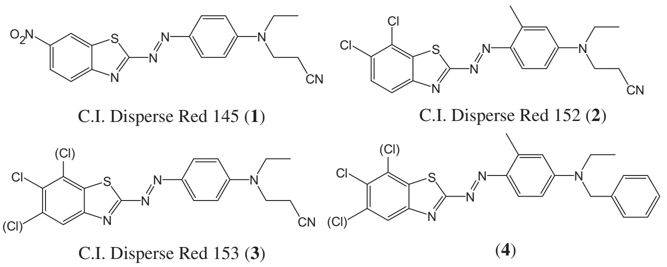

Scheme 1. Molecular structures of Disperse Red azo dyes **1**–**4**.

to February 2010), it is found that this is the first structural report about the azo dye compounds having the benzothiazole/azo/benzene skeleton as well as co-crystal structures.

2. Experimental section

2.1. Materials and measurements

Disperse red azo dyes **1**–**4** were purchased directly from commercial sources. All other reagents and solvents were of analytical grade and used without further purification. Acceptable red crystals of **1**–**4** suitable for X–ray diffraction measurement were produced by slow evaporation of the *N,N′*-dimethylformamide solutions of the purified azo dyes after several weeks in air at the room temperature. Compounds **3** and **4** are isomeric mixture with a 1:1 ratio of two isomers before growing single crystals in our experiment.

2.2. X-ray data collection and solution

Single-crystal samples of **1**–**4** were glue-covered and mounted on glass fibers for data collection on a Bruker SMART 1K CCD area detector at 291(2) K using graphite mono-chromated Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å). The collected data were reduced by using the program SAINT [23] and empirical absorption corrections were done by SADABS [24] program. The crystal systems were determined by Laue symmetry and the space groups were assigned on the basis of systematic absences by using XPREP. The structures were solved by direct method and refined by least-squares method. All non-hydrogen atoms were refined on F^2 by full-matrix least-squares procedure using anisotropic displacement parameters, while hydrogen atoms were inserted in the calculated positions assigned fixed isotropic thermal parameters at 1.2 times of the equivalent isotropic U of the atoms to which they are attached (1.5 times for the methyl groups) and allowed to ride on their respective parent atoms. All calculations were carried out on a PC computer with the SHELXTL PC program package [25] and molecular graphics were drawn by using XSHELL, XP and ChemDraw software. Details of the data collection and refinement results for compounds **1**–**4** are listed in Table 1, while selected bond distances and bond angles are given in Table 2.

3. Results and discussion

3.1. Structural description of Disperse Red azo dye 1

The molecular structure of **1** with atom-numbering scheme is shown in Fig. 1a. It crystallizes in the triclinic $P\bar{1}$ space group without the presence of any solvent molecule. The bond length of azo unit (N3–N4) of **1** is 1.272(3) Å indicative of double-bond character. In contrast, the N1–C7 bond length is 1.281(4) Å which is shorter than those of N1–C2, N3–C7 and N4–C8 in the lengths of 1.372(4), 1.390(4) and 1.387(3) Å, exhibiting predominantly conjugated double-bond character. The thiazole sulfur atom and the azo unit lie at the same side of N3–C7 single bond with the related bond angles of S1–C7–N3 (122.4(2)°), N1–C7–N3 (120.6(2)°), C7–N3–N4 (112.5(2)°) and N3–N4–C8 (114.0(2)°), which are consistent with previously reported methyl or methoxyl substituted benzothiazole azo dyes [9,10]. The torsion angles for

Table 1
Crystal data and structural refinements for four benzothiazole Disperse Red azo dyes.

Compound name	1	2	3	4
Empirical formula	$C_{18}H_{16}N_6O_2S$	$C_{19}H_{17}Cl_2N_5S$	$C_{18}H_{15}N_5Cl_2S$	$C_{23}H_{20}Cl_2N_4S$
Formula weight	380.44	418.35	404.31	455.39
T (K)	291(2)	291(2)	291(2)	291(2)
Crystal system	Triclinic	Monoclinic	Orthorhombic	Orthorhombic
Space group	$P\bar{1}$	$C2/c$	$Pnma$	$P2_12_12_1$
a/Å	7.732(2)	28.332(6)	7.421(4)	8.011(5)
b/Å	7.908(2)	5.784(2)	12.245(6)	12.238(7)
c/Å	15.664(3)	24.118(6)	20.593(11)	22.350(13)
α (°)	79.86(1)	90	90	90
β (°)	81.81(1)	95.131(3)	90	90
γ (°)	74.31(1)	90	90	90
V/Å^3	903.1(3)	3936.7(16)	1871.1(16)	2191(2)
Crystal size/mm	0.08 × 0.10 × 0.12	0.10 × 0.10 × 0.10	0.10 × 0.12 × 0.14	0.10 × 0.12 × 0.16
Z	2	8	4	4
D_c/Mg m^{-3}	1.399	1.412	1.435	1.380
μ (Mo/Kα)/mm^{-1}	0.206	0.450	0.471	0.410
F(000)	396	1728	832	944
Flack parameter	–	–	–	0.01(15)
Absorption correction	Multi-scan	Multi-scan	Multi-scan	Multi-scan
Data collected/unique	4539/3141	3447/1135	8800/1715	9130/3838
Limiting indices	$-9 \le h \le 8$, $-9 \le k \le 5$, $-18 \le l \le 18$	$-26 \le h \le 33$, $-5 \le k \le 6$, $-28 \le l \le 28$	$-6 \le h \le 8$, $-14 \le k \le 14$, $-24 \le l \le 24$	$-6 \le h \le 9$, $-13 \le k \le 14$, $-25 \le l \le 26$
Max./min.transmission	0.9837/0.9757	0.9564/0.9315	0.9544/0.9457	0.9602/0.9374
$R1/wR2$	0.0600/0.1804	0.0890/0.1949	0.0488/0.1359	0.0694/0.1461
Goodness of fit on F^2	1.089	0.999	0.946	0.909
Δ/e·Å^{-3} (max/min)	0.66/–0.39	1.22/–0.43	0.45/–0.30	0.29/–0.36

$R1 = \Sigma||Fo| - |Fc||/\Sigma|Fo|$, $wR2 = [\Sigma[w(Fo^2 - Fc^2)^2]/\Sigma w(Fo^2)^2]^{1/2}$.

Table 2
Selected bond distances (Å) and angles (°) of four benzothiazole Disperse Red azo dyes.

1		2		3		4	
Bond distances							
S1–C1	1.728(3)	S1–C1	1.710(8)	S1–C9	1.774(4)	S1–C1	1.733(7)
S1–C7	1.759(3)	S1–C7	1.747(9)	N1–N2	1.245(4)	S1–C7	1.722(7)
N1–C2	1.372(4)	N1–C2	1.385(11)	N1–C4	1.383(5)	N1–C7	1.261(10)
N1–C7	1.281(4)	N1–C7	1.298(11)	N2–C8	1.363(9)	N1–C2	1.412(10)
N3–N4	1.272(3)	N2–N3	1.307(8)	N3–C1	1.351(5)	N2–N3	1.297(9)
N3–C7	1.390(4)	N2–C7	1.399(10)	N5–C8	1.310(10)	N2–C7	1.438(10)
N4–C8	1.387(3)	N3–C8	1.367(10)	N5–C10	1.413(7)	N3–C8	1.396(9)
N5–C11	1.366(4)	N4–C11	1.348(10)	N4–C7	1.133(9)	N4–C11	1.387(10)
N5–C14	1.461(4)	N4–C15	1.430(10)			N4–C15	1.594(13)
N5–C17	1.509(5)	N4–C18	1.466(10)			N4–C17	1.430(10)
N6–C16	1.113(5)	N5–C17	1.123(13)				
Bond angles							
C1–S1–C7	87.5(2)	C1–S1–C7	87.3(4)	N2–N1–C4	121.3(3)	C1–S1–C7	85.9(4)
C2–N1–C7	110.5(2)	C2–N1–C7	108.6(6)	N1–N2–C8	111.9(6)	C2–N1–C7	107.2(7)
O1–N2–O2	122.7(3)	N3–N2–C7	109.6(6)	C8–N5–C10	109.1(7)	N3–N2–C7	110.1(6)
N4–N3–C7	112.5(2)	N2–N3–C8	116.3(6)	N5–C8–N2	122.2(9)	N2–N3–C8	117.8(6)
N3–N4–C8	114.0(2)	C11–N4–C15	123.1(6)			C11–N4–C15	119.4(7)
S1–C1–C2	110.1(2)	C11–N4–C18	120.5(6)			C11–N4–C17	124.1(7)
N1–C2–C1	114.8(3)	C15–N4–C18	116.3(6)			C15–N4–C17	115.5(6)
S1–C7–N1	117.0(2)	S1–C7–N1	117.8(6)			S1–C7–N1	121.6(6)
S1–C7–N3	122.4(2)	S1–C7–N2	121.4(6)			S1–C7–N2	120.4(5)
N1–C7–N3	120.6(2)	N1–C7–N2	120.8(7)			N1–C7–N2	118.0(6)
N4–C8–C9	124.6(3)	N3–C8–C9	116.8(7)			N3–C8–C9	117.4(6)
N4–C8–C13	117.4(2)	N3–C8–C13	123.9(7)			N3–C8–C13	122.4(6)

N4–N3–C7–S1 (178.2(3)°), N1–C7–N3–N4 (178.0(2)°), C13–C8–N4–N3 (179.7(2)°), N3–N4–C8–C9 (179.1(4)°) and C7–N3–N4–C8 (179.8(2)°) exhibit that all the 15 non-hydrogen atoms in the chromophore skeleton, except the *N*-substituted ethyl and cyanoethyl groups, are essentially coplanar with the mean deviation of 0.0228 Å (Fig. 1b), which is the typical structural feature for most azo dye molecules.

The *N*-substituted ethyl and cyanoethyl groups of **1** are found to point to the same side of the above-mentioned least-squares molecular plane. This may help to form a dimeric packing mode between adjacent molecules in a head-to-tail way in its crystal structure, where weak π–π stacking interactions are observed between the benzene ring of one benzothiazole unit and another adjacent phenyl ring with the centroid-to-centroid separation of 4.035(3) Å. However, the parallel aromatic rings are positioned in an offset way with a shorter mean interlayer contact of 3.547(4) Å since two compensative intermolecular C–H···O hydrogen bonding interactions between the O atom from nitro group and the H atom from cyanoethyl group are present simultaneously to stabilize this dimeric structure (Table 3). Additionally, neighboring dimeric units are further packed into a layer structure by means of π–π stacking interactions between two aromatic rings

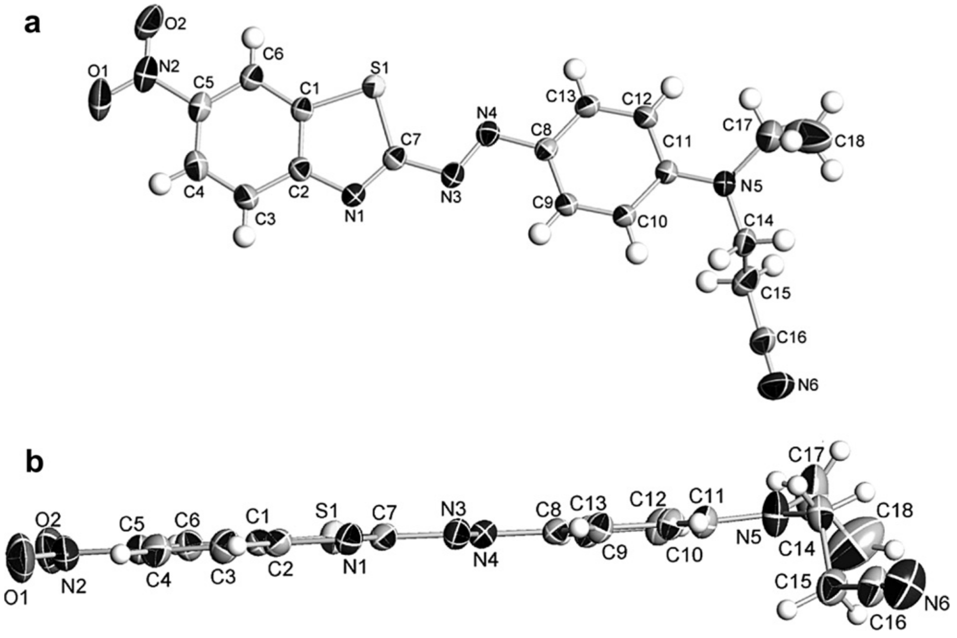

Fig. 1. ORTEP drawings of **1** with the atom-numbering scheme (top view for **a** and side view for **b**). Displacement ellipsoids are drawn at the 30% probability level and the H atoms are shown as small spheres of arbitrary radii.

Table 3
Hydrogen bonding interactions (Å, °) of **1**.

D–H···A	D–H	H···A	D···A	∠DHA	Symmetry code
C10–H10···N6	0.93	2.51	3.390(5)	159.0	$-x, 1-y, 1-z$
C15–H15A···O2	0.97	2.56	3.389(5)	143.0	$1-x, 1-y, 2-z$

of one benzothiazole unit and another adjacent phenyl ring with the centroid-to-centroid separations of 3.717(3) and 3.873(3) Å and the dihedral angle of 2.1(2)° (Fig. 2). The mean interlayer contact between neighboring dimeric units is 3.669(4) Å, indicative of different offset extension of molecules due to the less steric hindrance effect of *N*-substituted alkyl groups. This dimeric full layer packing fashion in **1** is unlike to the previously reported benzothiazole disperse azo dyes [9,10], where different sets of molecules are packed into their respective directions with large dihedral angles between them.

3.2. *Structural description of Disperse Red azo dye* **2**

The molecular structure of **2** with atom-numbering scheme is shown in Fig. 3. The compound crystallizes in the monoclinic *C*2/*c* space group also in the absence of any solvent molecule. Similar to **1**, the bond length of azo unit (N2–N3) is 1.307(8) Å corresponding to the double-bond character. The N1–C7 bond length is 1.298(11) Å which is shorter than those of N1–C2, N2–C7 and N3–C8 (1.385(11), 1.399(10) and 1.367(10) Å), exhibiting an apparent delocalization of electron density along the chain of connected atoms from N2 to N3. In addition, the thiazole sulfur atom and the azo unit of C.I. Disperse Red 152 lie at the same side of the molecular plane, and the two chlorine atoms of benzothiazole and the methyl group of benzene ring are positioned at the same side of the molecular plane. The whole molecule, except the *N*-substituted ethyl and cyanoethyl groups, is essentially coplanar with the mean deviation of 0.0116 Å, and the torsion angles for N3–N2–C7–S1, N3–N2–C7–N1, N2–N3–C8–C9, N2–N3–C8–C13 and C7–N2–N3–C8 are 179.8(8), 0.8(7)°, 178.1(6), 0.4(8)° and 179.9(6)°.

In the case of **1**, a head-to-tail stacking mode can be found and all molecules are stacked along the same direction. While in the crystal packing of **2**, π–π stacking interactions are found between the phenyl rings and thiazole rings of neighboring molecules with the centroid–centroid and interlayer separations of 3.778 and 3.494 Å, respectively (Fig. 4). It is noted that there are two sets of molecules in the crystal packing of **2**, and each set packs in a herringbone fashion and the dihedral angle between them is 74.1°.

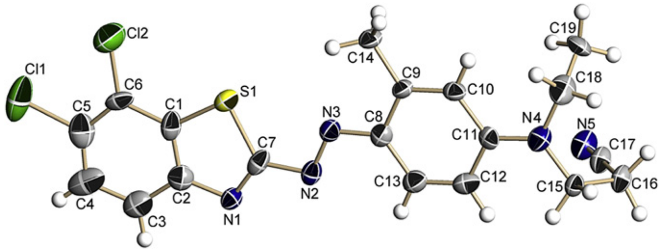

Fig. 3. ORTEP drawing of **2** with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and the H atoms are shown as small spheres of arbitrary radii.

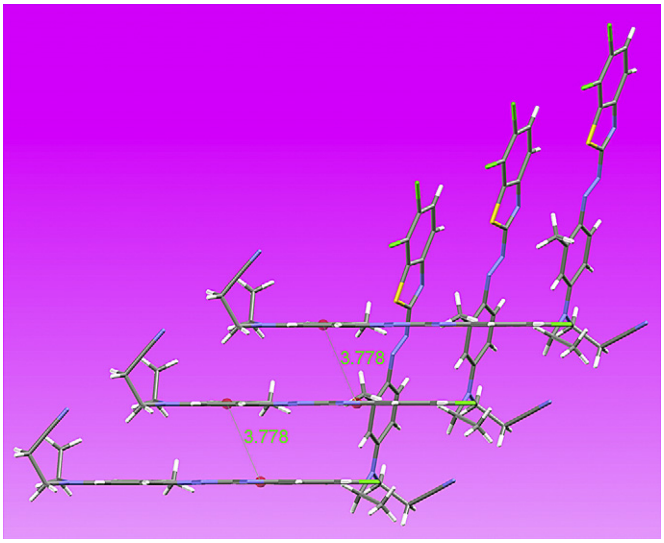

Fig. 4. Perspective view of the π–π stacking interactions in the crystal packing of **2**.

3.3. *Structural description of Disperse Red azo dye* **3**

The molecular structure of **3** with atom-numbering scheme is shown in Fig. 5. As we know, Disperse Red azo dyes **2** and **3** have similar molecular structures except that two isomers of 2-amino-5,6-dichlorobenzothiazole coupling components have been used in the diazotization of **3**. A crystallographically symmetric plane passes through the center of the aromatic plane and the two isomers are resultantly co-crystallized with the same site occupancy factor of 0.5. However, **3** crystallizes in the orthorhombic *Pnma* space group, indicative of higher crystallographic symmetry.

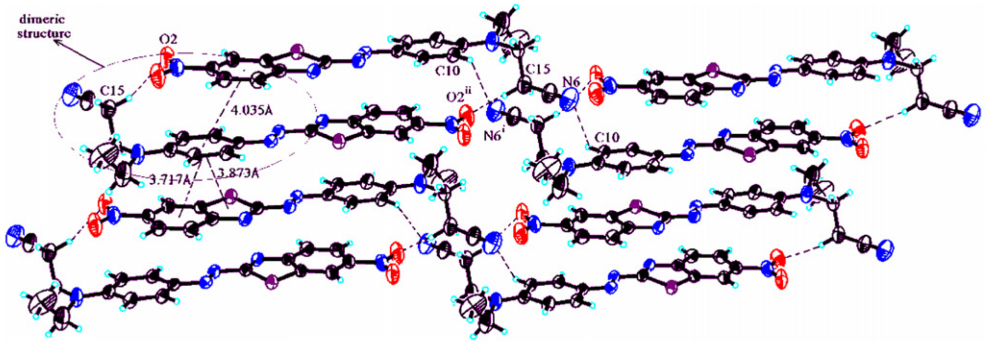

Fig. 2. Perspective view of the dimeric stacking and hydrogen-bond contacts in **1** where the centroid to centroid separations are shown. Hydrogen bonds are indicated as dashed lines and the labels are given only once for clarity. Translation of symmetry codes to the equivalent positions: $i = 1-x, 1-y, 1-z$; $ii = 1-x, 1-y, 2-z$.

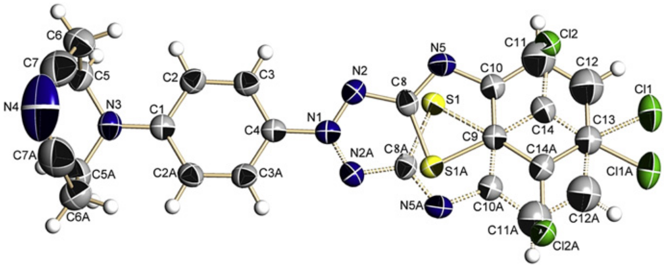

Fig. 5. ORTEP drawing of **3** with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and the H atoms are shown as small spheres of arbitrary radii.

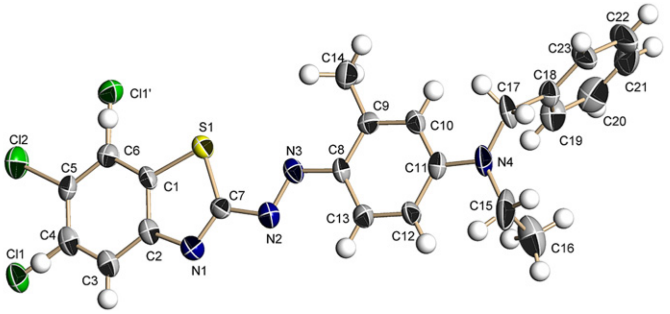

Fig. 6. ORTEP drawing of **4** with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and the H atoms are shown as small spheres of arbitrary radii.

The whole molecule, except the *N*-substituted ethyl and cyanoethyl groups, is essentially coplanar with the mean deviation of 0.0200 Å.

In the crystal packing of **3**, $\pi-\pi$ stacking interactions are found between the phenyl rings and the thiazole rings of neighboring molecules with the centroid-centroid and interlayer separations of 3.625 and 3.527 Å, respectively. Analogous to those in **2**, two sets of molecules are found and each set packs in a herringbone fashion with the dihedral angle of 55.4°.

3.4. *Structural description of Disperse Red azo dye* 4

The molecular structure of **4** with atom-numbering scheme is shown in Fig. 6. It crystallizes in the chiral orthorhombic $P2_12_12_1$ space group with a reasonable Flack parameter of 0.01(15) [26]. The whole molecule, except the *N*-substituted ethyl and benzyl groups, is essentially coplanar with the mean deviation of 0.0353 Å. The dihedral angle between the two phenyl rings within the molecule is 81.53°. Here Disperse Red azo dye **4** is also a co-crystal. Nevertheless, in this case, the two isomeric structures are refined disorderedly over two positions by the free variable method and the final site occupancy factors are given as 0.381(4): 0.619(4) for each isomer.

In the crystal packing of **4**, only a dimeric $\pi-\pi$ stacking mode are observed because of the steric hindrance of benzyl group, wherein the centroid–centroid and interlayer separations are 3.626 and 3.476 Å, respectively. Different from those in **1**–**3**, three sets of molecules are found the dihedral angles between them are 52.9, 28.7 and 61.3°. No obvious hydrogen bonding interactions can be observed in the crystal packing structures of **2**–**4**.

4. Conclusion

In summary, we have described the structural comparisons for four benzothiazole heterocyclic Disperse Red azo dyes (**1**–**4**) in this paper. X-ray single-crystal diffraction analyses show that all non-hydrogen atoms except the *N*-substituted (ethyl, cyanoethyl and benzyl) groups are essentially coplanar and the thiazole sulfur atom and the azo unit adopt the *cis* conformation in **1**–**4**. Furthermore, the *N*-substituted groups in **1**–**4** are found to point to the same side of least-squares molecular planes. However, they crystallize in different crystal systems and space groups without the presence of any solvent molecule, and dissimilar crystal packing modes are observed. In **1**, all molecules are packed along one direction and a dimeric packing manner between adjacent molecules in a head-to-tail way is found. In contrast, there are two (or three) sets of molecules in the crystal structures of **2** and **3** (or **4**) packed in the herringbone manner. As far as we are aware, this is the first structural report on the azo dyes having the benzothiazole/azo/benzene backbone as well as co-crystal structures.

Acknowledgements

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Appendix. Supplementary material

CCDC reference numbers 716120, 770646, 796721 and 796722 contain the supplementary crystallographic data corresponding to compounds **1**–**4** in this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.dyepig.2010.11.008.

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