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Chemical Interactions between 2-Mercaptobenzazoles and π-Acceptors

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Summary. 2-Mercaptobenzazoles (**1a–c**) interact with several π-acceptors such as tetracyanoethylene (*TCNE*) 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (*DDQ*), 2,3,5,6-tetrachloro-1,4-benzoquinone (*CHL*) dicyanomethyleneindane-1,3-dione (*CNIND*), 2,3-dicyano-1,4-naphthoquinone (*DCNQ*), 9-dicyanomethylene-2,4,7-trinitrofluorene (*DTF*), and 2,3-dichloro-1,4-naphthoquinone (*DCHNQ*) *via* the formation of charge-transfer (CT) complexes to yield various heterocyclic compounds.

Keywords. 2-Mercaptobenzazoles; Molecular interactions; π-Acceptors.

Chemische Wechselwirkungen zwischen 2-Mercaptobenzazolen und π-Akzeptoren

Zusammenfassung. Die 2-Mercaptobenzazole **1a–c** reagieren mit verschiedenen π-Akzeptoren wie Tetracyanoethylen (*TCNE*), 2,3-Dichlor-5,6-dicyano-1,4-benzochinon (*DDQ*), 2,3,5,6-Tetrachlor-1,4-benzochinon (*CHL*), Dicyanomethylenindan-1,3-dion (*CNIND*), 2,3-Dicyano-1,4-naphthochinon (*DCNQ*), 9-Dicyanomethylen-2,4,7-trinitrofluoren (*DTF*) und 2,3-Dichlor-1,4-naphthochinon (*DCHNQ*) unter Ausbildung von charge transfer – Komplexen (CT) zu heterocyclischen Verbindungen.

Introduction

The utility of 2-mercaptobenzazoles **1a, b** in the synthesis of several heterocyclic compounds [1–7] as well as their photoreactivity [8–10] have been reported. Additionally, 2-mercaptobenzothiazoles exhibited antitubercular [11], antispasmodic [12] and antibacterial [13, 14] activities. Moreover, many benzothiazoles bearing substituents at positions 2 and 3 evoke fungitoxic and herbicidal responses [15–18] and display inhibitory effects on the multiplication of polivirus and adenovirus [19]. In the light of these observations, it was considered to be of interest to test some basically different 2-mercaptobenzazoles (**1a–c**) as electron donors towards several π-acceptors (Fig. 1).

Results and Discussion

Our overall strategy is outlined in Figs. 2 and 3 which outline the interaction of **1a, b** with different π-acceptors.

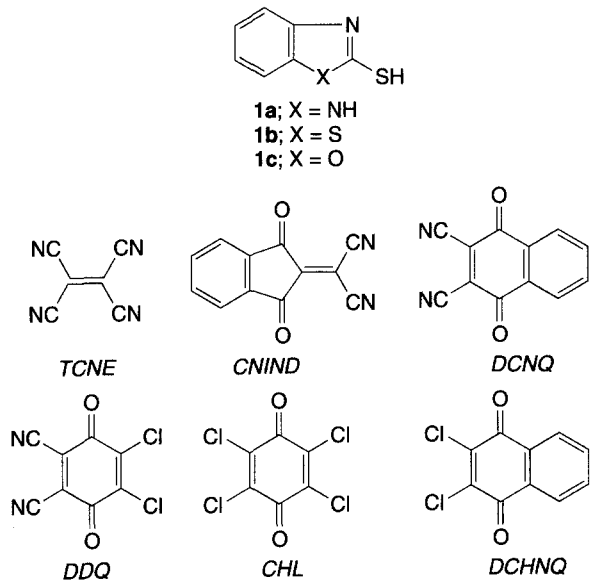

Fig. 1

Table 1. Spectral data for some CT complexes of **1a–c** with *TCNE*, *DTF*, *CNIND*, *DDQ*, *DCNQ*, and *CHL*

Donor	Acceptor	λ_{max} (nm)	K_{ct} ($l \cdot mol^{-1}$)	ε ($l \cdot mol^{-1} \cdot cm^{-1}$)	E (eV)
1a	*TCNE*	592	4.23	200	2.09
1b	*TCNE*	505	2.60	133	2.46
1c	*TCNE*	460(sh)	–	–	–
1a	*DTE*	615	6.18	500	2.02
1b	*DTF*	545	4.82	370	2.28
1c	*DTF*	510	2.66	333	2.43
1a	*CNIND*	548	2.20	667	2.26
1b	*CNIND*	480(sh)	–	–	–
1a	*DDQ*	670	9.26	625	1.85
1b	*DDQ*	575	4.80	200	2.16
1c	*DDQ*	505	2.68	266	2.46
1a	*DCNQ*	600	5.83	133	2.07
1b	*DCNQ*	530	2.80	67	2.34
1c	*DCNQ*	490(sh)	–	–	–
1a	*CHL*	540	2.00	200	2.30
1b	*CHL*	475(sh)	–	–	–

As shown in Table 1, the UV/Vis absorption maxima obtained by mixing both donor and acceptor components in ethyl acetate as solvent fall in the visible region (670–475 nm). These maxima are attributed to CT complex formation, since both donor and acceptor alone did not absorb in this region. Mixing of a twofold molar amount of *TCNE* with one mole of the donor **1a** leads to a CT complex which is in equilibrium with the two components. Thereafter, the reaction products 1-aminobenzo[4,5]imidazo[2,1-*b*][1,2]thiazepine-2,3,4-tricarbonitrile (**2**) and 2-benzimidazolyl-2-mercaptobenzimidazolyl-cyanoacetamide (**3**) as well as 2,2-dibenzimidazolylsulfide (**4**) were isolated. The interaction of **1b** with the same acceptor afforded 2-dicyanomethylene-benzothiazole (**13**) and the tricyanovinylation products **14** and **15**.

The formation of benzo[4, 5]imidazo[2,1-b]naphtho[2′,3′-d][1,3]thiazole-7,12-dione (**10**) and 10,11-dihydro-10-oxo-benzo[*d*]indeno[2,1:4,5]pyrrolo[1,2-*a*] imidazole-11,11-dicarbonitrile (**11**) in the reaction of **1a** with *DCHNQ* and *CNIND*,

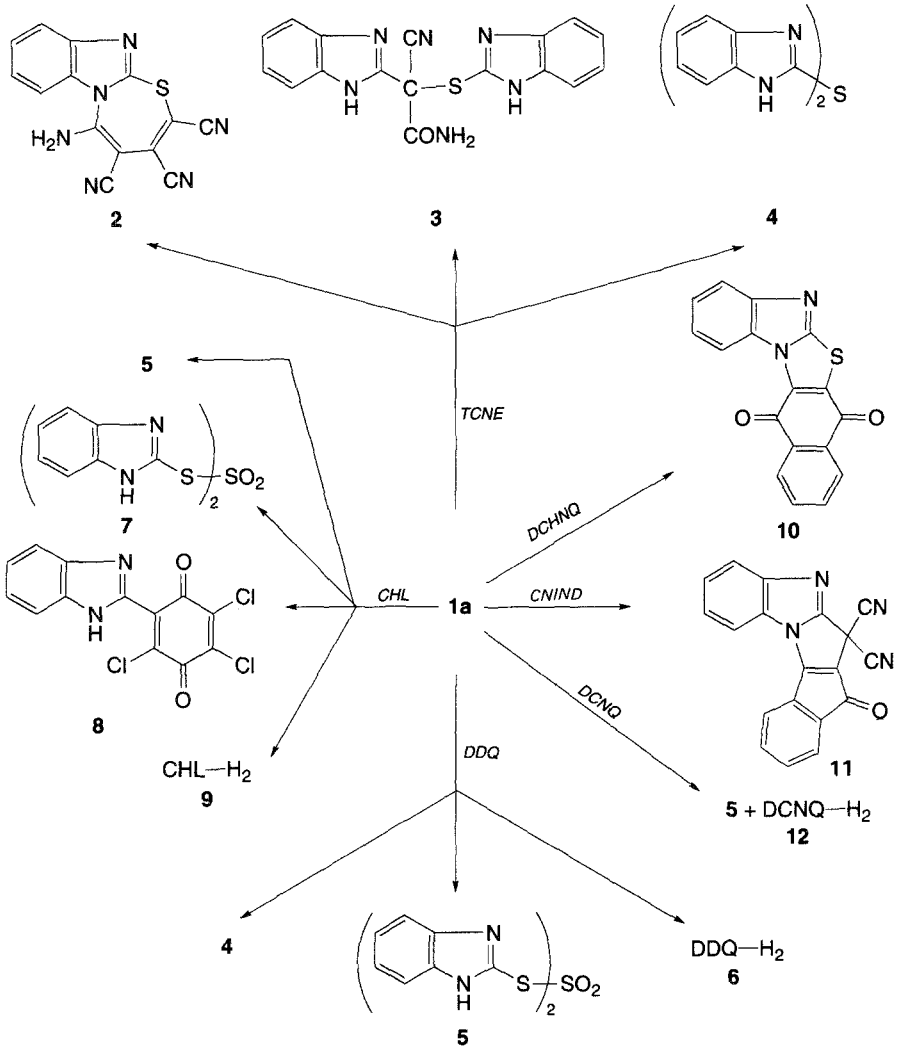

Fig. 2

respectively, reveals the participation of the NH group in **1a** in the course of the reaction. On the other hand, two moles of **1b** reacted with one mole of *CNIND* to afford 2-(1,3-benzothiazol-2-yl)-2-[1-(1,3-benzothiazol-2-yl)-3-oxo-3*H*-indeno-2-yl]-malononitrile (**2l**) in addition to the disulfide **16**.

The reaction of *DCHNQ* with **1b** afforded the disulfide **16**, the sulfide **18**, and the condensation product **20**. Interaction of *CHL* with benzazoles **1a**, **b** resulted in the formation of the products **5**, **7**–**9**, **16**, **18**, and **19**. *DDQ* acts as a dehydrogenating agent and yields compounds **4**–**6** in the case of reaction with **1a**, whereas compounds **6**, **16**, and **17b** were obtained in case of reaction with **1b**. Benzoxazolone **17c** and *DDQ*-H_2 were obtained from the reaction of **1c** with *DDQ* (Fig. 3).

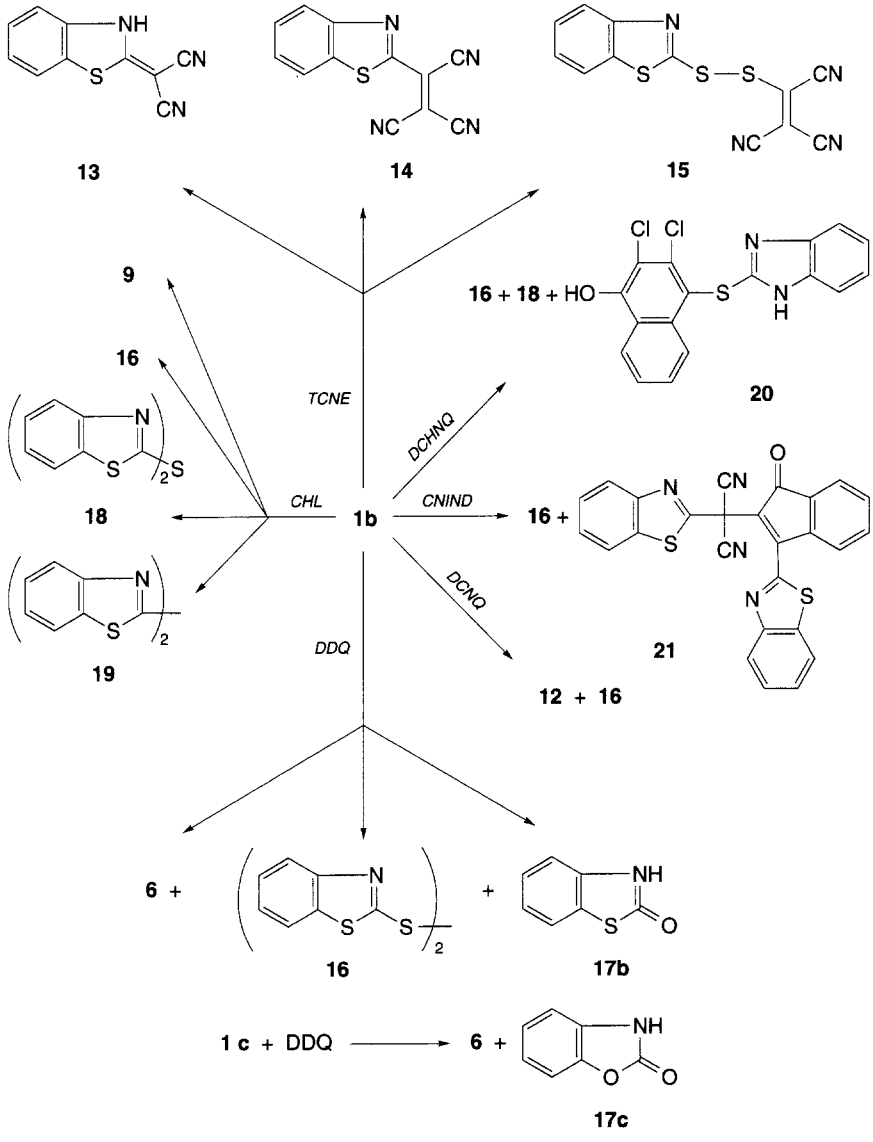

Fig. 3

Experimental

All melting points are uncorrected; UV/Vis spectra: Perkin-Elmer Lambda 2 spectrophotometer equipped with a thermostatted cell holder; IR spectra: Shimadzu 470 and Nicolet 320 FT-IR spectrophotometer (KBr); ^{1}H NMR spectra: Bruker WM200 (200) MHz, $CDCl_3$, *DMSO*-d_6 and CD_3COCD_3, δ (ppm), *TMS*); MS: Finnigan MAT 8430 (70 cV); elemental analysis: microanalytical department at Cairo University.

Table 2. Analytical and physical data of compounds **2–5**, **7**, **8**, *10*, **11**, **13–21**

Compund	m.p. (°C)	colour of crystal	Solvent of recrystallization	Mol. Formula[a] (M. Wt.)
2	355–57	Pale yellow	Acetonitrile	$C_{14}H_6N_6S$ (290.309)
3	248–50	Colourless	Ethanal	$C_{17}H_{12}N_6SO$ (348.388)
4	276–78	Colourless	Benzene	$C_{14}H_{10}N_4S$ (266.327)
5	232–34	Colourless	Ethanol	$C_{14}H_{10}N_4S_3O$ (346.458)
7	> 360	Colourless	Ethanol	$C_{14}H_{10}N_4S_3O_2$ (362.457)
8	246–48	Yellow	Ethanol	$C_{13}H_5N_2Cl_3O_2$ (327.554)
10	191–93	Red	*DMF*	$C_{17}H_8N_2SO_2$ (304.328)
11	313–15	Yellow	Acetonitrile	$C_{19}H_8N_4O$ (308.229)
13	294–96	Blue	Ethanol	$C_{10}H_5N_3S$ (199.233)
14	> 360	Red	Ethanol	$C_{12}H_4N_4S$ (236.258)
15	217–19	Pale yellow	Ethanol	$C_{12}H_4N_4S_3$ (300.390)
16b	178–80 (Ref. [27, 28]: 180)	Pale yellow	Ethanol	$C_{14}H_8N_2S_4$ (332.495)
17b	130–32 (Ref. [29]: 130)	Colourless	Ethanol	C_7H_5NSO (151.189)
17c	139–41 (Ref. [30]: 140)	Colourless	Benzene	$C_7H_5NO_2$ (135.122)
18	298–300	Colourless	Ethanol	$C_{14}H_8N_2S_3$ (300.429)
19	302–04 (Ref. [31, 32]: 305)	Colourless	Ethanol	$C_{14}H_8N_2S_2$ (268.363)
20	> 360	Reddish-brown	Ethanol	$C_{17}H_9NCl_2S_2O$ (378.302)
21	331–33	Yellow	Acetonitrile	$C_{26}H_{12}N_4S_2O$ (460.450)

[a] Elemental analyses (C, H, N, S, Cl) were in good agreement with calculated values

Table 3. ^{1}H NMR, IR and mass spectra of compounds **2–5, 7, 8, 10, 11, 13–15, 17, 18, 20,** and **21**

Compound	^{1}H NMR (δ, ppm)[a]	IR (KBr, cm^{-1})	MS (m/z (rel. intensity%))
2	7.30–8.00 (m, 4H, Ar-H); 8.80 (s, br, 2H, NH2)	3390, 3180 (NH_2), 2210, 2230 (CN), 1640, 1590, 1580 (AR-C=C)	290(M^+, 100), 264(5), 263(12), 238(7), 237(7), 205(11), 194(8), 168(21), 167(9), 145(10)
3	6.80–7.60 (m, 8H, Ar-H), 8.10 (s, br, 2H, $CONH_2$), 11.80 (s, br, 1H), 12.20 (s, br, 1H) NH-imidazole ring)	3380–3240 (NH, NH_2), 2210 (CN), 1710 (CO) 1610, 1580 (Ar-C=C)	348(M^+, 7), 266 (75), 208(100), 149(25)
4	7.22–7.78 (m, 8H, Ar-H), 11.90 (s, br, 2H, 2NH-imidazole ring.	3320–3280 (NH), 1620 (ArC=C)	266(M^+, 81), 234(100), 208(97), 207(24), 118(8), 90(4)
5	7.10–7.60 (m, 8H) Ar-H; 11.92 (s, br, 2H) 2NH-imidazole ring)	3380–3300 (NH), 3050 (Ar-CH), 1630–1610, 1580 (Ar-C=C)	347(M^+, 100), 346(6), 244(18), 206(8), 174(31)
7	7.40–8.10 (m, 8H, Ar-H), 11.95 (s, br, 2H, 2NH-imidazole ring)	3420–3320 (NH), 3070 (Ar-CH), 1640–1620 (Ar-C=C)	362(M^+, 4), 346(32), 250(45), 234(14), 208(59), 149(60), 134(100), 118(38), 106(57), 79(34)
8	7.05–7.42 (m, 4H, Ar-H), 11.72 (s, br, 1H, NH-imidazole ring)	3410–3320 (NH), 3060 (Ar-CH), 1670 (CO), 1620 1600, 1570 (Ar-C=C)	327/329(M^+, 86), 299(15), 291(11), 263(12), 248(35), 208(15), 118(58)
10	7.32–7.52, 7.65–8.10 (m, 8H, Ar-H)	3060 (Ar-CH), 1670 (CO), 1580 (Ar-C=C)	304(M^+, 100), 276(9), 248(7), 190(6), 176(12), 150(52)
11	7.22–7.58 (m, 4H, Ar-H)	3090 (Ar-CH), 2220 (CN), 1680 (CO), 1600, 1580–1560 (Ar-C=C)	308(M^+, 100), 250(81), 118(23), 97(18), 57(34)

Table 3. (*Continued*)

13	7.22–7.80 (m,4H, Ar-H), 12.20(s, br, 1H, NH-thiazole ring)	3360–3280 (NH), 2210 (CN), 1600, 1580 (Ar-C=C)	199(M^+, 100), 172(21), 146(23), 145(18), 108(11), 69(22)
14	7.20–7.70 (m, 4H, Ar-H)	2215 (CN), 1652, 1638, 1609 (Ar-C=C)	238(M + 2, 100), 211(68), 184(21), 135(34)
15	7.38–8.10 (m, 4H, Ar-H)	3060 (Ar-CH), 2220 (CN), 1640, 1600, 1590 (Ar-C=C)	300(M^+, 100), 268(33), 166(31), 134(28)
18	7.30–7.95 (m, 4H, Ar-H)	3080 (Ar-CH), 1610, 1580 (Ar-C=C)	300(M^+, 86), 268(10), 242(41), 166(100), 134(70), 108(33), 96(34), 57(28)
20	6.95–8.20 (m, 8H, Ar-H), 9.50 (s, br, 1H, OH)	3480–3300 (OH), 3070 (Ar-CH), 1580 (Ar-C=C)	378(M^+, 22), 376(100), 345(10), 320(14), 292(16), 276(7), 104(18), 76(22)
21	7.00–7.50, 7.65–7.80 (m, 12H, Ar-H)	2210 (CN), 1675 (CO), 1620, 1580 (Ar-C=C)	462(M + 2, 6), 460(12), 458(8), 332(100), 268(33), 224(21), 108(63)

[a] All compounds were measured in *DMSO*-d_6 except **2**, **3**, and **4** ($CDCl_3$) and **7** (CD_3COCD_3)

Electron acceptors. Tetracyanoethylene (*TCNE*, Merck) was recrystallized from chlorobenzene and sublimed. 2,3-Dicyano-5,6-dichloro-1,4-benzoquinone (*DDQ*, Aldrich) was recrystallized from benzene/chloroform (2:3). 2,3,5,6-Tetrachloro-1,4-benzoquinone (Chloranil, *CHL*, Aldrich) was recrystallized several times from benzene before use. 2,3,5,6-Tetrafluoro-1,4-benzoquinone (Fluoranil, *TFQ*, Janssen Belgium) was used without further purification. Dicyanomethyleneindan-1,3-dione (*CNIND*) was prepared according to the procedure described by *Chatterjee* [20] and recrystallized from acetonitrile. 9-Dicyanomethylene-2,4,7-trinitrofluorene (*DTF*) was prepared from 2,4,7-trinitrofluorenone (Aldrich) and malononitrile according to *Mukherjee* [21]. 2,3-Dicyano-1,4-naphthoquinone (*DCNQ*) was prepared from 2,3-dichloro-1,4-benzoquinone [22] (Merck) according to *Budni* [22]. For physical and spectroscopic data of the products, see Tables 2 and 3.

Electron donors. 1,3-Dihydro-2*H*-benzimidazole-2-thione (**1a**), 2(3*H*)-benzothiazolethione (**1b**) and 2(3H)-benzoxazolethione (**1c**) (Aldrich) were used after recrystallization from ethanol.

Organic solvents. The organic solvents used in the present investigation, ethyl acetate and acetonitrile, were purified following the procedures of *Vogel* [23] and Organikum [24], dried, and distilled.

UV/Vis spectrophotometric measurements of the complexes of 2-mercaptobenzazoles **1a–c** *with π-acceptors.* The stoichiometry of all complexes (D/A = 1/1) was determined by *Job's* method [25]. The association constants (K_{CT}) and molar extinction coefficients (ε) of the CT complexes examined were determined using *Benesi-Hildebrand*'s equation [26].

Preparative thin layer chromatography. Air-dried 1 mm layers of silica gel, Merck PF254, on plates (20 cm by 48 cm) were employed for preparative TLC; bands were detected by fluorescence quenching indicator upon exposure to 254 nm UV light and extracted with acetone.

Reaction of 2-mercaptobenzazoles **1a, b** *with TCNE*

A solution of **1a, b** (0.001 mol) in 15 ml of dry acetonitrile was added to a solution of *TCNE* (0.256 g, 0.001 mol) in 10 ml of dry acetonitrile and the reaction mixture was stirred for 48 h at room temperature.

In the case of **1b** the reaction mixture was filtered and the precipitate was washed with cold acetonitrile and recrystallized from ethanol to give pure crystals of compound **13** (47%). The filtrate was concentrated and chromatographed on thin layer plates using toluene/ethyl acetate (1:1) as eluent to give two zones; the first one contained compound **14** (15%), the second compound **15** (22%).

The mixture of **1a** and *TCNE* was concentrated and the residue was then chromatographed on thin layer plates using toluene/ethyl acetate (5:1) as eluent to give numerous zones, two of which were extracted. The fastest migrating one contained compound **4** (33%), the second zone **2** (27%). The base line of the TLC has been rechromatographed using toluene/ethyl acetate (1:1) as eluent to give only one zone containing compound **3** (18%) which was recrystallized from suitable solvent to afford pure crystals.

Reaction of 2-mercaptobenzazoles **1a–c** *with DDQ*

To a solution of *DDQ* (0.3405 g, 0.0015 mol) in 15 ml of dry acetonitrile, a solution of **1a–c** (0.001 mol) in 10 ml of dry acetonitrile was added and the reaction mixture was stirred for 72 h at room temperature. The colour of the reaction mixture changed spontaneously from green to yellowish brown. The mixture was filtered and the precipitate was washed with cold acetonitrile and recrystallized from ethanol to afford pure crystals of compounds **5** (24%) and **16** (42%). The filtrate was concentrated and the residue was then chromatographed on thin layer plates using toluene/ethyl

acetate (10:1) as eluent. Only one zone was well separated, containing 2,2-dibenzimidazolyl sulfide (**4**, 22%) in the case of **1a**, and the benzazolones **17b** (18%) and **17c** (31%) in the case of both **1a** and **1c**, respectively. The base line of the TLC has been rechromatographed using toluene/ethyl acetate (1:1) as eluent to afford *DDQ*-H_2 (**6**) in all cases (33%, 27%, and 38% for **1a**, **1b**, and **1c**, respectively) which was recrystallized from benzene to give pale yellow crystals decomposing at 310°.

Reaction of 2-mercaptoazoles with CHL

To a stirred solution of 0.246 g (0.001 mol) of *CHL* in 20 ml dry acetonitrile, 2-mercaptobenzazoles **1a**, **b** in 15 ml of dry acetonitrile were added. The stirring was continued for 96 h and the colour of the reaction mixture changed to yellowish brown. The reaction mixture was filtered and the precipitate was washed with cold acetonitrile and recrystallized from ethanol to afford pure crystals of **5** (21%) in the case of reaction of **1a** with *CHL*, whereas in the case of **1b**, compound **16** (23%) was obtained. The filtrate was concentrated and the residue chromatographed using toluene/ethyl acetate (5:1) as eluent to give two zones. The fastest migrating one contained *CHL*-H_2 (**9**) in the case of reaction with both **1a** and **1b** (17%, 15%). The slowest migrating zone contained compound **8** (19%) in the case of **1a** and **18** (27%) in the case of **1b**. The base line of the TLC has been rechromatographed using chloroform/methanol (5:1) as eluent. Only one zone was extracted which contained compound **7** (31%) in the case of **1a**, whereas in the case of **1b** compound **19** (21%) was obtained.

Reaction of benzazoles **1a, b** with *DCHNQ*

To a stirred solution of 0.227 g (0.001 mol) of *DCHNQ* in 20 ml of dry acetonitrile, the benzazoles **1a, b** (0.001 mol) in 15 ml acetonitrile were added at room temperature with stirring. The reaction mixture was left at RT for 48 h with stirring. In the case of reaction with **1a**, red crystals of **10** (89%) separated, whereas in the case of **1b** the reaction mixture was filtered and the precipitate was washed with cold acetonitrile and recrystallized from ethanol to afford the disulfide **16b** (28%). The filtrate was concentrated and the residue was chromatographed using toluene/ethyl acetate (5:1) as eluent. Two zones were extracted; the fastest migrating one contained **18** (23%), the second zone **20** (32%). Extraction of the zones with acetone and recrystallization from suitable solvent afforded the pure compounds.

Reaction of benzazoles **1a, b** *with CNIND*

To a stirred solution of 0.208 g (0.001 mol) of *CNIND* in 20 ml of dry acetonitrile, the compounds **1a, b** (0.001 mol) in 15 ml of dry acetonitrile were added dropwise at room temperature and the stirring was continued for 48 h. In the case of the reaction with **1b**, colourless crystals of disulfide **16** (31%) precipitated. Concentration of the filtrate and chromatographic purification of the residue using toluene/ethyl acetate (5:1) gave a yellow zone containing compound **21** (43%). However, in the case of the reaction with **1a**, concentration and chromatographic purification of the reaction mixture on thin layer plates using toluene/ethyl acetate (5:1) afforded **11** (68%). Recrystallization from suitable solvent afforded pure crystals.

Reaction of benzazoles **1a, b** *with DCNQ*

To a stirred solution of 0.208 g (0.001 mol) of *DCNQ* in 15 ml of dry acetonitrile, the benzazoles **1a, b** (0.001 mol) in 15 ml acetonitrile were added at room temperature. The colour of the reaction mixture changed gradually from green to brown. After standing for 72 h, colourless crystals precipitated. Recrystallization from ethanol afforded pure crystals of **5** (47%) in the case of the reaction with **1a**, and **16** (53%) in the case of **1b**. The filtrate was concentrated and chromatographed using toluene/ethyl acetate (1:1) as eluent to give only one zone containing *DCNQ*-H_2 (**12**) for both **1a** (26%) and **1b** (28%).

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