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Ortho-phenylenediamine Based Molecular Receptors in Anion Recognition

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***Abstract*: Recognition and sensing of anionic substrates by artificial host is one of the important areas in field of supramolecular chemistry. Among the various anion binding motifs, ortho-phenylenediamine is one of the important hydrogen bonding motif for anions and the receptors based on this motif can differentiate the anions of different topologies. The electronic and steric effects can influence the binding behavior of the ortho-phenylenediamine motif when it remains connected with the other groups (either chromophoric group or the hydrogen bonding group) via urea or thiourea linkages. To date several ortho-phenylenediamine- based urea/amide or urea-amide conjugates have been reported and this mini review highlights the importance of ortho-phenylenediamine based charge-neutral receptors in anion recognition with selected examples.**

***Keywords*: anion recognition, anion sensing, neutral receptors. ortho-phenylenediamine-based receptors.**

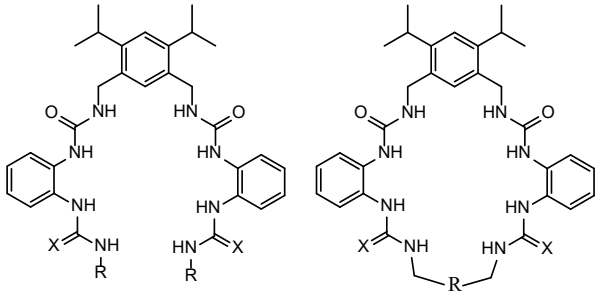
1. Introduction

Design and synthesis of abiotic receptors for anionic substrates is of immense interest in the field of supramolecular chemistry owing to their wide application in biological, environmental and chemical science [1]. In order to recognize and sense the anions, receptors based on different topologies are known in the literature. Receptors for anions range from neutral to charge in nature. The recognition of anion was predominantly achieved using charged host molecule, such as protonated polyamines or azamacrocycles, guanidinium cation, imidazolium or benzimidazolium, pyridinium, transition metal or lanthanide ion based complexes [2]. The disadvantage of charged host molecule lies on the fact that since such coulombic interactions (charge-charge) are non-directional, it is very difficult to achieve higher degree of selectivity. On the other hand, charge neutral receptors containing amide, urea /thiourea, carbamates, amidourea, pyrrole, indole or calixpyrrole with conventional hydrogen bond donors (N-H…..A, A = N, O.), have been widely used for recognition of anions [3].

However, the construction of more sophisticated receptor molecule using arrays of hydrogen bond donor functionalities, where both the selectivity and sensitivity of the recognition process can be finely tuned, is a great challenge to the supramolecular chemists. Careful scrutiny reveals that *ortho*-phenylenediamine is a useful motif for hydrogen bonding of anions and thus, it has been used, in many cases, as the building block for the construction of receptors for anions. Here, a brief review dealing with this important motif is presented here.

ortho-phenylenediamine-based receptors for anion:

The Reinhoudt *et al*. first utilized this motif to prepare a series of macrocyclic and acyclic hosts 1 – 6 [4]. The preorganized urea binding sites of these receptors selectively bind $H_2PO_4^-$. The anion binding properties of these receptors were investigated using ^{1}H NMR in DMSO-d_6. Receptors 1 and 3 were found to bind $H_2PO_4^-$, with complex stoichiometry and no association constants were determined accurately. They were also noticed that in presence of Cl^-, Br^-, NO_3^-, HSO_4^- the aryl urea and aromatic xylene hydrogen hardly shifted and their effects were too small to determine the association constant. On the other hand, 2 and 4 bound with $H_2PO_4^-$ in 2:1 stoichiometry in DMSO-d_6 with K_a = 5 x 10^7 M^{-2} (incorporation of C=S has no effect on the magnitude of binding constant value).



1. X = O, R = -C$_3$H$_7$
2. X = O, R = -Ph
3. X = O, R = -SO$_2$Ph
4. X = S, R = -Ph

5. R =
6. R = -CH$_2$CH$_2$-

Fig. 1. Structures of receptors 1-6

Anions such as Cl^-, Br^-, NO_3^- and HSO_4^- did not induce shifts in the ^{1}H NMR spectra of 27 and the same was true for receptor 3 except for Cl^- which bound with 3 in 1:1 fashion with the K_a = 250 M^{-1}. In termolecular complex of 2.2$H_2PO_4^-$ the first bound $H_2PO_4^-$ anion significantly contributes to the binding of second $H_2PO_4^-$ anion as a result of additional hydrogen bonding between the hydroxyl groups of two anions [5]. This was in accordance with the previously reported dimerisation of $H_2PO_4^-$ in uranyl salophene $H_2PO_4^-$ complex [6]. Recently, few reports in this aspect are worth mentioning [7]. In case of 1 and 3, although significant amounts of 1:1 complexes were formed,

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the lower affinity of these receptors towards binding the second anion was either due to the lower acidity of the second pair of urea moieties in 1 and 3 or due to the greater intermolecular self-association of the urea moieties, which is absent in the receptors 2 and 4 due to the presence of bulky phenyl rings. The macrocyclic receptors 5 (K_a = 2.5 x 10^3 M^{-1} and 500 M^{-1} for $H_2PO_4^-$ and Cl^-, respectively) and 6 (K_a = 4.0 x 10^3 M^{-1} and <50 M^{-1} for $H_2PO_4^-$ and Cl^-, respectively) formed 1:1 complexes with both $H_2PO_4^-$ and Cl^-. Receptor 6 bound with $H_2PO_4^-$ with at least 100-fold selectivity over Cl^- in DMSO-d_6. With 5 and 6 other anions (Br^-, NO_3^- and HSO_4^-) induced shifts were too small to determine either the stoichiometry or the association constants.

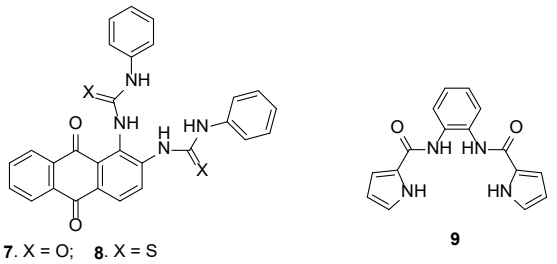


Fig. 2. Structures of receptors 7-9

Das *et al*. utilized the feature of *ortho*-phenylenediamine in designing the colorimetric chemosenors 7 and 8, containing anthraquinone as chromogenic signaling subunit and urea/thiourea as binding sites, for selective sensing of fluoride ion [8]. These receptors did not show affinity for other halide ions (Cl^-, Br^-, and I^- ions). The complexation induced color change (from light yellow to pale red) in the visible region of the spectrum was observed upon addition of F^- ion in DMSO/CH_3CN solution of the receptors 7 and 8 when the temperature of the mixture was systematically raised from room temperature to 60 °C. No such colour change was noticed for other anions under similar experimental conditions.

Cheng and coworkers designed and synthesized *o*-di-(pyrrole-2-carboxamides)-phenylene 9 [9] and identified its two pseudopolymorphs [10] depending on the nature of the solvent used. A helical assembly was observed when it was incorporated with methanol molecules *via* hydrogen bonding. A channel assembly was formed when it was associated with DMSO molecules. The ^1H NMR study revealed that 9 could recognize certain inorganic anions such as $H_2PO_4^-$ (K_a =100 M^{-1}), F^- (K_a = 320 M^{-1}), Cl^- (K_a <10 M^{-1}), Br^-, and I^-. The two-pyrrole carboxamide arms of 9 were found to generate a cleft that was really suitable for binding anion involving hydrogen bonds and the binding stoichiometry in all cases was 1:1 [11]. X-ray crystallographic analyses of the F^-, Cl^-, and AcO^- complexes of receptor 9 showed that in all cases in the solid state the anionic guests were not bound by all of the available hydrogen-bond donors in the receptor species [12]. Gale *et al*. used the same compound 9 for the binding studies of anions in d_6-DMSO containing 0.5% water and reported its carboxylate ion binding ability [13]. The lack of a chromophoric group in this compound hindered the use of optical methods as the detection tool to probe the receptor anion interaction. But this was solved, later on, by synthesizing the compounds 10 – 14 [14]. Changes in the UV-vis and fluorescence spectra in the presence of anions revealed that probes 10 – 14 typically displayed a strong response to cyanide. The appearance of the ratiometric phenomenon upon interaction with anions further enhanced spectral differentiation for anion-sensing. The receptors showed varied interaction modes for different anions stemming from different structures and different pK_a values of the hydrogen bond donors. Depending on the degree of interaction between the anion and the recognition pocket, the electronic perturbation on the chromophore altered the HOMO-LUMO gap and changed the color of the solution, the fluorescence, and the fluorescence intensity.

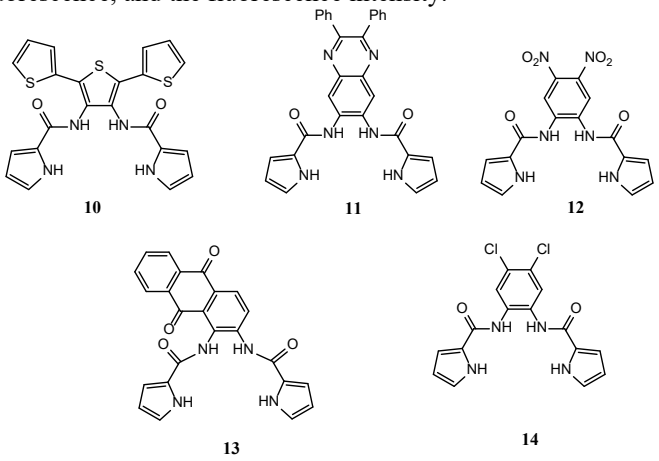


Fig. 3. Structures of receptors 10-14

In contradistinction to these results, the X-ray crystal structure of the benzoate complex of receptor 15 showed the anion bound by all four of the urea NH protons in an essentially symmetrical arrangement [15]. Introduction of electron withdrawing groups on the central aromatic ring affected the binding affinity for anions and thus the receptor 16 was noted to bind benzoate with higher binding constant value. Interestingly, the cleft of 16 bound benzoate in a different fashion from that of 15. In relation to this, the presence of electron withdrawing $–NO_2$ groups in compound 17 played different role in the association of anions. Significant improvements in the observed binding constants for both AcO^- and $C_6H_5CO_2^-$ were noted for 16 relative to 15. The anion binding stability constant values for compound 17, however, were only slightly higher than those observed for compound 15. Gale *et al.* explained this as a result of an increase in the preorganization of binding site within 16 relative to 17 arising from the localization of acidity within the molecule. The presence of electron withdrawing groups on the central ring influences the formation of intramolecular CH---O hydrogen bonds with the urea carbonyl oxygen and thus enables the molecule 16 to adopt a more preorganized conformation. On contrary, the $–NO_2$ groups in 17 control the conformation of the binding site involving CH---O interactions in a different way. Fig. 5 demonstrates this aspect. In order to explore this phenomenon, Gale *et al*. synthesized compounds 18, 19 and 20. Due to similar reason, the compound 19 showed high affinity for anions.

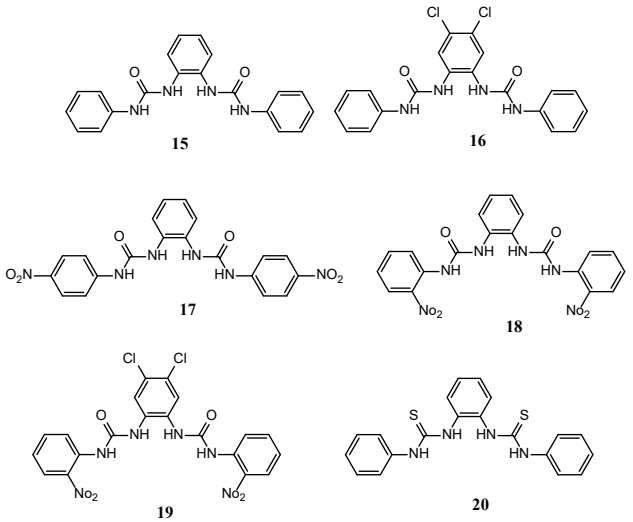


Fig. 4. Structures of receptors 15-20

When bis-thiourea 45 was taken, the measured stability constants with carboxylates were almost an order of magnitude lower than those obtained with 40 (the urea analogue). But in general the stability constants of thioureas with anions are higher due to the greater acidity of the NH protons [16] Such finding is attributed to the fact to the larger sulfur atom that distorts the shape of the binding site (Figure 2A.1) with the outer thiourea NH groups no longer capable of simultaneously coordinating to the anion.

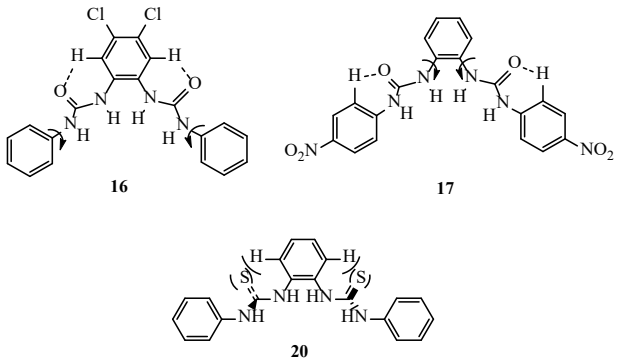


Fig. 5. Intramolecular hydrogen bonding and the conformational aspects of 16, 17 and 20

Kim *et al*. also synthesized *ortho*-phenylenediamine-based some colorimetric anion sensors 21 and 22 where 4-nitrophenyl was treated as a signaling unit and urea/thiourea moieties as binding sites [17]. The receptors, effectively and selectively, recognized the biologically important F^- and carboxylate anions from other anions such as Cl^- and Br^- in DMSO.

Interestingly, when two *ortho*-phenylenediamine-based bis urea groups are combined in a single receptor, complex formation with dicarboxylates led to the formation of hydrogen – bonded molecular tapes in the solid state [18]. Compounds **23** and **24** belong to this category.

In addition, anion binding chemistry of two another new *ortho*-phenylenediamine based bis – urea compounds **25** and **26** were discussed by Gale *et al*. in 2007 [19]. They reported the crystal structures of the acetate and benzoate complexes of **25** and **26**..

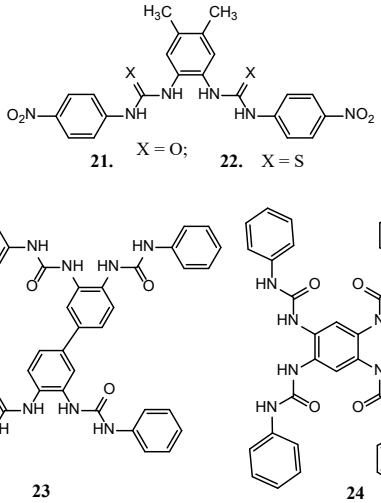


Fig. 6. Structures of receptors 21-24

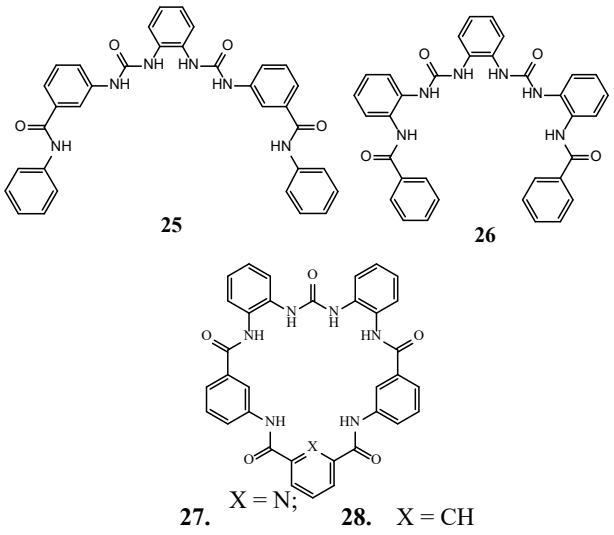


Fig. 7. Structures of receptors 25-28

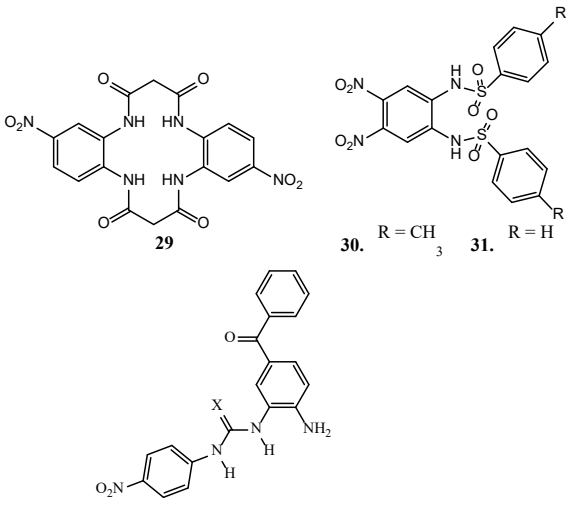
The same group reported the macrocyclic structures 27 and 28, which showed dramatic differences in the mode of interaction of anions. While the macrocycle 27 formed stable hydrogen-bonded complexes with carboxylate anions and displayed a 100-fold selectivity for acetate versus dihydrogen phosphate in d_6-DMSO containing 0.5% or 5% water. In contrast, macrocycle 28 showed lower affinity and selectivity for carboxylates than 27. The difference in stability was attributed to the absence of pre-organizing influence of the pyridine group as maintained in 27 [20].

Lin and coworkers reported the synthesis and anion binding properties of macrocycle 29 [21]. UV-Vis and ^{1}H NMR titration experiments in DMSO or DMSO-d_6 revealed that the receptor was selective for F^- over AcO^-, $H_2PO_4^-$, Cl^-, Br^- and I^- and that the recognition process caused a change in the color of the receptor due to hydrogen bond formation and not because of deprotonation. The result was explained by means of theoretical investigations.

Lin and coworkers also synthesized and studied the

recognition properties of 1,2-bis-(*p*-methylphenylsulfonamido)-4,5-bisnitrobenzene 30 and 1,2-bis-(phenyl-sulfonamido)-4,5-bisnitrobenzene 31 with halide anions by UV–vis and ^{1}H NMR titration experiments [22]. The studies of UV–vis spectra clearly showed that the affinity constants of 30 and 31 to F^- are about 1.4 x 10^4 and 2.5 x 10^4 M^{-1}, respectively, almost 1200- and 1500-fold greater than that for Cl^-, Br^- and I^-. The color of the solution changed from yellow to red with the increase of F^- anion concentration. When the concentration of F^- ion was 30-fold greater than that of receptor 30, there was no further change of electronic spectrum or color.

Recently, Kang *et al.* reported two colorimetric chemo sensor 32 and 33, with two signaling unit, benzophenone and *p*-nitrophenyl groups [23]. These receptors can discriminate F^- and carboxylates from other anions such as NO_3^-, ClO_4^-, HSO_4^-, Cl^-, Br^- and I^- in DMSO. Moreover, compounds 32 and 33 were proved to be efficient for naked eye detection of F^- in DMSO.



29 **30.** R = CH_3 **31.** R = H

32. X = O **33.** X = S

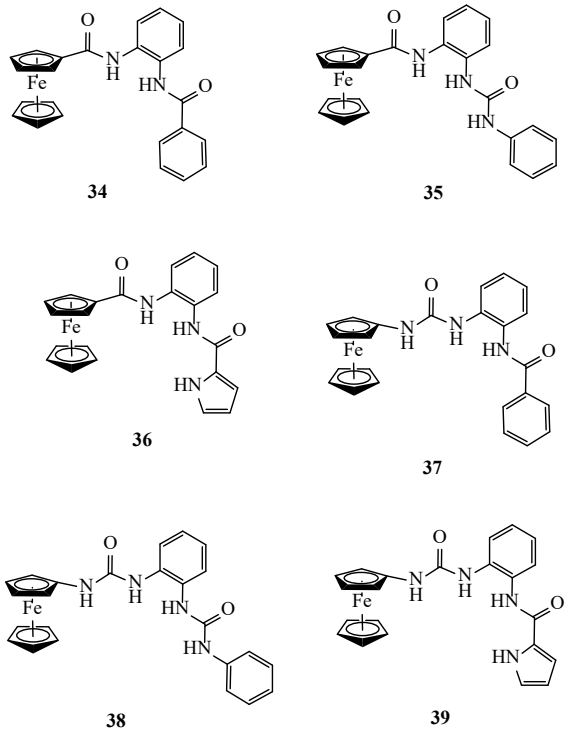
Fig. 8. Structures of receptors 29-33

Gale *et al*. reported a series of ferrocene fuctionalized anion receptors 34 – 39 based on *ortho*-phenylenediamine scaffold which was found to undergo anion-triggered unusual electrochemical deposition giving a new way to detect anionic substrates [24]. Electrochemical investigations of the ferrocene derivatives showed both anodic and cathodic processes, which, if assumed to be a one-electron transfer, can be considered to be close to reversible in nature (peak-to-peak potential 69 mV and ratio of the anodic to cathodic peak 0.98). The electrochemical behavior of the compounds showed a distinct change depending upon the presence of the binding anion.

Katayev *et al.* reported the synthesis, characterization and anion-binding properties of new pyrrole–pyridine-based macrocyclic polyamides 40a and 40b [25]. The new receptors displayed 10-fold selectivity for HSO_4^-, $H_2PO_4^-$ and AcO^- anions over other anions studied.

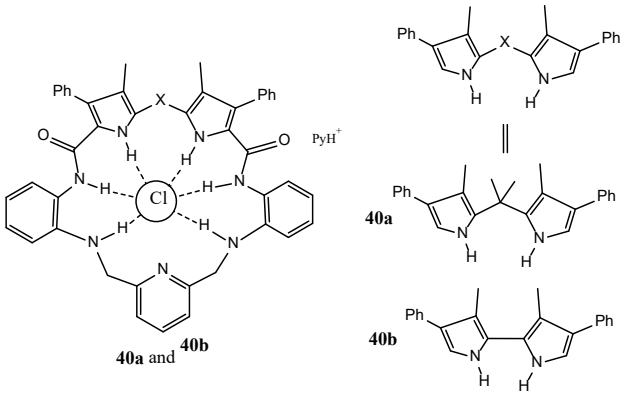
Receptors 40a and 40b exhibited lower affinity for Cl^- and F^- anions than for oxoanions. Dipyrrlomethane-based receptor 40a was found to be selective for the HSO_4^- anion with association constant an order of magnitude higher than other inorganic anions. The bipyrrole-based receptor 40b was found to be selective for $H_2PO_4^-$ and AcO^- anions. The relatively high affinities of these polyamide receptors toward this selection of anions even in DMSO were explained by the preorganised conformation for oxoanion binding.

Chauhan *et al*. utilized *ortho*-phenylenediamine cleft in the form of phenazine derivatives 41 and 42 having bis-urea/thiourea binding sites [26]. The interaction and colorimetric sensing properties of 41 and 42 with different anions were investigated by naked eye, UV–vis and fluorescence spectroscopy in DMSO. The selectivity trend in the binding affinities of the anions for 41 and 42 followed the order of $F^- > AcO^- > H_2PO_4^- >> Br^- > Cl^- > HSO_4^- > I^-$. The observed binding sequence was not completely consistent with the anion basicity. The receptors/sensors 41 and 42 not only capable for the easy colorimetric detection of F^- and $H_2PO_4^-$ and AcO^- ions, but also are amenable to 'color tuning' depending upon the type and amount of anions used.



34 **35** **36** **37** **38** **39**

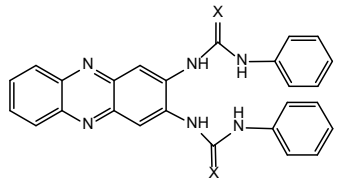
Fig. 9. Structures of receptors 34 – 39



40a and **40b**

40a **40b**

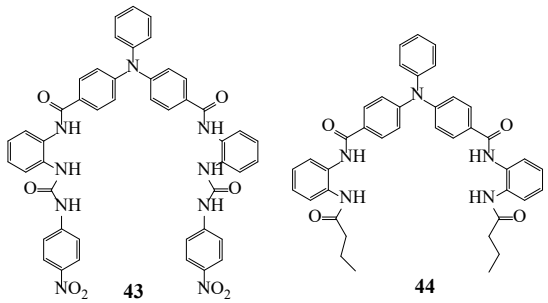
Fig. 10. Structures of receptors 40a and 4b and their chloride complexes



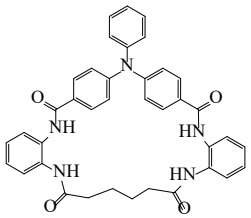
41 X = O **42** X = S

Fig. 11. Structures of receptors 41 and 42

Recently, Ghosh et. al. reported triphenylamine-based receptors 43-45 for recognition of aliphatic dicarboxylates of various chain lengths [27], [28]. In all the designs *o*-phenylenediamine in the form of an amide or amide-urea conjugate has been utilized as the binder of carboxylate. These molecules are found to bind the dicarboxylates with moderate binding strength under a semi rigid, propeller shaped, fluorescent triphenylamine spacer.



43 **44**



45

Fig. 12. Structures of receptors 43 – 45

The binding behavior was studied in polar solvents using ^{1}H NMR, fluorescence and UV-vis spectroscopic methods. The selectivity and sensitivity of the receptors depends on the number of hydrogen bonding groups in the *o*-phenylenediamine motif, disposed around the triphenylamine core. Binding takes place at the charge neutral binding sites of the receptors with concomitant change in emission. The macrocylic receptor 45 is more symmetric and efficient in selective binding and sensing of a particular dicarboxylate, glutarate, in the present study. Receptor 43 shows higher binding constant values than receptor 44 and discriminates the different dicarboxylates effectively due to 1) more acidic character of the urea protons as well as 2) least flexible character of the geometry. Further, the receptor 43 displayed visual colour change from yellow to red only in the presence of malonate anion.

2. Conclusion

The *ortho*-phenylenediamine is easily functionalizable anion binding motif and often used as their amide, urea, thiourea and amide-urea derivatives for anion recognition. Anion selectivity could be finely tuned by introducing electronic and steric components to the receptors. This allows creation of great deal of variety in the receptor structures. Thus, the *ortho*-phenylenediamine should continue to be important in anion recognition because of its ease of synthesis and anion complexing capability. Future advances should focus on binding in aqueous solvent as well as use of these receptors in transmembrane transport of various biologically important anions.

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