

Structure of seven organic acid–base adducts formed between acids, 2-aminophenol, and 2-amino-4-chlorophenol

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HIGHLIGHTS

- ▶ Seven organic acid–base adducts have been prepared and structurally characterized.
- ▶ The hydrogen bond interaction modes in all of the adducts have been ascertained.
- ▶ The classical hydrogen bonds are the major forces in all of the structures.
- ▶ The secondary interactions also play important roles in the packing of the adducts.

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ABSTRACT

Studies concentrating on hydrogen bonding between the base of 2-aminophenol, 2-amino-4-chlorophenol, and acidic compounds have led to an increased understanding of the role 2-aminophenol, and 2-amino-4-chlorophenol have in binding with acidic compounds. Here anhydrous and hydrated multicomponent crystals of 2-aminophenol, and 2-amino-4-chlorophenol have been prepared with 2,4,6-trinitrophenol, *p*-nitrobenzoic acid, 3,5-dinitrobenzoic acid, 5-nitrosalicylic acid, 3,5-dinitrosalicylic acid, and fumaric acid. The crystals and complexes of the seven organic acid–base adducts were characterized by X-ray diffraction analysis, IR, mp, and elemental analysis. The seven crystalline forms reported are organic salts except **2**. All of the adducts were formed in solution and obtained by the slow evaporation technique. All supramolecular architectures involve extensive N–H···O, O–H···O, and CH···O hydrogen bonds as well as other nonbonding interactions. The role of weak and strong hydrogen bonding in the crystal packing is ascertained.

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1. Introduction

The design and construction of multicomponent supermolecules or supramolecular arrays utilizing noncovalent bonding is a rapidly developing area in supramolecular synthesis. However, self-assembled supramolecular architectures are often stabilized as a result of the synergy of a variety of weak noncovalent interactions [1–3]. Thus, the supramolecular synthesis successfully exploits hydrogen-bonding and other types of non-covalent interactions, in building supramolecular systems [4].

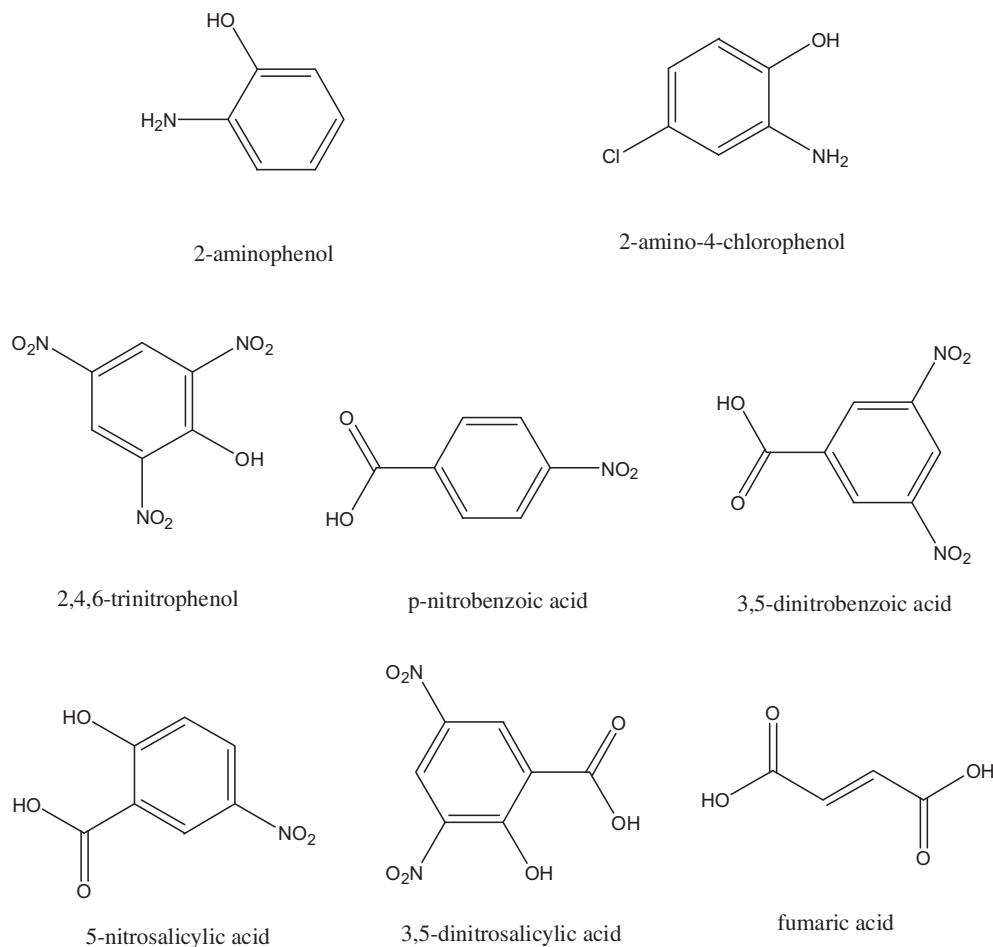
Carboxylic acids represent one of the most prevalent functional groups in crystal engineering because they possess a hydrogen bond donor and acceptor with a geometry that facilitates self-association through supramolecular homosynthons via centrosymmetric dimer or catemer [5–7]. Furthermore, it is now recognized that carboxylic acids are ideal candidates for multicomponent crystals

since they form persistent supramolecular heterosynthons with a number of different complementary functional groups such as amine, and aromatic nitrogen, etc. For instance, much has been said about the use of carboxyl and pyridinyl groups in the design of supramolecular systems [8–13]. Besides the COOH group, the functional groups such as amine, halogen, and phenol OH groups are all good groups in forming organic solid through non-covalent interactions [14]. Aminophenols containing equal stoichiometries of –OH, and –NH₂ groups have been widely studied to understand the supramolecular synthons existing in their assemblies [15]. Among these supramolecular architectures, however, only a very few reports described the crystals composed of aminophenols derivatives and carboxylic acids [16].

In order to understand the interaction modes aminophenols derivatives have in binding with acidic derivatives, we began to study the aminophenols–acids system, also aiming to find the role the weak noncovalent interactions played in forming the final supramolecular frameworks. Thus, in the following, we report the preparation and crystal structures of seven supramolecular

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Scheme 1. The building blocks discussed in this paper.

compounds assembled via nonbonding interactions between carboxylic acids and 2-aminophenol (L1), and 2-amino-4-chlorophenol (L2) (Scheme 1). In this study, we got seven organic compounds composed of acidic units and 2-aminophenol derivatives, namely (2-aminophenol): (2,4,6-trinitrophenol) [(HL1)⁺ · (pic⁻), pic⁻ = picrate, L1 = 2-aminophenol] (**1**), (2-amino-4-chlorophenol): (p-nitrobenzoic acid): H₂O [(L2) · (Hnba) · H₂O, Hnba = p-nitrobenzoic acid] (**2**), (2-aminophenol): (3,5-dinitrobenzoic acid) [(HL1)⁺ · (dnb⁻), dnb⁻ = 3,5-dinitrobenzoate] (**3**), (2-amino-4-chlorophenol): (3,5-dinitrobenzoic acid) [(HL2)⁺ · (dnb⁻)] (**4**), (2-aminophenol): (5-nitrosalicylic acid) [(HL1)⁺ · (5-nsa⁻), 5-nsa⁻ = 5-nitrosalicylate] (**5**), (2-amino-4-chlorophenol): (3,5-dinitrosalicylic acid) [(HL2)⁺ · (3,5-dns⁻), 3,5-dns⁻ = 3,5-dinitrosalicylate] (**6**), and (2-amino-4-chlorophenol): (fumaric acid)_{0.5} : H₂O [(HL2)⁺ · (fum²⁻)_{0.5} · H₂O, fum²⁻ = fumarate] (**7**) (Scheme 2).

2. Experimental section

2.1. Materials and physical measurements

The chemicals and solvents used in this work are of analytical grade and available commercially and were used without further purification. The FT-IR spectra were recorded from KBr pellets in range 4000–400 cm⁻¹ on a Mattson Alpha-Centauri spectrometer with 4 cm⁻¹ resolution. Microanalytical (C, H, N) data were obtained with a Perkin-Elmer Model 2400II elemental analyzer. Melting points of new compounds were recorded on an XT-4 thermal apparatus without correction.

2.2. Preparation of supramolecular compounds

2.2.1. (2-Aminophenol): (2,4,6-trinitrophenol) [(HL1)⁺ · (pic⁻), pic⁻ = picrate] (**1**)

2-Aminophenol (10.9 mg, 0.10 mmol) was dissolved in 3 mL methanol. To this solution was added 2,4,6-trinitrophenol (23 mg, 0.1 mmol) in 10 mL methanol. Brown block crystals were obtained after several days by slow evaporation of the solvent (yield: 26 mg, 76.87%). mp 147–148 °C. Elemental analysis: Calc. for C₁₂H₁₀N₄O₈ (338.24): C, 42.57; H, 2.96; N, 16.56. Found: C, 42.51; H, 2.89; N, 16.52. Infrared spectrum (KBr disc, cm⁻¹): 3589s(v(OH)), 3440s(v_{as}(NH)), 3306s(v_s(NH)), 3190s, 3100s, 2940m, 2880m, 2848m, 2820m, 2676w, 2568w, 2370m, 2330m, 1740m, 1645m, 1614s, 1600s, 1560s, 1525s(v_{as}(NO₂)), 1480m, 1440m, 1360s, 1324s(v_s(NO₂)), 1260s, 1240m, 1210m, 1160m, 1020m, 980m, 920m, 878m, 840m, 790m, 720m, 680m, 660m, 620m.

2.2.2. (2-Amino-4-chlorophenol): (p-nitrobenzoic acid): H₂O [(L2) · (Hnba) · H₂O, Hnba = p-nitrobenzoic acid] (**2**)

2-Amino-4-chlorophenol (14.3 mg, 0.1 mmol) was dissolved in 2 mL methanol. To this solution was added p-nitrobenzoic acid (17 mg, 0.1 mmol) in 2 mL methanol. Colorless block crystals were afforded after several days by slow evaporation of the solvent (yield: 18 mg, 54.76%, based on L2). mp 213–214 °C. Elemental analysis: Calc. for C₁₃H₁₃ClN₂O₆ (328.70): C, 47.46; H, 3.95; N, 8.52. Found: C, 47.37; H, 3.89; N, 8.49. Infrared spectrum (KBr disc, cm⁻¹): 3596s(v(OH)), 3230s(v_{as}(NH)), 3176s(v_s(NH)), 3079 m, 2989s, 2926s, 2682w, 2567w, 1763w, 1648s(v(C=O)), 1620m,

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