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We report four structures of primary ammonium salts of the anti-inflammatory agent diclofenac (D), and we complete the salt sequences from t-butylammonium to Tris and mono- to triethanolammonium, seeking to optimize formulation properties.

Primary ammonium salts form columns made from hydrogen bonded (HB) rings. The highest-melting primary ammonium salts have $R_4^3(10)$ columns (melting points by DSC in triplicate; estimated errors in parentheses). The six independent ion pairs of the cyclohexylammonium salt are related by pseudosymmetry.

R group	Z'	Graph set for HB ring(s)	M. p. /°C
t-butyl	1	$R_4^2(8)$ & $R_4^4(12)$	166(2)
cyclohexyl	6	2 independent $R_4^3(10)$	191(2)
benzyl	1	$R_4^4(12)$	108(2)
adamantyl	1	$R_4^3(10)$	242(2)

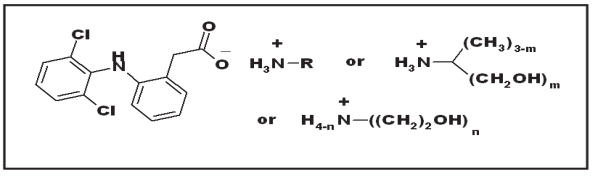
Hydroxymethyl derivatives have HB to OCO^- from OH as well as NH^+. Although $R_4^3(10)$ columns persist when m=1, they are distorted and presumably bring less stability.

m	Z'	H bond donors to OCO^-	M. p. /°C
1	1	$R_4^3(10)$, $R_2^2(9)$ NH^+&OH...O^-	143.7(6)
2	1	$R_3^3(8)$ NH^+&OH...O^-; $R_4^2(14)$	179(2)
3 (TUDPIR)	1	$R_3^3(8)$ NH^+&OH...O^-; $R_4^2(14)$	207.2(3)

Hydroxyethyl derivatives with n=1 or 2 also use these groups, but when n=3 the NH^+ is blocked [1]. Two ion pairs of the mono-hydroxyethyl compound differ in their HB: one D lacks the ubiquitous $S_1^1(7)$ intramolecular N-H…O^-, and only one pair has cation-cation NH^+…OH. Pseudosymmetry relates the other two.

n	Z'	H bond donors to OCO^-	M. p. /°C
1	4	$R_4^2(8)$ NH^+...O^-; $R_2^2(9)$	140.3(2)
2 (ZIKPOY)	1	$R_4^2(8)$ NH^+...O^-; $C_2^2(9)$	131.2(3)
3 (TEKVAG)	1	$C_2^2(12)$ OH...O^-	135(2)

HB motifs but not Z' obviously affect stability. We thank the NCS, Southampton, for data on these often difficult specimens.



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Halogen Interactions Using Variable Temperature Single Crystal X-ray Diffraction

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The studies of intermolecular interactions involving halogen atoms in small organic molecules have been a controversial topic in contemporary research [1]. While a number of research groups have indicated that F behaves differently than Cl and Br in building crystal lattices [2]. mostly using Cambridge Structural Database; many other research groups have shown significant amount of experimental evidence of the active role of weak interactions such as C-H···F, C-F···F and C-F···π like C-H···X, C-X···X, C-X···π (X = Cl and Br) in crystal packing [3]. The natures of these interactions are yet to be well understood. The importance of fluorine in pharmaceutical industry, especially in medicinal application of small fluorinated organic molecules have gained significant interest in scientific literature [4]. We have been interested in this field as fluorine is one of the major substitutions found in drugs and drug intermediates and the C-F group in these play a major role in their biological activity [4b,c].In order to understand the nature and modes of weak interactions offered by a C-F group, we have chosen few model molecules such that these interactions can be studied both in the presence and absence of stronger hydrogen bonding interactions. We have also studied the crystal structures of the same set of compounds where F is replaced by Br or Cl. The altered packing modes of these molecules show that aromatic C-F groups provide significant interactions to stabilize crystal packing. These crystal structures have been studied at different temperatures to get an indication about the nature (attractive or repulsive) of these interactions. The salient features of our recent studies will be presented and the significance of weak interactions offered by the so called "organic fluorine" will be highlighted.

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Subtle Interplay of Hydrogen Bonds and Weak Interactions in Drugs

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Classical hydrogen bonds [1] are known to take the leading role in generating the supramolecular assemblies, both in the solution and in the solid state and influence the formation of various crystal lattices of all organic, organometallic and biological macromolecules [2]. Weak