European Journal of Chemistry 1 (2) (2010) 61-66

European Journal of Chemistry

Journal homepage: www.eurjchem.com

The supramolecular structures of oximes: an update and the crystal structure of 1,3-diphenyl-propan-2-one oxime

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ARTICLE INFORMATION

Received: 27 April 2010
Received in revised form: 21 May 2010
Accepted: 21 May 2010
Online: 30 June 2010

KEYWORDS

H-bonding
Oximes
1,3-diphenyl-propan-2-one oxime
X-ray diffraction
Crystal structure

ABSTRACT

The crystal structure of 1,3-diphenyl-propan-2-one oxime, $C_{15}H_{15}NO$, is described. The compound crystallises in the monoclinic space group *C*2/*c*. Centrosymmetrically related molecules are linked to form R_2^2 (6) dimers. An update, since 2003, of a systematic analysis of the hydrogen bonding patterns in oxime structures with and without competitive O-H...A type acceptors (an acceptor other than the nitrogen of the oxime) functional group is made, taking into account their moieties. The majority of these oximes form dimeric, R_2^2 (6), structures but R_3^3 (8) and R_4^4 (12) were also found. C3 chains which were classically claimed as the usual oxime H-bond pattern were rarely observed. They are mostly found in aldoxime structures.

1. Introduction

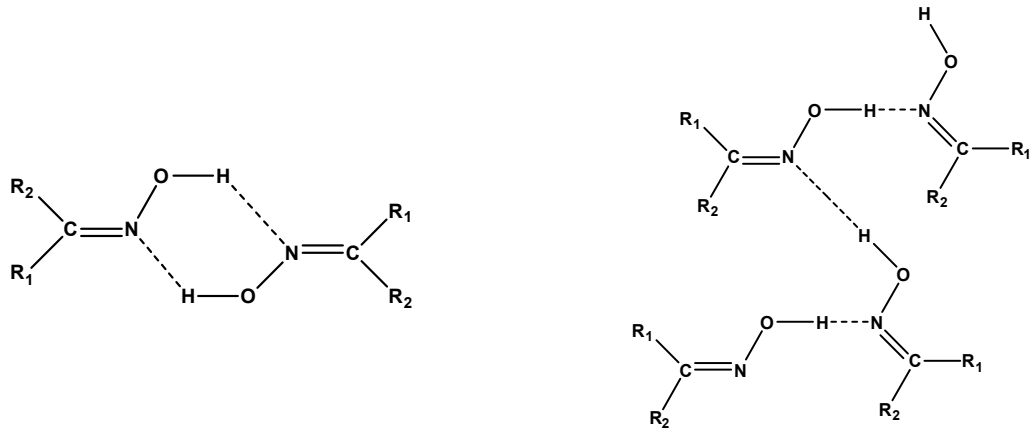
Hydrogen bonds are viewed as the strongest and most directional of the intermolecular interactions and thus play the major part in the formation of supramolecular structures. Potential H bond donors and acceptors are able to combine in different ways with strong H donors tending to combine with strong H acceptors [1]. Hydrogen bonding patterns of the oxime functional group have not been studied as intensively as their carboxylic and amidic counterparts. However, Bertolasi *et al.* [2] analyzed the hydrogen-bond patterns in crystalline oximes and this was followed by a review of hydrogen patterns in aromatic and aliphatic dioximes by Bruton *et al.* [3]. In these studies, it was found that oximes are usually associated *via* O-H···N hydrogen bonds of length around 2.8 Å. The most usual arrangements for H bond motifs are R_2^2 (6) dimers or C3 catemers (Figure 1). Bruton *et al.* [3] emphasise the importance of the 'hydrogen bonding capability of the oxime functional group (—C(R)=NOH)' in aromatic and aliphatic dioximes. The frequent occurrence of the O—H···N hydrogen bonded, R_2^2 (6) dimers means that they can be used as synthons for supramolecular synthesis in the case of dioximes. Furthermore, it seems that oxime groups possess stronger hydrogen-bonding capabilities than those found in alcohols, phenols and carboxylic acids [4]. Although perhaps not as important as synthons in supramolecular aggregation, the hydrogen bonding of ketoximes is of interest since they are important reagents as they do provide an important route to the preparation of synthetic polyamides via the Beckmann rearrangement and serve as antidotes in organophosphate poisoning [5]. However to date there has not been any systematic analysis of H bond motifs of oxime functional group, e.g. ketoximes, aldoximes, O-alkylated ketoximes (Figure 2). Oxime structures with another competitive O-H···A type acceptor (an acceptor other than the nitrogen of the oxime) have not been discussed. Since 2003 many new oxime structures have been published and so, apart from the structural characterization of a new ketoxime, 1,3-diphenyl-propan-2-one oxime, **I**, (Scheme 1) an update of their H-bond capability is made in this work. In fact crystals suitable for X-ray diffraction of **I** were obtained when the oxime (1,3-diphenyl-propan-2-oxime) was prepared as a precursor in the preparation of ultra pure 1,3-diphenyl-propan-2-one.

2. Experimental

2.1. Synthesis and purification of compound 1

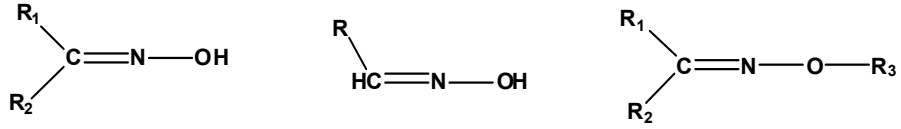
1,3-diphenylpropan-2-one oxime, **I**, was obtained from the condensation reaction of 1,3-diphenylpropan-2-one with hydroxylamine hydrochloride. A solution of 12 mmol of hydroxylamine hydrochloride and 30 mmol of NaOH in an ethanol/H_2O mixture (v/v, 4:1) was added to 10 mmol of 1,3-diphenylpropan-2-one and heated to reflux until completely dissolved. After 1 h the reaction mixture was cooled to room temperature and acidified with HCl (aq), precipitating a white solid. The solid was extracted with CH_2Cl_2, and the organic phase washed with water, dried and evaporated, yielding a white solid with brown impurities. Washing with Et_2O afforded 1.65 g of pure 1,3-diphenylpropan-2-one oxime (overall yield = 77 %). Crystals suitable for X-ray diffraction were grown from CH_2Cl_2.

ISSN 2153-2249 (Print) / ISSN 2153-2257 (Online)
DOI:10.5155/eurjchem.1.2.61-66.76



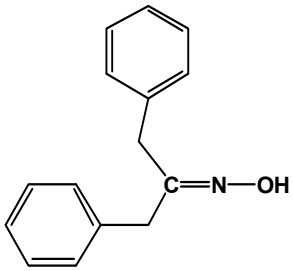
I II

Figure 1. The most common hydrogen bonding motifs in oximes: **I**- $R_2^2(6)$ rings and **II**– C3 chains, according to Bertolasi *et al.* [2] and Bruton *et al.* [3].



(a) (b) (c)

Figure 2. Oximes and their moieties discussed on this work. (a) Ketoximes; (b) Aldoximes; (c) O-alkyl-ketoximes.



Scheme 1. Schematic representation of compound **I**.

2.2. Crystallographic measurements

Crystal data, data acquisition conditions and refinement parameters for **I** are listed in Table 1. The intensity data was collected on a Bruker-Nonius CCD diffractometer. Data collection, cell refinement and data reduction were made with software package of the diffractometer, *COLLECT* [6]. Cell refinement: *DIRAX/LSQ* [7]; data reduction: *EVALCCD* [8] and absorption correction [9]. The structure was solved using the following software: *SIR2004* [10,11] and refined using the *OSCAIL* [12] version of *SHELXL97* [13]. H atoms were treated as riding atoms with C—H (aromatic), 0.95 Å and C—H (CH_2), 0.99 Å. The position of the hydroxyl H atom was located on a difference map, and refined as a riding atom and rotating group about the N—O bond. The molecular diagrams were made with ORTEP [14] and PLATON [15].

3. Results and Discussion

3.1. Crystal structure of compound I

Figure 3 shows a diagram of the molecular structure for 1,3-diphenyl-propan-2-one oxime, **I**, with the adopted label scheme. The bond lengths and angles show no unusual features [16]. The dihedral angles between the mean planes of each of the phenyl rings, (identified with atom labels C1*X* and C3*X*, where *X* has values of 1 to 6), with respect to the best plane of the 2-ketoxime moiety are of 79.91(4)° and 87.92(4)°, respectively. The conformations of the phenyl groups with respect to the C3–C2 and C2–C1 bonds are respectively, the phenyl C3*X* ring is in a *cis* type conformation to the ketoxime while the phenyl ring C1*X* adopts a *trans* conformation.

Table 1. Crystal data, data acquisition conditions and refinement parameters for 1,3-diphenyl-propane-2-one oxime.

Empirical formula	$C_{15}H_{15}NO$
Formula weight	225.28 g
Temp	123 (2) K
λ	0.71073 Å
Crystal system	Monoclinic, *C*2/*c*
Cell constants	*a* =20.6027(10) Å
	b = 5.7547(3) Å
	c =20.6827(11)Å
	β = 104.651(2)°
Volume	*V* =2372.5(2) Å^3
Molecules per unit cell, *Z*	8
D_x	1.261 Mg m^{-3}
F(000)	960
Crystal size/shape/colour	0.50 × 0.10 × 0.06 mm (needle, colourless)
Θ range for data collection	3.0–30.6°
Absorption coefficient	*μ*= 0.075 mm^{-1}
Radiation source	Fine-focus sealed tube
Monochromator	Graphite
θ range	θ_{min} = 3.0°; θ_{max} = 30.6°; ω scans
Limiting indices	*h* = -18⍰29; *k* =-7⍰5; *l* = -29⍰29
Reflections collected/unique	6181 / 3389; 2737 with $I > 2\sigma(I)$; R_{int} = 0.0166
Max and Min transmission	T_{min} = 0.962, T_{max} = 0.995
Data/restraints/parameters	3389 / 0 / 138
Goodness-off-fit on F^2	1.015
Final *R* indices	$R[F^2 > 2\sigma(F^2)]$ = 0.0433; $wR(F^2)$ = 0.1092
Final *R* indices (all data)	*R* = 0.0555; $wR(F^2)$ = 0.1177
$\Delta\rho_{max}$, $\delta\rho_{min}$	0.33 e Å^{-3} and -0.21 e Å^{-3}
Refinement method	Full matrix least-squares on F^2
CCDC no.	736962

This relative conformation of the aromatic rings towards the functional group precludes the possibility of the occurrence of intramolecular π···π· or C–H···π stacking interactions. In **I** the supramolecular structure is formed by an O–H···N hydrogen bond which links the molecules into R_2^2 (6) dimers [17]. In addition a C–H···π interaction contributes to the supra molecular structure stabilisation. The geometric parameters

are summarised in Table 2. The O-H···N bond forms an R_2^2 (6) dimer ring, in which O2 (*x*, *y*, *z*) acts as a hydrogen donor to N2 (0.5-*x*, 0.5-*y*, 1-*z*) as depicted in Figure 4. The C12–H12···Cg1 (0.5-*x*, -0.5+*y*, 0.5-*z*), (Cg1 is the centre-of-gravity of the ring C1*X*), C-H···π interaction links the dimers to form corrugated sheets which run parallel to (100), Figure 5. R_2^2 (6) dimers of this type are commonly found in oxime structures as reported by Bruton *et al*. [3] and they were also observed in the update of the supramolecular structures of oximes presented in the next section.

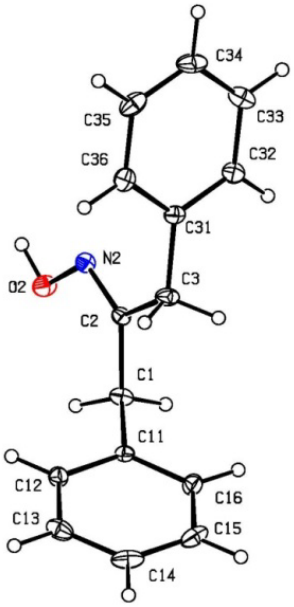


Figure 3. A view of 1,3-diphenylpropan-2-one oxime with our numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

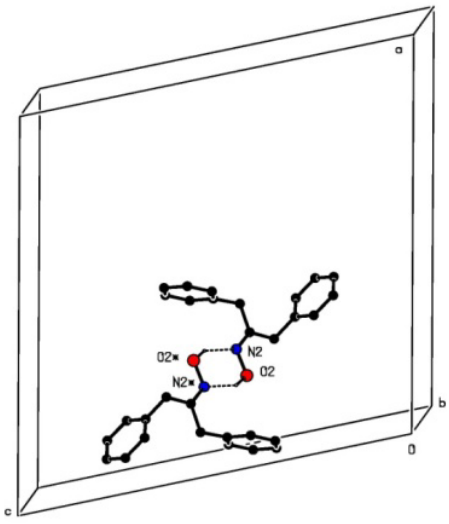


Figure 4. A view of the R_2^2 (6), O–H...N hydrogen-bonded dimer. Hydrogen atoms not involved in the motif are not included.

Table 2. Intermolecular interactions (Å, °) for 1,3-diphenylpropan-2-one oxime. π_i is the centroid of the C11-C16 ring.

D–H....A	D–H	H···A	D···A	D–A···A
O(1) –H(2) ···N(2)^{i}	0.90	2.03	2.8299(13)	149
C(14) –H(14) ··· π_i ^{ii}		2.72	3.4497(12)	134

Symmetry codes: ^{i}1/2-*x*, -1/2-*y*, 1-*z*; ^{ii}1/2-*x*, -1/2+*y*, 1/2-*z*.

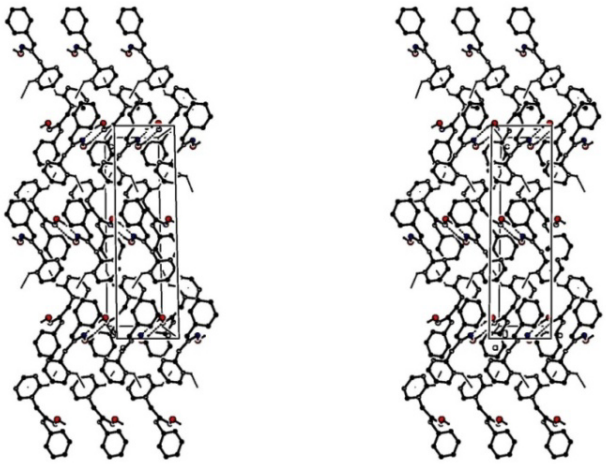


Figure 5. A stereographic view of part of the crystal structure of **1**, showing dimers linked by the C-H···π interaction to form sheets which run parallel to (100). Hydrogen atoms not involved in the motifs are not included.

3.2. Supramolecular structures of oximes

Oxime functional group offers a hydrogen bond donor (OH···) and two hydrogen bond acceptor sites (the C=N and O–H lone pairs). In the solid state the O=H...N interactions are preferred even with the angular constraints due to the rigid geometry of oxime. The formation of dimers is common among oximes. A further investigation of the H–bond motifs for oximes was performed using the search on the CSD [18], Version 5.31 (November 2009). The search was based on oxime structures reported during or after 2003.

Data were selected on the basis of the following criteria; 3D coordinates were determined, the R factor was ≤ 0.10, no disorder, no errors were present, no polymers were included and no ions were present, only organic structures were examined and the data were from single crystal studies only. The list of these structures is given in supplementary information. The list contains entries for 216 structures of oximes consisting of 119 entries for ketoximes (moiety (a) in Figure 2), 40 entries for aldoximes (moiety (b) in Figure 2) and 57 for O-substituted ketoximes (moiety (c) in Figure 2). In the analysis which follows we include, in addition, the data for **I** giving a total of 217 structures with 120 entries for ketoximes. Dioximes are not included in the list or in the analysis.

3.2.1. Ketoximes

Ketoximes examined were grouped in two different sets: those containing competitive acceptors (O–H (oxime ···A) or donors (D-H ···N (oxime)); A=acceptor; D=donor; with 95 entries and those without other competitive acceptors and donors comprising of 25 entries. Ketoximes were further divided into 3 categories according to their chemical moiety e.g. aromatic or aliphatic ketoximes (type a1), cyclic ketoximes (type a2) and ketoximes with amine group (type a3) as depicted in Figure 6. The number of structures of each type found on the search is given in Table 3.

In the absence of competitive acceptors, ketoximes have been described as having two classical kinds of H bonding arrangements, the R_2^2 (6) dimer and the C3 chain [2]. The analysis of the present data for ketoxime shows that R_2^2 (6) dimers were found in 43% of the total structures and, in spite of the small number for the sample, they seem to occur in structures without other competitive acceptors or donors: in 7 out of 8 cases for structures of type a1 (aromatic or aliphatic

Table 3. Number of oxime structures found on CSD after 2003. Dioximes are not included on the count.

	Ketoxime	Cyclic Ketoxime	-NH_2 Ketoxime	Aldoxime	O-alkyl etoxime
No of Structures	50	53	17	40	57
With competitive D/A	42	38	11	34	57
R^2_2 (6) dimers	16	24	13	5	-
R^2_2 (6) dimers in competitive D/A	9	11	7	1	-
R^3_3 (9) dimers	1	1	-	1	-
R^4_4 (12) dimers	-	1	-	-	-
C2 chains	-	-	-	1	-
C3 chains	-	-	1	4	-

ketoximes), in 13 out of 15 cases for structures of type a2, (cyclic ketoximes) and in 5 out of 6 cases for structures of type a3 (amino ketoximes).

The formation of the R_2^2 (6) dimers in structures with competitive donors or acceptors is less usual: it was found that in type a1, (aromatic or aliphatic ketoximes) they occurred in 20% of the cases in 28% of cases of structures type a2 (cyclic ketoxime) and in 64% of a3 (amino ketoximes). It is worthy to note that most of the amino ketoximes contain an intramolecular short contact between the lone pairs of the oxime oxygen atom and one of the H atoms of the $-NH_2$ group as depicted in an example shown in Figure 7 [19]. The search that was made shows that C3 chains, R_3^3 (9) trimers or R_4^4 (12) rings in ketoximes are quite rare (see diagrams in Figure 8) and they are present only on structures without other donors or acceptors. In fact just one structure with an R_3^3 (9) ring was found for aromatic or aliphatic ketoximes (type a1**)**, one structure with an R_4^4 (12) ring was found for cyclic ketoximes (type a.2) and a C3 chain was found, in acetamidoxime, [19], the simplest of all amino ketoximes (type a3).

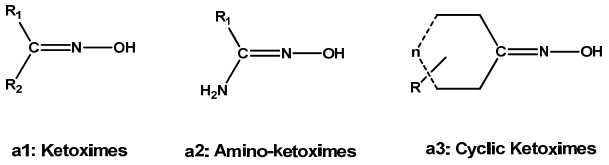


Figure 6. Types of ketoximes found on CSD search after 2003. On the search 120 ketoximes were selected. Of those 95 had a competitive hydrogen donor (-OH or –NH) on R_1 or R_2 and/or a competitive hydrogen acceptor (=O: or –N:) on R_1 or R_2.

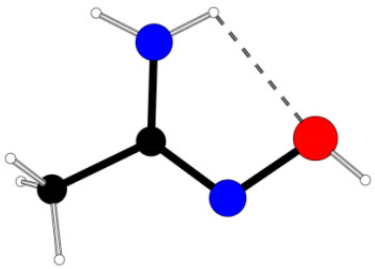


Figure 7. Example of an S(5) intramolecular short contact in ketoximes with amine group.

3.2.2. Aldoximes and O-alkyl-ketoximes

Following the same criteria used for ketoximes, the examined aldoximes (b moiety in Figure 2) were grouped in two different sets, one of those without other competitive acceptors or donors and those containing the competitive hydrogen donors or acceptors (see Table 3). Only 40 entries for the aldoxime moiety were found in the search and 6 of them, mostly aromatic (refer to Figure 9), were without competitive donors or acceptors. Structures exhibiting R_2^2 (6) dimers are less common than those found in ketoximes, there are 5 of them in total. R_3^3 (9) trimers, one occurrence, C3 chains, four occurrences, are more common than in the ketoximes and there is one occurrence of a C(2) oxime-oxime O···O chain. Nevertheless, it is not possible to derive any conclusion from these few data that are available. Dimers within the oxime functional group are not possible in O-alkyl-ketoximes (c moiety in Figure 2) and in most of their structures the N of the oxime moiety is not involved in the H···bond pattern which defines their supramolecular structure.

3.2.3. Less common oxime-oxime interactions-rings and chains

Cyclic R_4^4 (12), R_3^3 (9) clusters and C3 chains induce much less strain in the molecular geometry, particularly in angular values than do the R_2^2 (6) dimers which, in addition, do not profit from the cooperative effects of the multiple H interactions as in the case of the R_4^4 (12), R_3^3 (9) rings.

An indication of the strain within the molecular geometry would ideally be given by the angle O-H···N. The angular analysis of the structures showed that 71% of the angles at the H atom in R_2^2(6) dimers lie between 140° and 160° with a mean value of 149°. The angle at the H atom in C3 chains, (five structures involving 6 individual molecules) lie in the range of 156° to 178° with an mean value of 170°; in R_3^3 (9) trimers, (three structures involving 12 individual molecules) the angles lie in the range of 162° to 179° with a mean angle of 171° and the angle in a single R_4^4 (12) structure the angle at the H atom is 178°, (the R_4^4 (12) ring is centred on the 4_1 axis in space group $I4_1/a$). For the C2 chain the O-H...O angle is 158°. However these values should be envisaged as indicative ones, because the H atoms on the molecules in this study are either constrained, refined or in positions obtained from difference maps (some are in positions which are essentially wrong). Nevertheless, the figures indicate that, in general, the angle in the R_4^4 (12), R_3^3 (9) rings and C3 chains is significantly higher in general than in the R_2^2 (6) dimers. This feature does not seem enough to prevent the formation of the R_2^2 (6) dimeric structure indicating the importance of other interactions besides classical H bonding for the stabilization of the solid state. In fact, as in 1,3-diphenylpropan-2-one oxime, most of the dimeric structures contain (when possible) weak CH···π interactions or CH–X interactions (X=halogen). In the majority of these compounds with the less common oxime-oxime interactions there are no weak C-H···π or π....π interactions present although in the structures of (E)-3-pyrazolyl carbaldoxime and the related *tris*((E)-3-pyrazolyl carbaldoxime) chloroform clathrate, [24]. There are donors and acceptors in the pyrazole ring which interact with each other and which do not prevent formation of a C3 chain or R_3^3 (9) rings, respectively. In (Z)-5-chlorofuran-2- carbaldehyde oxime [25] the competitive acceptors are not involved in the hydrogen bonding and the molecules are linked by C3 chains.

It is worth noting that there is evidence in solution (from infra-red studies) and in gaseous phase (from theoretical calculations) that the hydrogen bond topology also favours the formation of cyclic aggregates probably due to the absence of the secondary weak interactions that may stabilise the dimeric form in the solid state [26].

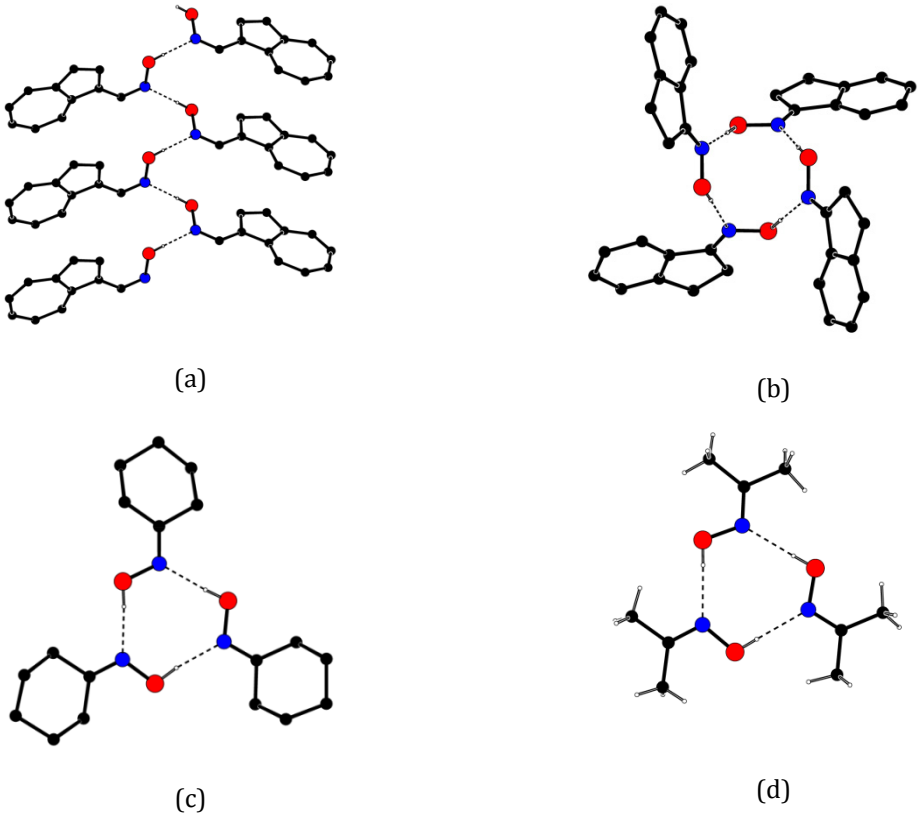


Figure 8. Oximes exhibiting unusual H-bond patterns in solid state: a) C(3) catemer, b) R_4^4 (12) rings and c and d) R_3^3 (9) trimers. (a) (Z)-Azulene-1-carboxaldehyde oxime Kedziorek *et al.* [20], (b) indan-1-one oxime, Clive *et al.* [21], (c) (E)-Cyclohexanone oxime, Lutz *et al.* [22] and (d) Acetoxime, Parsons *et al.* [23]. Hydrogen atoms not involved in the hydrogen bonding motifs are not in all figures except for (d).

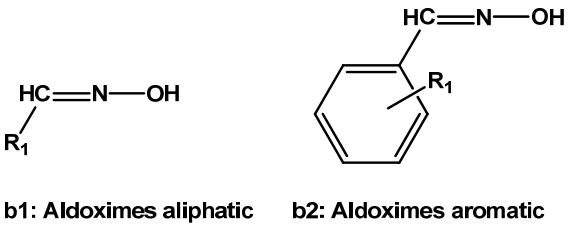


Figure 9. Types of aldoximes found on CSD search after 2003. On the search 40 aldoximes were found. Of those 34 had a competitive hydrogen donor (-OH or –NH) on R_1 or R_2 and/or a competitive hydrogen acceptor (=O: or –N:) on R_1 or R_2.

3.2.4. Oximes with competitive donors or acceptors

When a competitive acceptor is present in the molecule it was found that the supramolecular structure was in most cases determined by the O–H (oxime)···A hydrogen bond or by D–H···N(oxime) (A=acceptor; D=donor). In fact, among the ketoximes or aldoximes, which do not form the rings or chains discussed above, the "A acceptors" for the O–H (oxime moiety) are mostly nitrogen atoms of heterocyclic rings (28 structures) or carboxylic C=O (12 structures) and few –O– of aromatic or ethers (8 structures). Similarly, the majority of competitive donors for :N– (oxime) are aromatic or aliphatic hydroxyl (–OH) groups (16 structures) and –NH from secondary amines (9 structures). Since previous studies suggest that strong H donors tend to combine with strong H acceptors [1], it appears that the –OH moiety of the oxime behaves as a strong donor while the :N– acceptor as a weak acceptor.

Supplementary material

All crystallographic data for this paper are deposited in Cambridge Crystallographic Data Centre (CCDC 736962). The data can be obtained free of charge at www.ccdc.cam.ac.uk or from Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax +44 (0) 1223 336033, e-mail: deposit@ccdc.cam.ac.uk.

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