Short communication

Synthesis, crystal structure, IR-spectral data and some properties of 3,5-diamino-1,2,4-triazolium tetrafluoroborate and hexafluorosilicate

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ABSTRACT

3,5-Diamino-1,2,4-triazolium tetrafluoroborate $(LH)BF_4$ and hexafluorosilicate $(LH)_2SiF_6$ have been isolated and characterized by single-crystal X-ray structure determination, IR spectroscopy, mass spectrometry, solubility data, potentiometry. The N–H···F and N–H···N hydrogen bonds play an important role in a formation of 3-D structures of two compounds. The relationship between the salts structure and some properties is discussed.

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1. Introduction

Functionalized derivatives of 1,2,4-triazole, particularly 3,5-diamino-1,2,4-triazole (L), attract an interest and are actively studied as ligands in the synthesis of d-metals complexes [1–3], precursors of condensed N-heterocyclic systems [4,5], corrosion inhibitors [6,7], biologically active compounds with a wide range efficiency [8,9] and as high-energy compounds [10–12]. On the other hand, ionic complexes with protonated form of N-containing organic bases, including 3,5-diamino-1,2,4-triazole, and complex fluoro-anions such as SiF_6^{2-} become convenient models to study an influence of interionic N–H···F bonds on the structural characteristics and properties of these compounds [13–16]. As it has been noted [15,16], the high H-acceptor affinity of covalently bound fluorine in SiF_6^{2-} anion result in a strong H-bonding interactions in complexes $(AmH)_2SiF_6$ (Am–N–donor base) that is affected on the disproportionation of Si–F bond lengths, solubility and thermal stability of salt. In present communication we are describing some results of synthesis, X-ray structure determination and physico-chemical investigations of two new salts – 3,5-diamino-1,2,4-

triazolium tetrafluoroborate $(LH)BF_4$ and hexafluorosilicate $(LH)_2SiF_6$.

2. Results and discussion

2.1. Crystal structure

In the structure of $(LH)BF_4$ (I) five from six H atoms from each LH moiety participate in a formation of strong (1.98–2.33 Å) N–H···F hydrogen bonds. The sixth hydrogen atom, which belongs to one of the amino-groups, forms N–H···N bond with the non-protonated N4 atom from another LH unit. A pair of such N–H···N bonds connects two LH moieties, mutually oriented in "head-to-tail" manner, into a dimer (Fig. 1). The N3–H3···F4 contact, involving pyridine-type nitrogen atom, appears to be the shortest hydrogen bond in this structure (Table 1). These bonds connect each LH cation to four BF_4^- anions. The F4 atom acts as a μ_3-bridge, being bound to two hydrogen atoms from two different LH units, each of other fluorine atoms are connected to one H-atom. In turn, each anion is bound to 4LH⁺ units.

Such bridging role of both anions and cations, and the planar geometry of LH resulting in a formation of layered three-dimensional structure (Fig. 2). Three fluorine atoms from each BF_4^- – anion are bound with three LH units belonging to one layer, and the forth F atom is connected to the LH cation from the next layer. Because of small size of BF_4^- moieties the distance of 3.2 Å

Fig. 1. Coordination of LH+ moiety in structure of I.
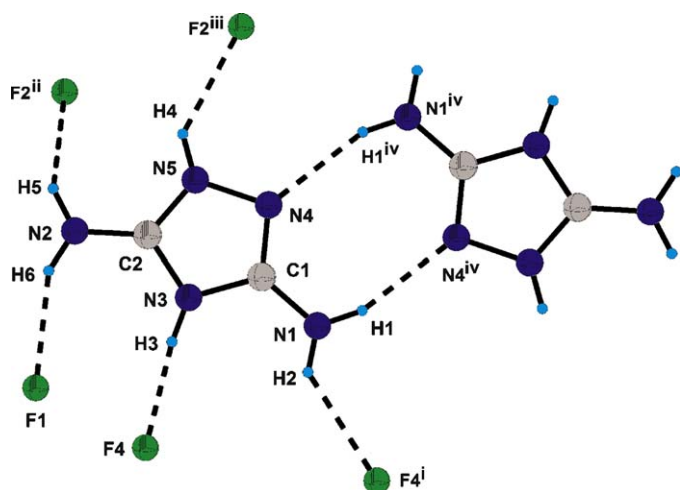

Fig. 2. Layered structure of I.

between the closest LH units from two neighboring layers appears to be rather short.

In the structure of $(LH)_2SiF_6$ (II) the organic cation acts similar as in I role. Five hydrogen atoms are bonded to four SiF_6^{2-} anions (Fig. 3). Because of slightly higher than that in BF_4^- fluorine atom charge the distances H···F appear to be moderately shorter (1.90–2.11 Å) in comparison with $(LH)BF_4$. Possibly because of more "spherical" shape of SiF_6^{2-} anions and, therefore, higher spatial accessibility of fluorine atoms, all N–H···F bond lengths in II lie in a narrow range (Table 1). Similarly to that in I, the H-atom connected to the pyridine-type nitrogen forms the shortest N–H···F bond. Two F atoms act as μ_3-bridges, other four as μ_2-bridges, resulting in a connection of each SiF_6^{2-} anion to 8 organic moieties (Fig. 4). Inorganic anions and organic cations are interconnected into slightly puckered double layers, whereas further association of these layers occurs by non-valence interactions. Contrary to I, in the structure II the N–H···N bond involves removed from the N4 atom amino-group. It leads to an additional chain-like bonding of LH units within each layer.

It is interesting to compare the geometry of neutral and protonated forms of guanazole. In the structure of molecular 3,5-diamino-1,2,4-triazole one may observe a noticeable asymmetry of C–NH$_2$ bonds. It can be attributed to the difference in the surrounding of both carbon atoms. The C1 atom is connected to two–N–groups, and C2 one is bound to –N–and–NH–groups. In the
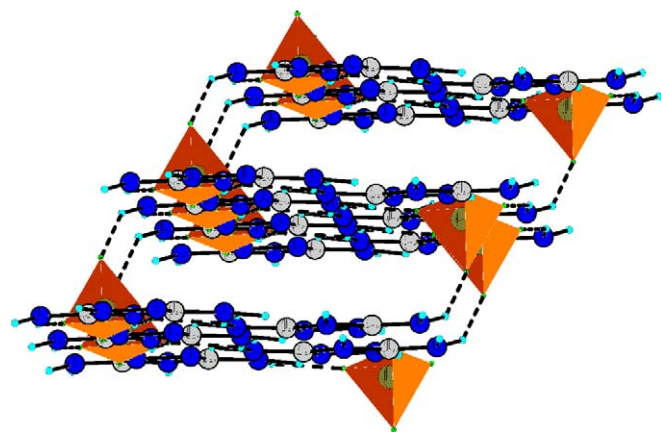
structure of molecular guanazole the C1–NH$_2$ distance is 1.353 Å, and the C2–NH$_2$ one is 1.376 Å [17]. Such a tendency appears also in LH units: C1–NH$_2$ bond lengths are 1.341(4) Å in BF_4^- and 1.352(3) Å in SiF_6^{2-} derivatives respectively, and C2–NH$_2$ distances are 1.320 Å in both guanazolium salts. Similar values of 1.341(9) and 1.320(9) Å were found recently in the structure of guanazolium picrate [18]. It was also observed, that strictly identical in the structure of neutral guanazole N3–C1 and N3–C2 bond lengths of 1.363 Å become markedly different in its protonated form. Respective values appear to be 1.372(5) and 1.352(4) Å for tetrafluoroborate, 1.384(4) and 1.352(3) in hexafluorosilicate, 1.372(8) and 1.343(1) in picrate respectively. Also the protonation leads to a small deformation of the whole triazole cycle, with increasing the C–N–C angle from 102.7° in the neutral molecule to 107–108° in cationic form, and decreasing of both N–C1–N angle from 110° to 106°, and N–C2–N one from 116° to 111–112°.

2.2. Results of IR spectroscopy

In the IR spectrum of the ligand (Table 2) vibrations of amino- and the NH-groups of the triazole cycle $\nu_{as,s}(NH_2)$ and $\nu(NH)$ appeared as a row of absorption bands with maxima at 3400, 3365, 3310, 3238, 3119 cm^{-1}. In the spectra of salts these bands were found at 3395, 3330, 3142 cm^{-1} (I) and the 3350, 3162 cm^{-1} (II). The observed transformation of the spectra appears because of protonation of one of the N–pyridine-type nitrogen atoms and the

Table 1
Parameters of hydrogen bonds in compounds I (top) and II (bottom).

Atoms	D–H	H···A	D···A	<(DHA)
N1–H2···F4^i	0.86	2.33	3.015(4)	136.3
N3–H3···F4	0.86	1.98	2.836(3)	173.3
N2–H6···F1	0.86	2.13	2.927(3)	153.1
N2–H5vF3^{ii}	0.86	2.29	2.922(4)	130.7
N5–H4···F2^{iii}	0.86	2.24	2.925(3)	136.0
N1–H1···N4^{iv}	0.86	2.16	2.983(5)	160.9
Atoms	D–H	H···A	D···A	<(DHA)
N1–H2···F3^i	0.86	2.11	2.918(4)	156.9
N1–H1···F1^{ii}	0.86	2.02	2.849(5)	160.2
N3–H3···F1^{iii}	0.86	1.90	2.703(4)	154.6
N2–H5···F3^{iv}	0.86	2.00	2.833(5)	162.9
N5–H4···F2^v	0.86	2.01	2.751(4)	143.1
N2–H6···N4^{vi}	0.86	2.17	3.019(5)	167.8

Symmetry transformations: (i) $3-x$, $2-y$, $-z$ (ii) x, $y-1$, z (iii) $x-1$, $y-1$, z (iv) $2-x$, $1-y$, $-z$.
Symmetry transformations: (i) x, y, $z-1$, (ii) $x-1$, y, $z-1$, (iii) x, y, $z-1$, (iv) $x+1$, $y+1$, z, (v) x, $y+1$, z, (vi) $x+1$, y, z, (vii) $x-1$, y, z.
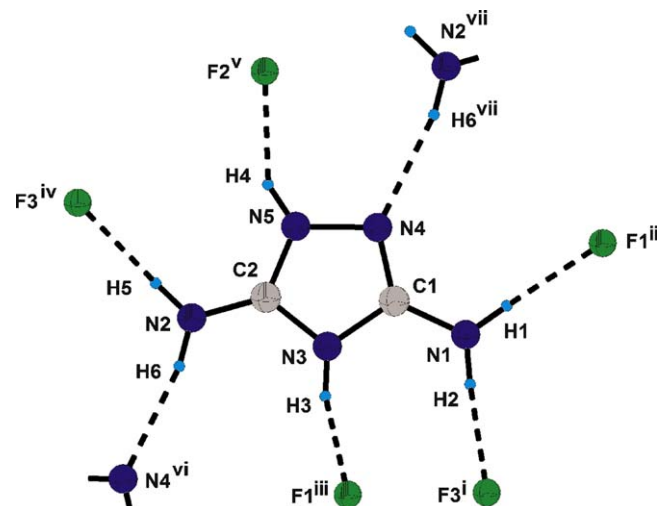

Fig. 3. Coordination of LH+ moiety in structure of II.

Table 2
IR spectral data (cm^{-1}) for 3,5-diamino-1,2,4-triazole and complexes I and II.

	[C$_2$H$_6$N$_5$][BF$_4$]I	Assignment	[C$_2$H$_6$N$_5$]$_2$[SiF$_6$]II
3400 s.	3395 sh.	$\nu_{as,s}$(NH$_2$), ν(NH), ν(N$^+$H)	
3365 sh.			3350 s.br.
3310 s.	3330 s.br.		
3238 m.			
3119 s.	3142 sh.		3162 m.br.
1626 s.	1693 s.	δ(NH$_2$)	1695 s.
	1656 s.		1660 s.
1585 m.	1600 sh.	ν_{ring}, δ(HNC), δ(NH$_2$)	1595 sh.
1563 s.	1531 s.		1536 m.
1490 s.			
1417 s.	1411 m.		1420 w.
1350 m.	1344 m.	ν(NN), δ(CNN), r(NH$_2$)	1347 m.
	1256 m.		1245 w.
	1225 m.		
	1170 m.	ω(NH$_2$), δ(CNN), δ(HCN)	1165 sh.
1150 m.			1140 w.
1123 m.			
1060 s.	1057 m.	ν(CN), r(NH$_2$), δ($\geqslant^+$NH)	1065 m.
			1050 m.
1016 m.	1020 sh.	$\nu_{puls.}$, ν(BF)	1025 m.
	1013 s.		1011 m.
	960 sh.	δ(NCN), δ(CNN), δ($\geqslant^+$NH), ρ(NH$_2$),	
	840 m.		
807 m.	790 m.		810 sh.
740 sh.	740 sh.	ν(SiF), r(NH$_2$), δ(CCH)	741 v.s.br.
727 m.			
670 sh.	670 sh.		
650 m.br.	657 sh.		645 sh.
615 sh.	621 w.		
540 m.	530 w.	δ(NCN), δ(CNN), δ(BF$_2$)	545 w.
505 sh.			
		δ(SiF$_2$)	482 m.
			420 sh.

Note: w. = weak, m. = medium, s. = strong, v. = very, sh. = shoulder, br. = broad.

formation of system N–H···F bonds instead of N–H···N ones realized in pure ligand [19].

As seen from Table 2 salts formation accompanied by a change in the nature of absorption of complex valence-deformation vibrations of the ligand at 1600–1400 cm^{-1} with contributions of ν_{ring}, δ(NNC), and, probably, δ(NH$_2$). Thus, in the spectrum of II instead of the bands at 1585 and 1563 cm^{-1}, found in azoles spectrum, there are bands at 1595 and 1536 cm^{-1}. In the spectrum of I recorded these bands appear at 1600 and 1531 cm^{-1}, respectively. High-frequency shift of the azole's band at 1585 cm^{-1} may be due to protonation of pyridine nitrogen atom with a maximum value of the negative π-electron charge [20].

Identification bands vibrations ν(SiF) in the spectrum of II at 750–710 cm^{-1} is difficult because of the presence in this region of skeletal vibrations of the azole cycle and bending vibrations of amino groups. Intense absorption at 741 cm^{-1} in the spectrum of II is due to superposition of vibrations ν(SiF) and deformation vibrations of the cation. In turn, a sharp increase the intensity of

the band pulsation vibrations of the ring ν_{puls} about 1013 cm^{-1} in the spectrum of I in comparison with the spectrum of initial triazole relates to the superposition of mentioned vibration with the deformation vibrations ν(BF) tetraftoroborate anion [21]. Deformation vibrations δ(BF$_2$) and δ(SiF$_2$) in the spectra of I and II represented by a low-intensity band at 530 cm^{-1} and doublet band of medium intensity at 482, 420 cm^{-1}, respectively (Fig. 5).

2.3. Some physico-chemical properties

Some physico-chemical properties of I and II compounds are listed in Table 3. Obtained pH values of water solutions I and II show on their hydrolitic unstability, typical for tetrafluoroborates and hexafluorosilicates [22,23]. The pH value of solution II is close to the similar values for pyridinium hexafluorosilicates [15,16].

As was shown on the examples of the piridinium hexafluorosilicates with functionalized cations [15,16], values of solubility explored salts one may depend on the h parameter:
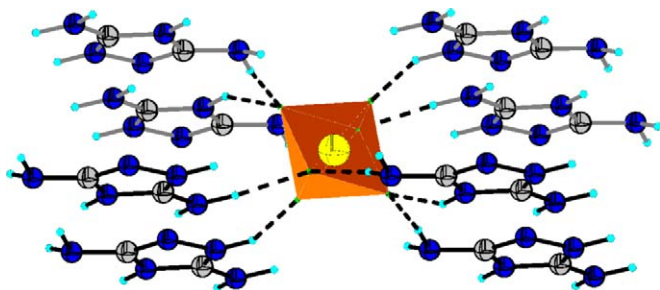

Fig. 4. Bridging role of SiF$_6^{2-}$ anions in II.
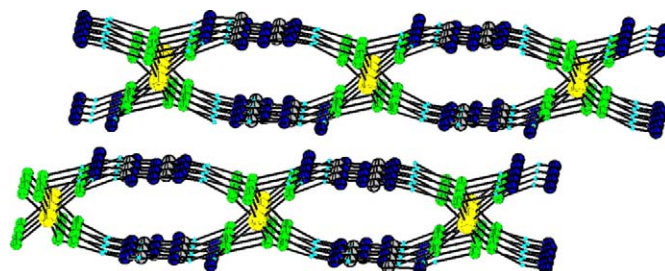

Fig. 5. Layered structure of II.

Table 3
Temperatures of thermolysis beginning, melting points, solubility of I and II in water and ₚH of salt solutions.

Compound	t_0 (°C)	m.p. (°C)	C (mol. %), 25 °C	pH 0.001 M solutions, 25 °C
(LH)BF$_4$ (I)	110	170–172	0.63	3.64
(LH)$_2$SiF$_6$ (II)	146	248–250 (decomp.)	0.28	3.33

Table 4
Solubility of pyridinium hexafluorosilicates and II in water and h values.

Compound	C (mol. %), 25 °C	h (Å^{-1})
[2-CH$_3$C$_5$H$_4$NH]$_2$[SiF$_6$]	11.60	0.71
[2-HO(O)CC$_5$H$_4$NH]$_2$[SiF$_6$]	5.33	0.76
[3-HO(O)CC$_5$H$_4$NH]$_2$[SiF$_6$]	3.33	1.09
[4-HO(O)CC$_5$H$_4$NH]$_2$[SiF$_6$]	0.80	1.08
[2-H$_2$NC$_5$H$_4$NH]$_2$[SiF$_6$]	5.60	1.38
[2,6-(H$_2$N)$_2$C$_5$H$_3$NH]$_2$[SiF$_6$]	0.06	2.35
[4-H$_3$NHN(O)CC$_5$H$_4$NH][SiF$_6$]	0.87	1.80
[C$_2$H$_6$N$_5$]$_2$[SiF$_6$]	0.28	1.78

Table 5
Crystal data and structure refinement for compounds I and II.

	Formula	
	C$_2$N$_5$H$_6$BF$_4$	(C$_2$N$_5$H$_6$)$_2$SiF$_6$
fw	186.93	342.33
temp (K)	200	200
space group	P$\bar{1}$	P$\bar{1}$
a (E)	5.2029(6)	6.1567(11)
b (Å)	7.4504(5)	7.4881(16)
c (Å)	9.2854(6)	7.5529(12)
α (°)	74.419(11)	99.651(4)
β (°)	85.386(17)	107.189(2)
γ (°)	83.738(18)	109.324(6)
V (Å^3)	344.15(5)	300.00(10)
Z	2	1
D_{calcd} (g/cm^3)	1.804	1.895
λ (pm)	0.71069	0.71069
μ (mm^{-1})	0.196	0.29
GOF indicator	1.151	1.108
$R1$;	0.0856[a]	0.0569
w$R2$ ($I > 2.00\sigma(I)$)	0.2418	0.1507
Max. & Min. Residue of electronic dencity	0.446, −0.365	0.401, −0.53

$R1 = \sum||F_o|-|F_c||/\sum|F_o|$,,, where N_o = no. of reflns and N_p = no. of refined params.
[a] Possibly rather high $R1$ value for I caused some water contamination due to high hydrophilicity of BF$_4^-$ anion.

$$h = \frac{n}{d(D \cdots A)_{av}}$$

n – amount of short interionic contacts (H-bonds), $d(D \cdots A)_{av}$ – average donor–acceptor distance in the structure of complex. Calculated from the structural data h values for II, correspondent values for piridinium salts and solubility data are placed in Table 4. One may note, that the h and C values for II agree with anti-exponential dependance (Fig. 4 in [16]).

3. Conclusions

One may conclude that in ionic structures of I and II 3,5-diamino-1,2,4-triazole derivatives the pyridine type nitrogen atom of heterocyclic core appears to be protonated. The N–H⋯F bonds, formed in I and II compounds perform structure-directing and stabilizing function, forming a supramolecular 3D-structure. Involvement of fluoro-ligands in the H-bonding is accompanied by disproportionating of the bond lengths M–F (M = B, Si), and in the case of complex II inverted correlation between the Si–F

(1.675(2)–1.696(2) Å) distances and corresponding N(H)⋯F bond lengths (N⋯F 2.705(4)–2.915(4) Å) has been observed. The presence of such a correlation in structures of "onium" hexa-fluorosilicates has been noted in the literature [13–16]. One of the appearances of the H-bonds stabilizing effect is disclosed by authors the relationship between the number of short interionic H-bonds in structure II (and related pyridinium hexafluorosilicates) and solubility of these salts.

One may conclude, that considering different charges of BF$_4^-$ and SiF$_6^{2-}$ anions, it is impossible to make a strict comparative analysis of the structure–properties relationship of I and II salts. However, taking into account the above-mentioned similarities in the H-bonds systems in I and II, observed differences in solubility and thermal stability of salts can, in some approximation, reflect decreasing in the stabilizing contribution of inter-ionic N–H⋯F bonds due to the lower H-acceptor ability of the BF$_4^-$ anion in comparison with SiF$_6^{2-}$ one [24].

4. Experimental

4.1. Materials

HBF$_4$ (40%, Aldrich), H$_2$SiF$_6$ (48%, Aldrich), 3,5-diamino-1,2,4-triazole (98%), Aldrich were used without further purification.

4.2. Instrumentation

The IR-absorption spectra were recorded on a spectrophotom-eter Specord 75IR (ambient temperature, range 4000–400 cm^{-1}, samples as suspension in Nujol mulls between KRS-5 windows). The mass spectra were registered on a spectrometer MX-1321 (direct input of a sample in a source, energy of ionizing electrons 70 eV). The isothermal conditions of experiments on detection of a solubility and hydrolysis of hexafluorosilicate and tetrafluorobo-rate salts ($t = 25 \pm 0.2$ °C) were provided with the help of an ultra thermostat U15. The thermogravimetric experiments were carried out on a OD-102 derivatograph of a system F. Paulik, J. Paulik and L. Erdey in an ambient conditions (Al$_2$O$_3$ internal standard, heating rate of a sample 5 °C/min).

4.3. Synthesis of 3,5-diamino-1,2,4-triazolium tetrafluoroborate (I)

The compound with the composition (LH)BF$_4$ (I) was obtained in an approximately qualitative yield by the interaction of L in methanol solution with HBF$_4$ (40%) in mole ratio 1:3. Colorless transparent crystals of I with m.p. 170–172 °C. Anal. found, %: B 5.93, N 37.14, F 39.25. Calcd. for I, B 5.78, N 37.47, F 40.66. Mass spectrum: [ML]$^+$ ($m/z = 99$, $I = 100$%), [ML–2H$_2$CN]$^+$ ($m/z = 43$, $I = 88$%).

4.4. Synthesis of 3,5-diamino-1,2,4-triazolium hexafluorosilicate,(II)

The compound with the composition (LH)$_2$SiF$_6$(II) was obtained in an approximately qualitative yield by the interaction of L in methanol solution with H$_2$SiF$_6$ (48%) in mole ratio 1:3. Colorless transparent crystals of II with m.p. 248–250 °C (with decomposi-tion). Anal. found, %: Si 8.33, N 40.68, F 35.17. Calcd. for II, Si 8.21, N 40.92, F 33.30. Mass spectrum: [ML]$^+$ ($m/z = 99$, $I = 100$%), [ML–2H$_2$CN]$^+$ ($m/z = 43$, $I = 45$%), SiF$_3^+$ ($m/z = 85$, $I = 24$%).

4.5. X-ray structural determination

Data were collected on a Rigaku AFC7 diffractometer equipped with a Mercury CCD area detector using graphite-monochromatized Mo-K$_\alpha$ radiation at 200 K. The data were corrected for Lorentz and polarization effects. A multiscan absorption correction was applied to all data sets. Structure was solved by direct methods using SIR-92 [25] program implemented in program package TeXsan [26] and refined with SHELX-97 [27] software (program packages TeXsan and WinGX) [28]. The figures were prepared using DIAMOND 3.1 software [29]. Some details of the data collection, data processing, and refinement are given in Table 5.

Acknowledgements

Appendix A

Crystallographic data (excluding structure factors) for the structure in this paper have been deposited with the Cambridge Crystallographic Data Centre as the supplementary publication no. CCDC 771983 & CCDC 771981 for I and II, respectively. These data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge, CB12 1EZ, UK, fax: +44 1223 366 033, e mail: deposit@ccdc.cam.ac.uk or on the web www: http://www.ccdc. cam.ac.uk.

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