Synthese von Organischen und Anorganischen Derivaten der Imidazol-2-ylidene und 2-Methylenimidazoline

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Synthesis of Organic and Inorganic Derivatives of Imidazol-2-ylidenes and 2-Methyleneimidazolines

DISSERTATION

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2. Berichterstatter:	Prof. Dr. H. A. Mayer

To my

Mother and family

Die vorliegende Arbeit wurde am
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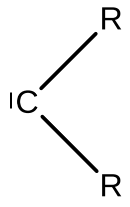
1 Introduction

1.1 Carbenes

1.1.1 Definition

Carbenes can be defined as divalent carbon intermediates, where the carbene carbon atom is linked with two adjacent groups by covalent bonds, and possesses two nonbonding electrons **1**. These electrons have either anti-parallel spins (singlet state) **1a** with bond angle 100-110º and cannot be observed by ESR, or parallel spins (triplet state) **1b** with bond angle 130-150º and can be observed by ESR [1]. Many carbenes, like CH_2 can be found in either state, though one may be more common.



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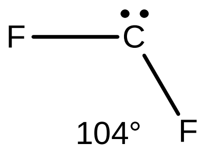
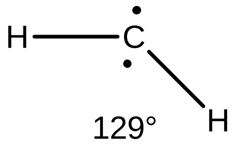


1a



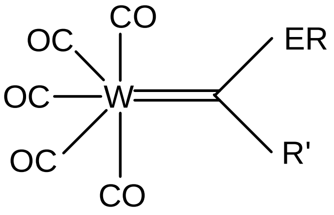
1b

1.1.2 Preparation of carbenes.

The first attempts to prepare carbenes were made by Dumas and Regnault when they tried to synthesize methylene by dehydrating methanol using phosphorus pentaoxide or concentrated sulfuric acid [2]. Later on, Butlerov obtained ethylene from the reaction of methyl iodide with copper and made the plausible suggestion that it arose by the dimerization of methylene [1]. Geuther proposed that the basic hydrolysis of chloroform involved the formation of intermediate dichloromethylene, which has now well established [1].

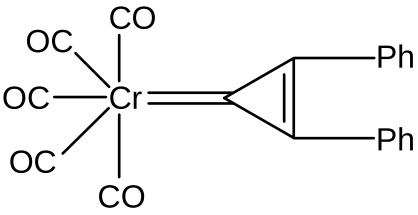
A second period of carbene research was initiated by the discovery of isonitriles and fluminic acid derivatives. Modern work in the field of methylenes began around 1910 with the investigations of Staudinger on the decomposition of diazo compounds and ketenes [2].

Fischer *et al*. initiated the organometallic chemistry of carbenes with the synthesis of stable carbene complexes **2** in 1964, while in 1968 the carbene complex **3** was reported [3]. The isolation of a carbene complex containing the parent methylene group :CH_2 was achieved by Schrock in 1975 [4].



2

E = O, S
R = Me
R' = H



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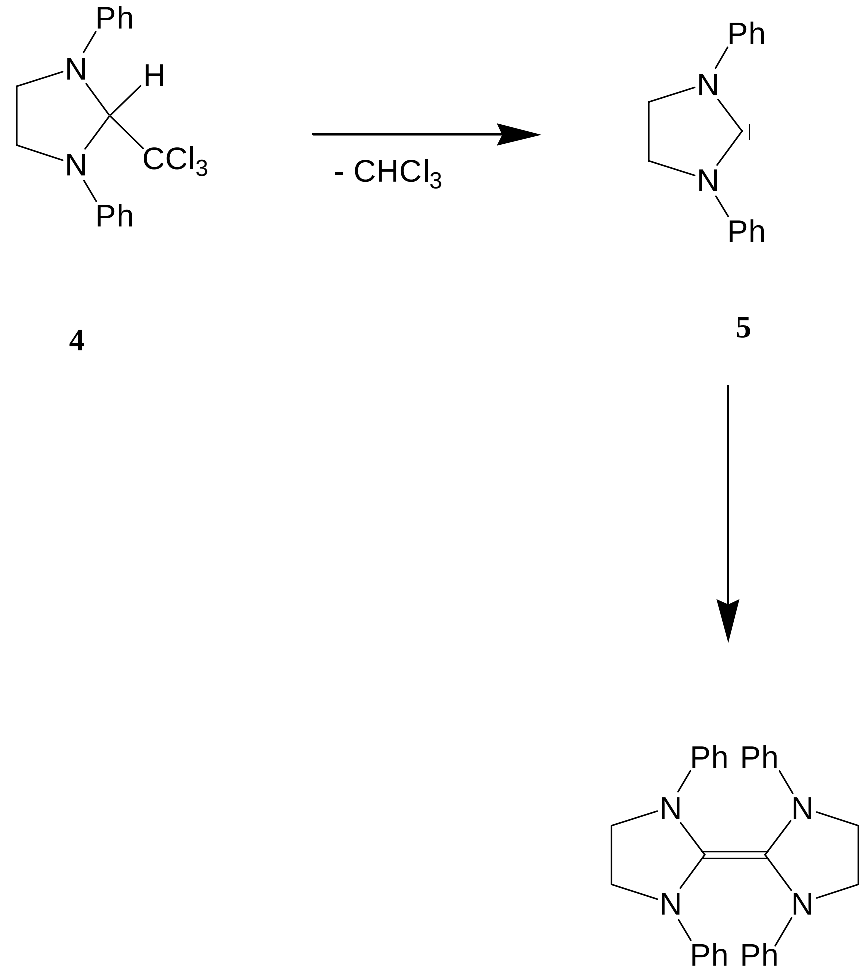
1.1.3 Diaminocarbenes.

In the early 1960s, Wanzlick realized that the stability of carbenes could be enhanced by the presence of amino substituents. Moreover, he tried to prepare the 1,3-diphenylimidazolidin-2-

ylidene **5** from imidazolidine **4** by thermal elimination of chloroform [5]. At that time the dimeric electron-rich olefin **6** was isolated.

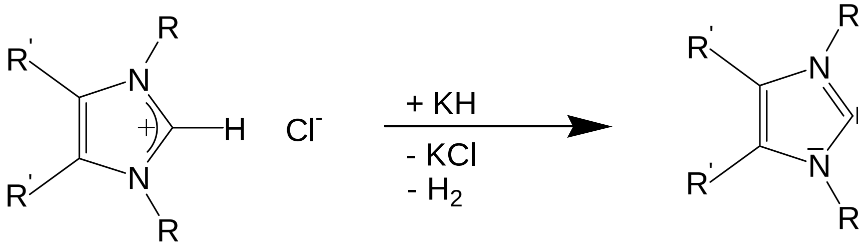


4

5

6

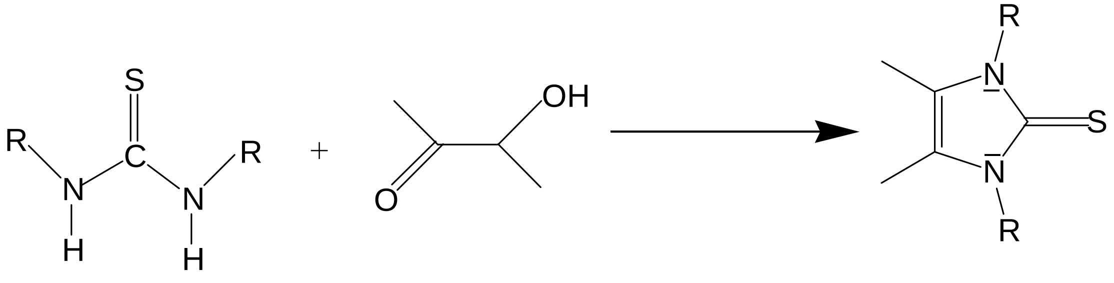
In 1970, Wanzlick and co-workers demonstrated that the imidazolium salt **7** could be deprotonated by potassium *tert*-butoxide to afford the corresponding imidazol-2-ylidenes **8** which were trapped, but not isolated [6]. Following this principle, almost two decades later Arduengo *et al*. prepared a stable crystalline carbene compound **8c,** which was obtained by deprotonation of the 1,3-di-1-admantylimidazolium chloride **7c** with sodium or potassium hydride in the presence of catalytic amount of either *tert*-butoxide or the dimethylsulfoxide anion [7-9].



7a-c **8a-c**

a : R = Ph, R' = H
b : R = R' = Ph
c : R = Ad, R' = H

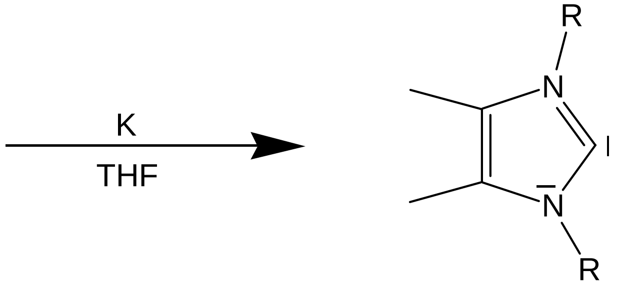
In 1993 Kuhn and co-workers developed a new and versatile approach to the alkyl-substituted *N*-heterocyclic carbenes **10a-c** in very good yield. This original synthetic strategy relied on the reduction of imidazolin-2-thions **9a-c** with potassium in boiling THF [10].



a: R = Me
b: R = Et
c: R = i-Pr

9a-c

9a-c

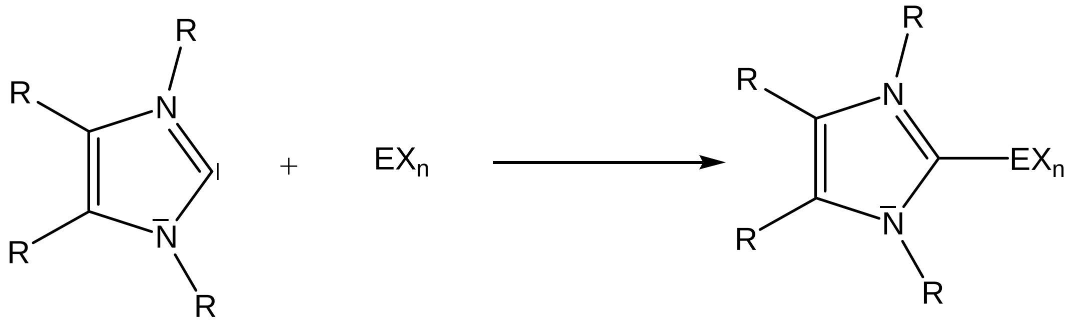


10a-c

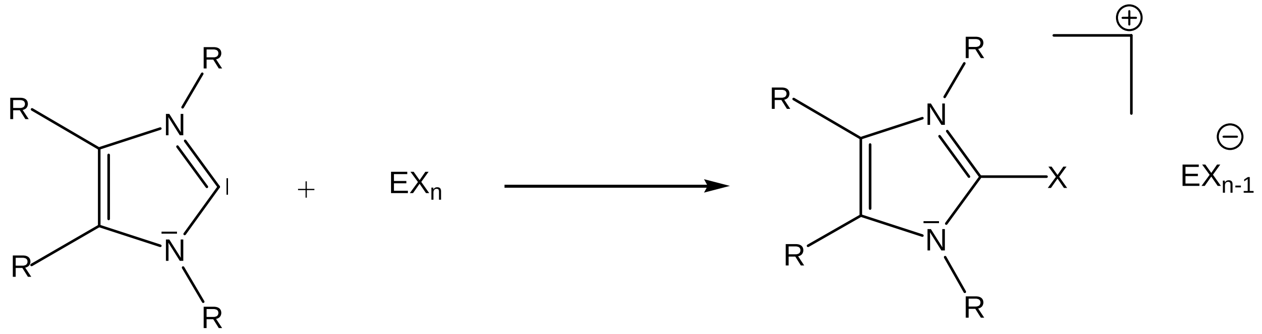
There are several reasons for the stability of *N*-heterocyclic carbenes**.** Firstly, the high electronegativity of nitrogen atom stabilizes the lone pair on the carbon center between them in the plane of the ring through an inductive σ-effect. Secondly, the unoccupied carbon p orbital can be stabilized by mesomeric interaction with the nitrogen lone pairs. Bulky substituents bound to the nitrogen centers have conclusively been shown to play a vital role in the stability of isolatable carbenes [11].

1.1.4 The nucleophilicity of 2,3-dihydroimidazol-2-ylidenes.

Imidazol-2-ylidenes **10** have found unusual attention in the inorganic chemistry as strong nucleophiles. They react with polar compounds of the main group elements. It has been reported that they can coordinate through the central element to give compound **11** (EX_n = BH_3 [12], AlH_3 [13], $SiCl_4$ [14], $SOCl_2$ [15], SeI_2 [16]), or can abstract a halogen atom to give compound **12** (EX_n = SF_4, SO_2ClF, SO_2F_2 [15], SO_2Cl_2 [17]).

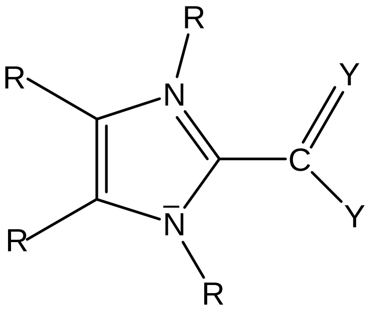


10 **11**

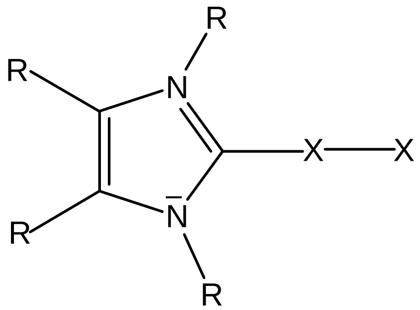


10 **12**

The nucleophilic attack of compound **10** at the molecules Y═C═Y leads to the formation of stable betaines **13a-b**, (Y = O [18], S [19]), and with halogen containing compounds leads, to the formation of charge transfer adducts **14** ($X_2 = Cl_2$ [20-21], I_2 [22]).



13a-b



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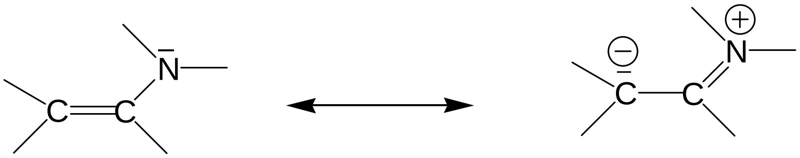
1.2 2- Methyleneimidazolines.

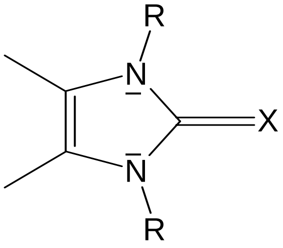
1.2.1 Preparation of 2-methyleneimidazolines.

The term “enamine” was first introduced by Wittig and Blumenthal [23] as the nitrogen analog of the term “enol”. The electronic structure of an enamine can be simply represented by the Lewis structure **15a** with contributing of resonance form **15b** [24].



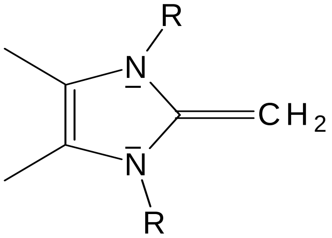
15a **15b**

2-Chalcogenimidazolines **16a-b** (X = Se [25], Te [26]) may be regarded as electroneutral selonates or tellurates from their coordination chemistry [27], as well as from their reaction with electrophilic main group centers [28-29]. Replacement of the chalcogen atoms with the methylene fragment leads to compound **17**, in which the exocyclic methylene fragment attached at C-2 should bear a negative formal charge. 2-Methyleneimidazolines act as electroneutral carbanions R_3C^-.



16a-b

a : X = Se
b : X = Te
R = iPr

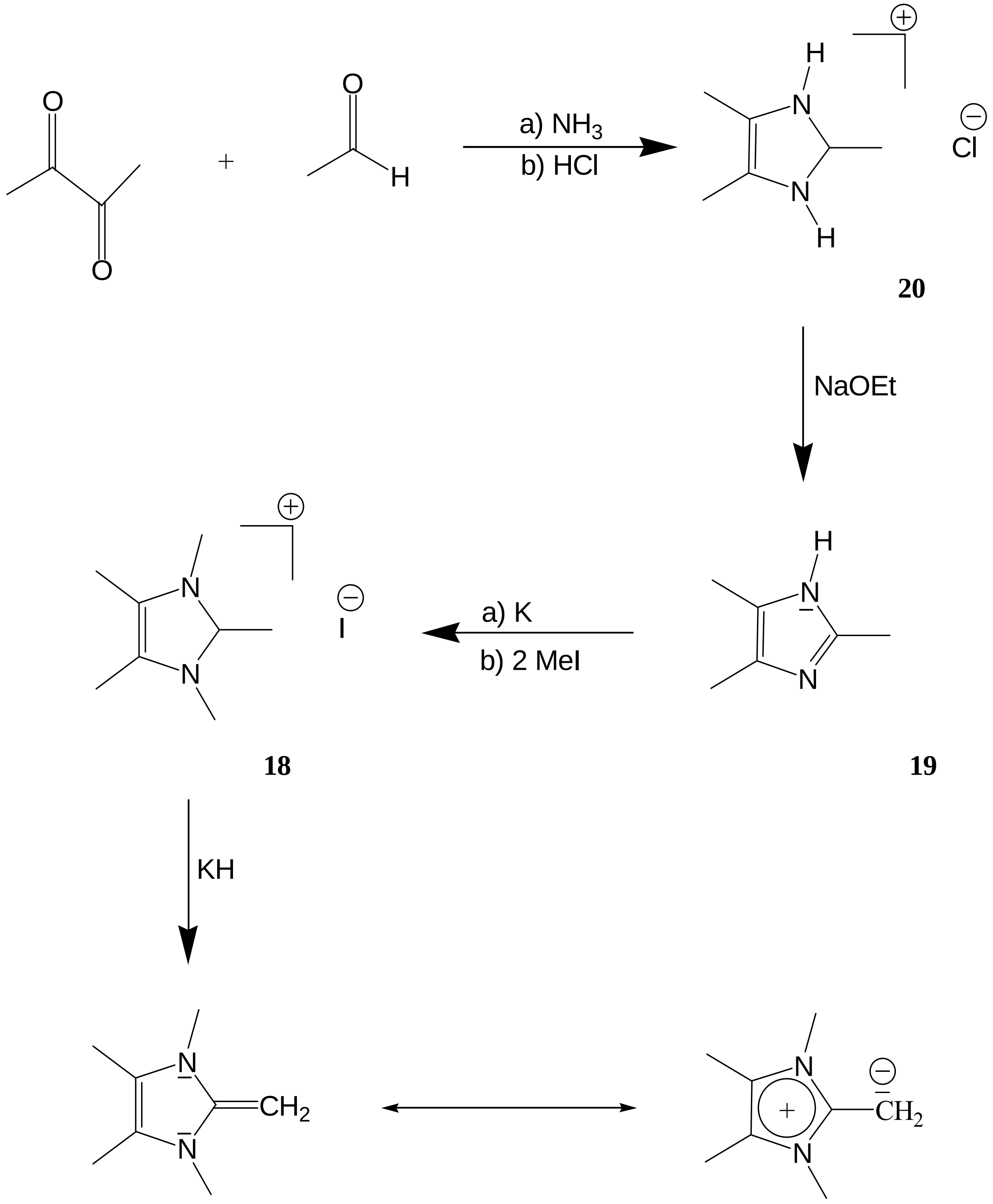


17

R = Me

The C═C bond is strongly polarized by the mesomeric electron donation of the amino groups. This effect is demonstrated by the chemistry of the cyclic endiamine [30]. The process of π-electron transfer to the exocyclic methylene carbon atom can be enhanced by the incorporation of the nitrogen atoms into heterocyclic imidazole ring system.

The synthesis of the 2-methyleneimidazolines **17** is achieved by the deprotonation of the imidazolium cation **18** by using of potassium hydride [31]. They are obtained as air sensitive crystals in good yield. The compound **18** is obtained from the reaction of **19** with potassium and methyl iodide, while compound **19** is obtained from the reaction of **20** with sodium ethoxide in ethanol.



20

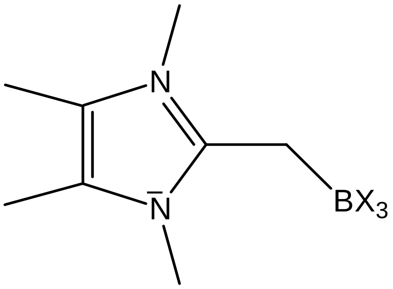
19

18

17

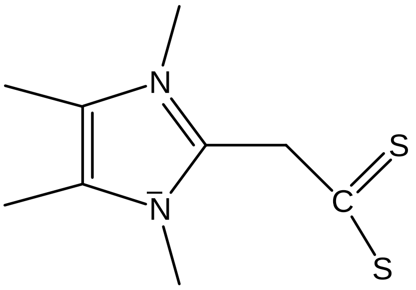
1.2.2 Reactions of 2-methyleneimidazolines.

2-Methyleneimisdazloine **17** reacts with a wide variety of main group electrophiles to give element-alkyl compounds. With boranes BX_3 (X = H, F) electroneutral alkylborates **21** are obtained. Compound **17** reacted with methyl iodide to give the 2-ethylimidazollium iodide. With CS_2 the elctroneutral dithiocarboxylate **22** is obtained [32].



21

X = H, F



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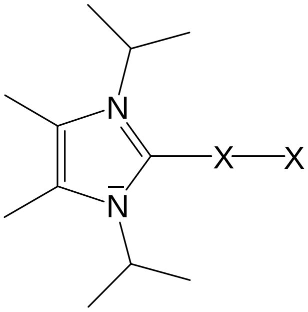
2 Aim of Study

The aims of this study are the preparation of some useful organic and inorganic derivatives of the imidazol-2-ylidene **10c** and 2-methyleneimidazloine **17** by using new and simple methods. Moreover, the compounds will be characterized by the physical methods in addition to the X-ray structure analysis.

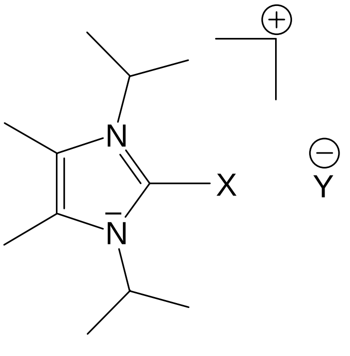
These newly prepared derivatives are classified as:

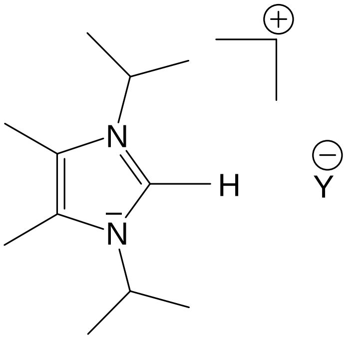
1- Halogen charge transfer adducts of the type **23** of 2,3-dihydroimidazol-2-ylidenes.



23 .

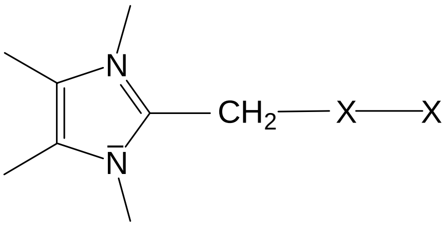
2- Salt derivatives of the types **24a-b** of 2,3-dihydroimidazol-2-ylidenes.





24a **24b**

3- Halogen charge transfer adducts of the type **25** of 2-methyleneimidazolines.



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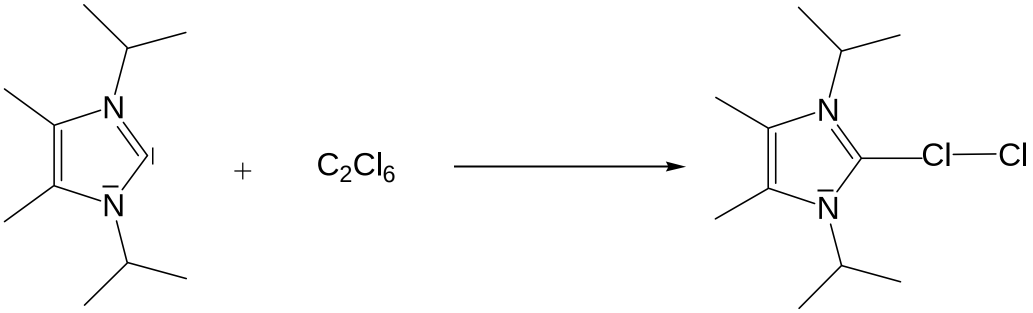
3 Results and Discussion

3.1 Reactions of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene with halogen containing compounds.

3.1.1 Synthesis of 2-chloro-1,3-diisopropyl-4,5-dimethylimidazolium chloride.

Chlorine reacts with **10c** to give a mixture of products. Therefore organic chlorinating reagents are used for the synthesis of **26**. The reaction of **10c** with hexachloroethane gave the charge transfer adduct **26** in excellent yield.



10c **26**

Charge transfer adducts of chlorine have rarely been characterized in the solid state. Triorganophosphanes can be presented as R_3PCl_2 (R = Ph) [33], or as charge transfer adduct $R_3PCl—Cl$ (R = *iso*-Pr) [34].

Kuhn *et al*. published the synthesis of the adduct **26** some time ago via the reaction of **10c** with 1,2-dichloroethane without structural characterization [20].

The salt crystallizes in the monoclinic space group C2/c, the crystal structure of **26** (fig.1, tab.1-5) reveals the presence of a nearly linear C—Cl—Cl fragment [Cl—Cl 3.159(3) Å; C—Cl(1)—Cl(2) 166.1(1)º] which is discussed as weak chlorine interaction causing hypervalency at the central chlorine atom [35]. The Cl—Cl distance lies clearly within the *Van der Waals* interionic range. The limit value discussed of the *Van der Waals* Cl—Cl interaction being a distance of 3.5 Å [36]. The shortest experimentally determined intermolecular Cl—Cl distance is observed in the crystal structure of ClF (3.070 Å) [37].

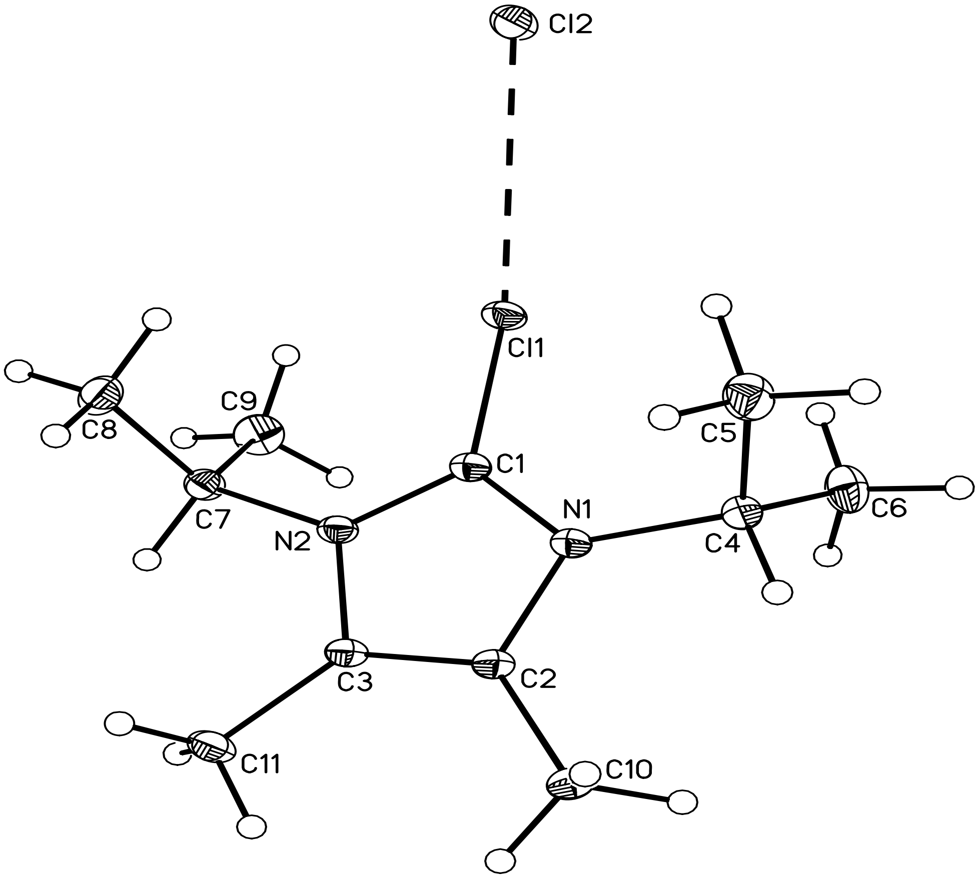
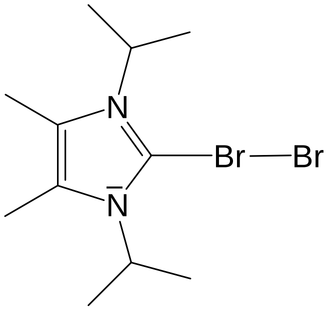


Fig.1 : The crystal structure of $C_{11}H_{20}N_2Cl_2$ (**26**).

3.1.2 Synthesis of 2-bromo-1,3-diisopropyl-4,5-dimethylimidazolium bromide.

The reaction of **10c** with bromine produced compound **27** as stable solid in high yield.



27

The crystal structure of **27** (fig. 2, tab. 6-10) indicates a molecular structure in which the bromide ions are linked to the imidazolium cations through weak halogen to halogen bonds generating a near linear coordination geometry at the central bromine atom [Br—Br 3.1016(8) Å; C(1)—Br(1)—Br(2) 176.33(15)º] [38-39]. The structure of the heterocyclic ring including the exocyclic carbon to bromine bond [C(1)—Br(1 1.881(5) Å] is as expected for imidazolium ions. Interestingly, crystals of **27** are not isomorphous to their chlorine [35] and iodine [38] analogues in which a parallel orientation of the five membered rings has been detected. Apparently, the puckered orientation in **27** does not influence markedly the C—Br—Br bonds.

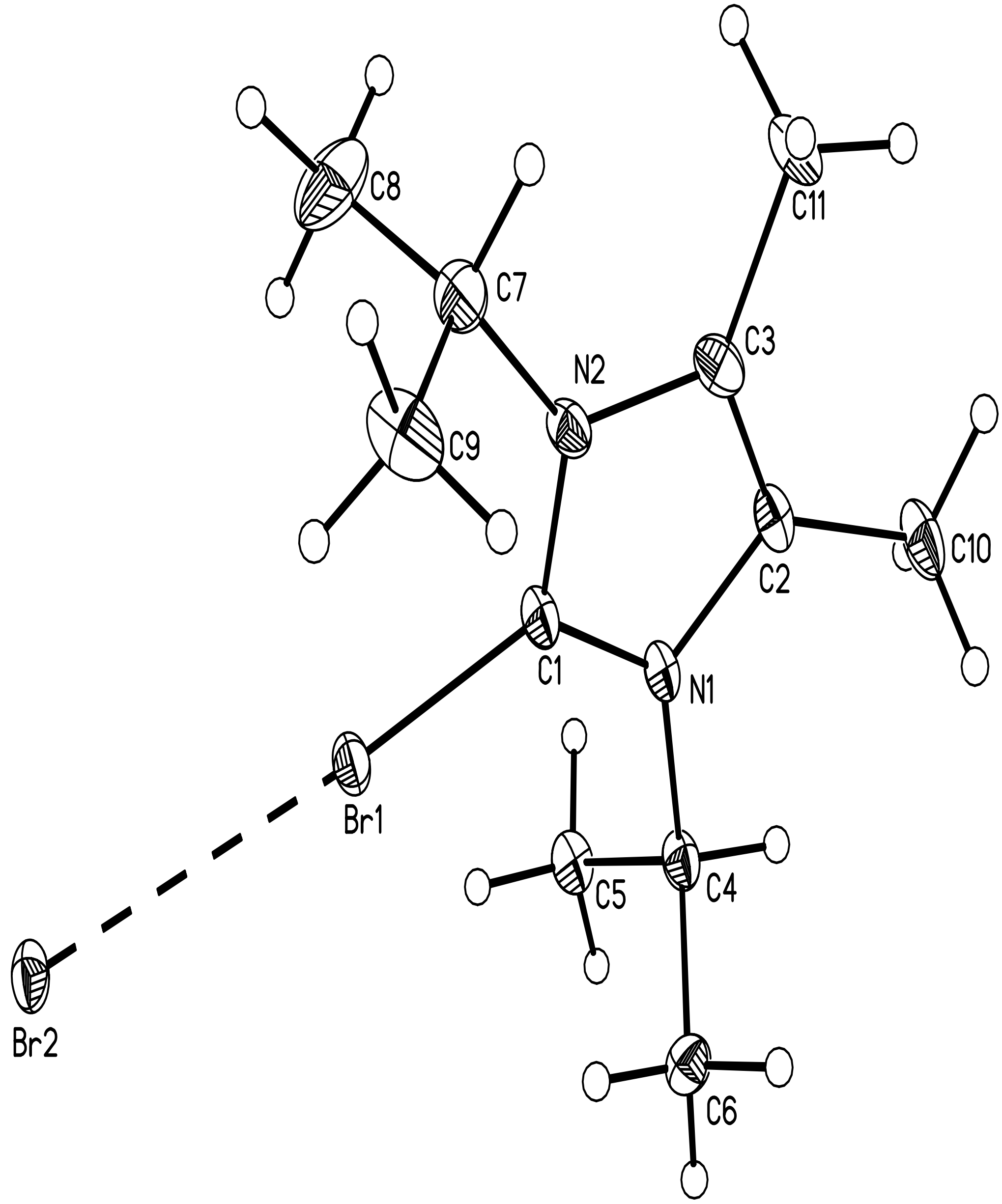
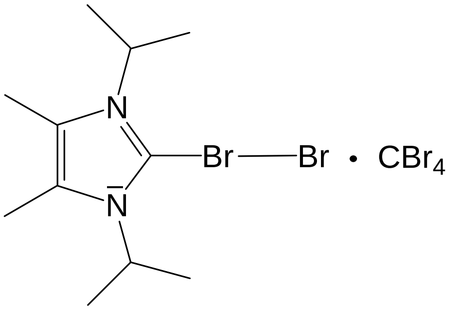


Fig.2 : The crystal structure of $C_{11}H_{20}N_2Br_2$ (**27**).

3.1.3 Synthesis of 2-bromo-1,3-diisopropyl-4,5-dimethylimidazolium bromide tetra bromomethane adduct.

The reaction of **10c** with excess of tetrabromomethane produced compound **28** as a stable solid.



28

The crystal structure of **28** (fig. 3, tab. 11-15) indicates that the incorporation of tetrabromomethane molecule into the unit cell does not markedly influence the structure of the C—Br—Br fragment [C(2)—Br(4) 1.864(8), Br(2)—Br(4) 3.190(16) Å; C(2)—Br(4)—Br(2) 170.6(6)º] [38]. It is thought that the CBr_4 molecule is connected to the bromide ion in a similar manner [Br(2)—Br(3) 3.147(18); Br(3)—C(10) 1.946(16) Å; Br(2)—Br(3)—C(10) 173.8(9)º]. The distance between Br(3) and Br(4) is clearly outside the *Van der Waals* range (4.25 Å). Interestingly, the tetrahedral geometry of CBr_4 molecule is only slightly distorted by this interaction [C—Br 1.927-1.957 Å, Br—C—Br` 108,5 -110.1º] [38].

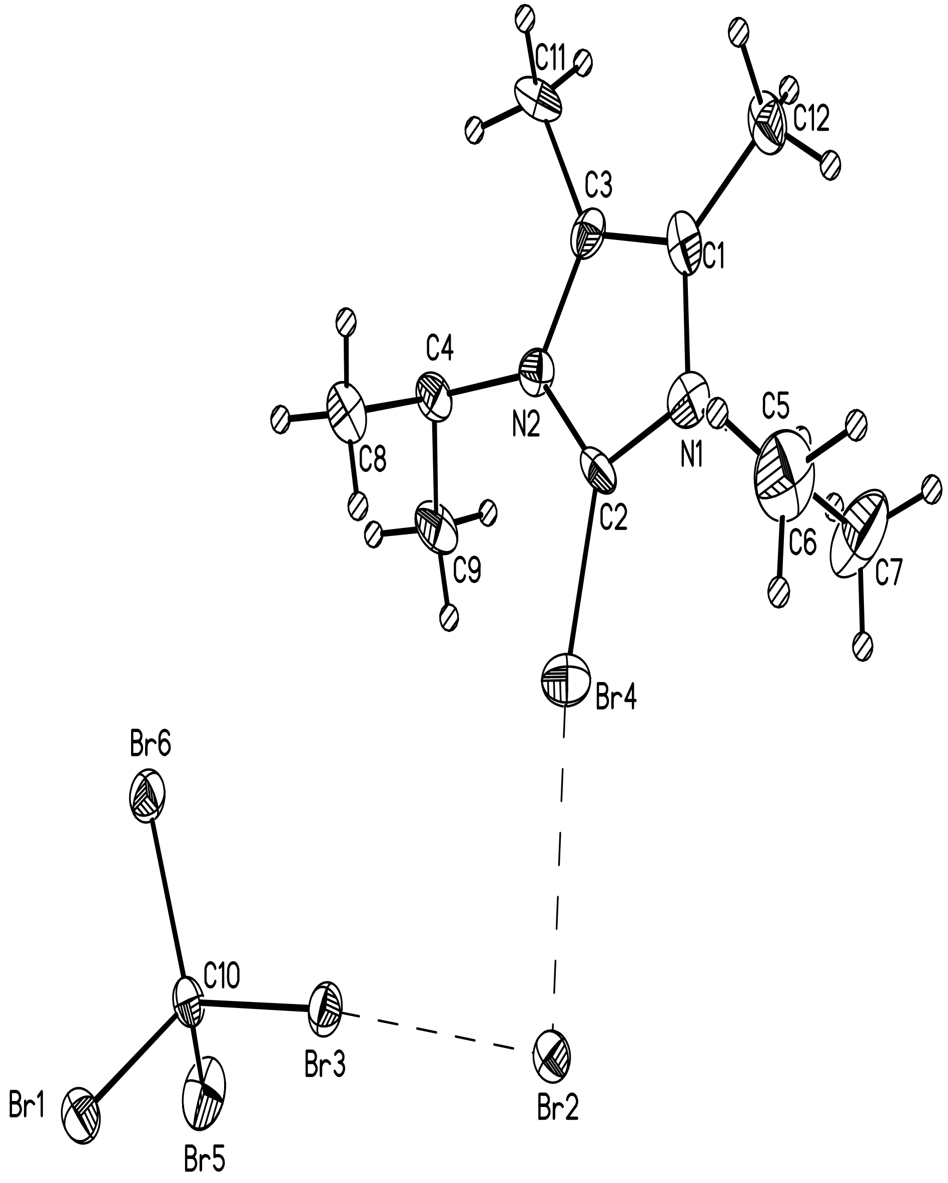
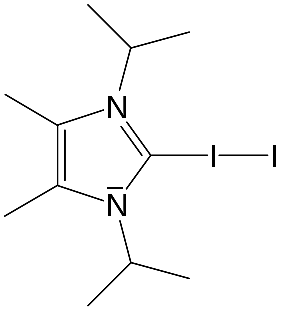


Fig.3 : The crystal structure of $C_{12}H_{20}N_2Br_6$ (**28**).

3.1.4 Synthesis of 2-iodo-1,3-diisopropyl-4,5-dimethylimidazolium iodide.

The reaction of **10c** with iodine in THF produced compound **29** as stable adduct in excellent yield.



29

Imidazol-2-ylidenes [22, 40] and triorganophosphanes [41] form adducts with iodine, which are understood as hypervalent connection of the iodine or as charge transfer complex.

The molecular structure of **29** may be considered as an isolated transition state which resulted from the nucleophilic attack of the carbene on the iodine molecule. The hypervalency caused at the central iodine atom (fig. 4, tab.16-20) is revealed by it's geometry [C(1)—I(2)—I(1) 178.8º] and by the significant lengthening of the iodine to iodine bond [I(2)—I(1) 3.348(8) Å][38]. The C—I bond distance [2.121(14) Å] is longer than normal carbon to halogen bonds. The compound **29** was also found to be analogous to the isolable phosphane adduct Bu_3P—I—I [P(1)—I(2)—I(1) 177.6º, I(2)—I(1) 3.326(1) Å] [41].

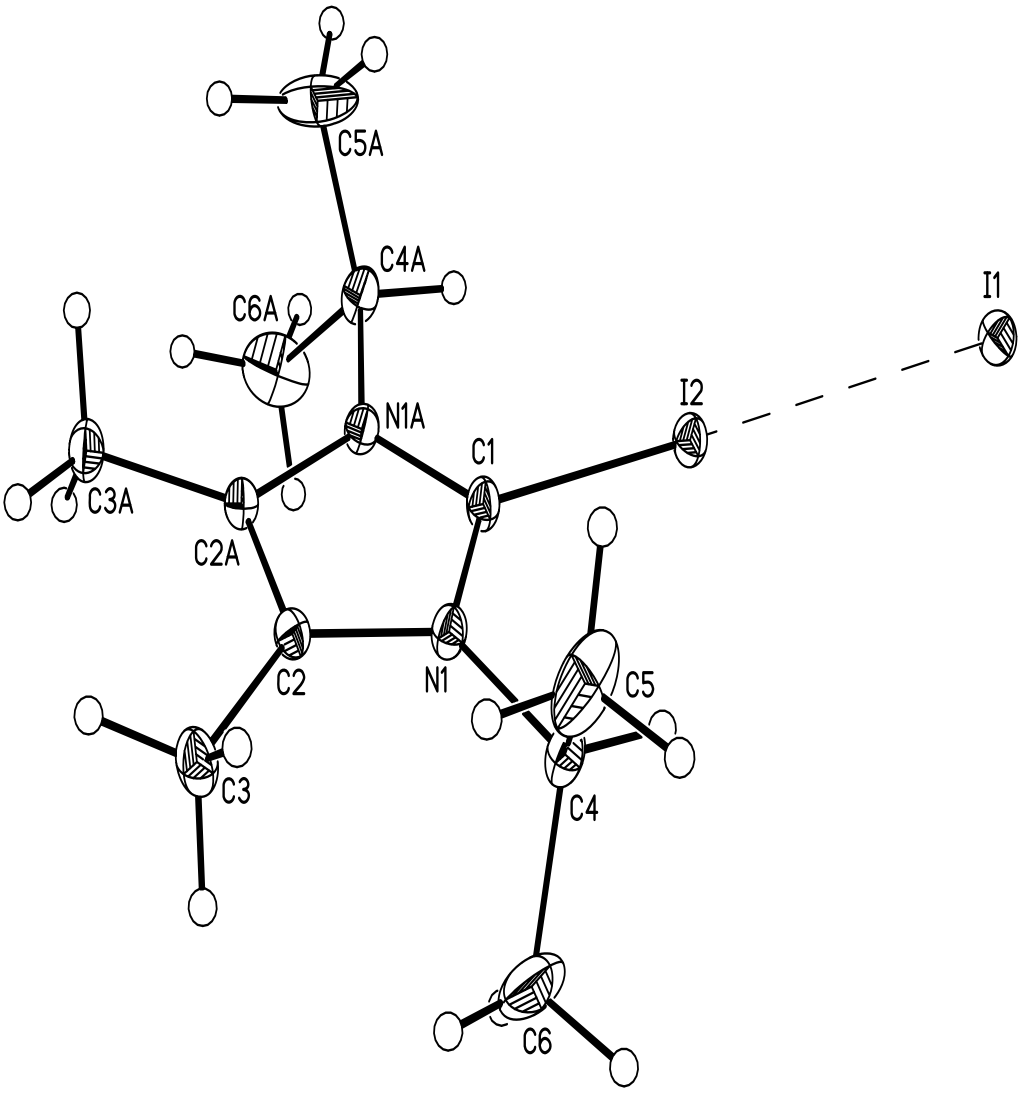
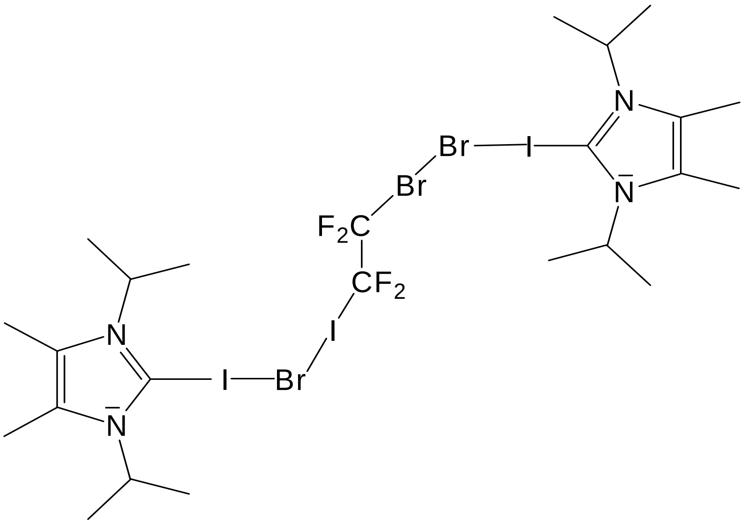


Fig.4 : The crystal structure of $C_{11}H_{20}N_2I_2$ (**29**).

3.1.5 The reaction of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene with 1-bromo-2-iodotetrafluoroethane.

The reaction of **10c** with 1-bromo-2-iodotetraflouroethane produced compound **30** as a stable crystalline solid in high yield.



30

The direct reaction of carbene with IBr did not furnish a satisfying result. Therefore, the IBr transmission was achieved by using 1-bromo-2-iodotetrafluoroethane. Crystals of **30** lie in the centrosymmetric monoclinic, P2(1)/n space group. Here the atoms I(2) and Br(2A) were refined on split positions with the same probability of occupation (fig. 5, tab. 21-25). All interhalogen interactions of iodine and bromine in **30** lying within the *Van der Waals* range values [I(1A)—Br(3) 3.101(6), I(2)—Br(3) 3.220(6), Br(2A)—Br(3A) 3.313(7), I(1AA)—Br(3A) 3.101(6) Å]. The crystal structure of the **30** exhibits linear C—I—Br interactions [C(1A)—I(1A)—Br(3) 177.8(8), Br(3)—I(2)—C(12) 174.1(7), C(12A)—Br(2A)—Br(3A) 168.1(8), Br(3A)—I(1AA)—C(1AA) 177.8(8)°][42]. This results is in agreement with the hypervalency suggested in the formula pattern.

In the crystal structure of iodine monobromide, the intramolecular I—Br distance is 2.52 Å and the shortest intermolecular I—Br distances are 3.18 and 3.76 Å [43]. For the analogous organic I to Br connection, the I—Br bond distance is 3.303 Å, the bond angle C—I—Br is 179.39(14)°, and the C—I bond distance is 2.114(5) Å [44].

The crystals contained 1-bromo-2-iodotetrafluoroethane molecules, which are responsible for the elongation of the distances I(1A)—Br(3) and I(1AA)—Br(3A). Apart from the high basicity of **10c**, the interaction effect is also evident between the virtual bromide ions Br(3) and Br(3A) and the halogen atoms Br(2A) and I(2). On the basis of the lengthening of the I—Br bond [3.101(6) Å] in **30**, the molecule can be envisaged as an ionic $[\mathrm{ImI}]^+\mathrm{Br}^-$ compound, with the ions strongly interacting with each other [45].

The surprising elongation of the Br(2A)—Br(3A) [3.311(7) Å] distance opposite to Br(3)—I(2) [3.220(6) Å] may be due to the orientation of the ethane molecule in the crystal.

The tertiary phosphine adduct of mixed halogens $[R_3PI]Br$ is also known. The I—Br Bond distance is 3.062(2) Å, and the P—I—Br bond angle is 178.0(1)°, but no evidence for the corresponding $[R_3PBr]I$ was observed [46].

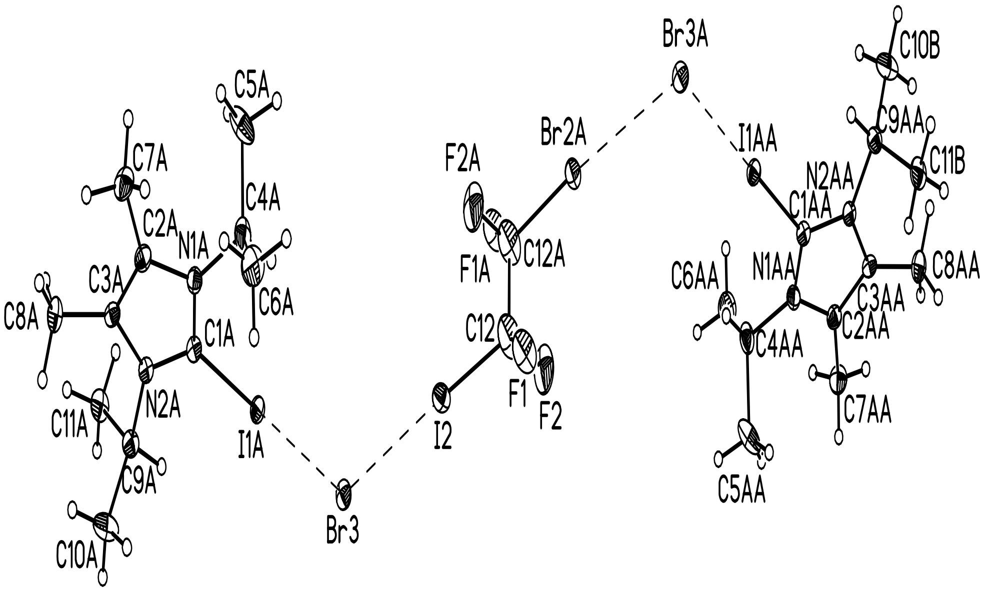
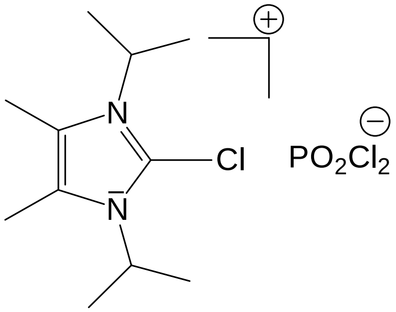


Fig.5 : The crystal structure of $C_{24}H_{40}N_4Br_3I_3F_4$ (**30**).

3.2 Nucleophilic substitution reactions of 2-haloimidazolium halides.

3.2.1 Synthesis of 2-chloro-1,3-diisopropyl-4,5-dimethylimidazolium dichlororphosphate

The reaction of **26** with $POCl_3$ in the presence of water produced compound **31** as a stable salt in good yield.



31

Dichlorophosphate exists as a stable organic salt, M[PO_2Cl_2] [47] and acts in metal complexes as a coordinating ligand. Two structure reports of their salts have been mentioned, but they do not include a description of the anion, apparently as a consequence of disordering problems [48]. Compound **31** is obtained from the hydrolysis of $POCl_3$ [49] in the presence of **26**.

The salt crystallizes in the orthorhombic space group *P*nma (fig. 6, tab.26-30) [50]. Both ring atoms of the cation and the PCl_2 fragment of the anion lie on crystallographic mirror planes. A view on the interionic distances reveals the presence of separated ions in the unit cell. The closest cation to anion Cl—Cl distance [Cl(1)—Cl(2) 508.2(1) pm] is out of the range of the *Van der Waals* radii, and even longer than those observed inside both the cations [Cl(1)—Cl(1#) 497.0(1) pm] and the anions [Cl(2)—Cl(2#) 467.4(1) pm] but shorter than the closest cation to anion separation. This finding contrasts markedly to the significant Cl—Cl interaction in **26** (315.9 pm) [35] and documents the low nucleophilicity of the PO_2Cl_2 anion towards the 2-chloroimidazolium ion. Comparing the structure of the cations in **31** and **26**, a change is detected in the orientation of the isopropyl groups. The presence of intraionic C—H—Cl hydrogen bridges is also detected [C(5)—H(5A) 90(4); H(5)—Cl(1) 277(3), C(9)—H(9) 93(3), H(9)—

Cl(1) 280(3) pm; C(5)—H(5)—Cl(1) 123(2), H(5)——Cl(1)—C(1) 82.5(5), C(9)—H(9)—Cl(1) 118(2); H(9)——Cl(1)—C(1) 81(8) º] which may be influenced by the absence of interionic Cl—Cl interaction in **31**.

The structure of the PO_2Cl_2 anion follows the expectations of the structural phosphorous chemistry [51] and the prediction of the VSEPR rule [52]. On comparison with the carbene base adducts of PO_2Cl we see only minor changes in the structure of the PO_2Cl fragment underlining the marked nucleophilicity of the carbene ligand there [53]. Comparison with structural data of $[PO_2Cl_2]^-$ ligand complexes [54-55] reveals a minor change in the structure of the ligand on coordination apart from some differences in the O—P—O' angles. It is therefore suggested that the metal to oxygen bonds in the coordination compounds of the dichlorophosphate ligand are mainly of electrostatic nature both in the terminal [54] and bridging [56] coordination mode.

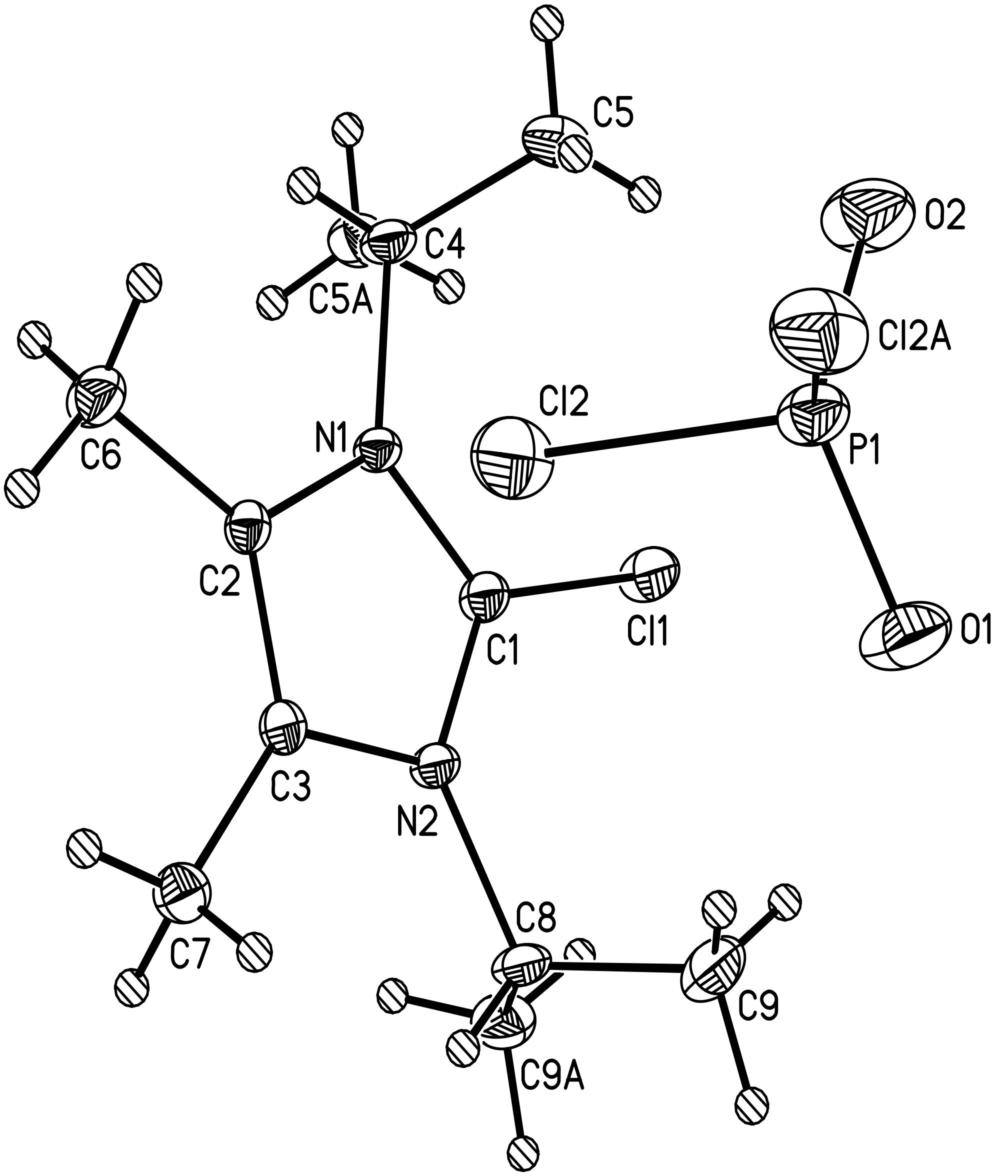
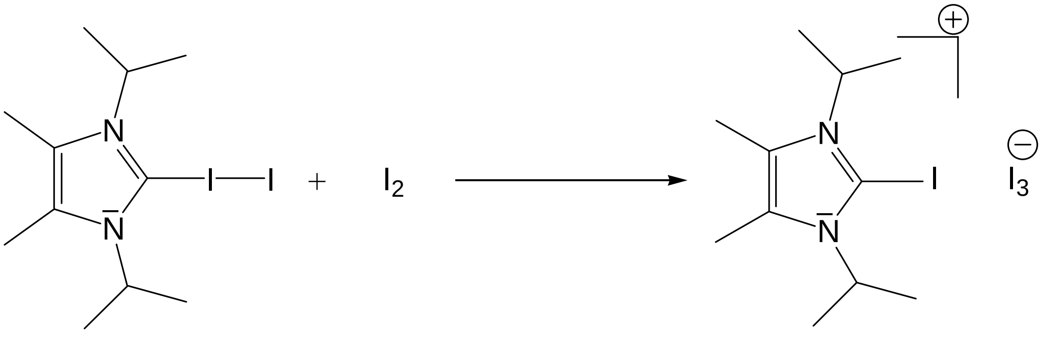


Fig.6 : The crystal structure of $C_{11}H_{20}N_2PCl_2O_2$ (**31**).

3.2.2 Synthesis of 2-iodo-1,3-diisopropyl-4,5-dimethylimidazolium triiodide

The reaction of I_2 with **29** gave compound **32** as a stable crystalline solid in high yield.



29

32

The salt crystallizes in the monoclinic space group P2(1)/c (fig.7 tab. 31-35) [57]. The C—I distance is 2.082(11) Å, which is smaller than the distance in **29** [2.121(4) Å] [38], but longer than the distance in a carbene analogous triiodide compound [2.07(6) Å] [58]. The internal ring angle at the carbene center [N—C—N 109.0(9) º] is larger than that in **29**. The triiodide fragment is almost linear [I(3)—I(2)—I(4) 179.15 º] and has moderately symmetric bond length distribution [I(3)—I(2) 2.947(2). I(3)—I(2) 2.891(2) Å]. The difference between the two I—I distances [Δ = 0.056(2)] is much less than that found in $[PPh_3I]I_3$ where Δ = 0.140(1) [59]. The weak interactions of the cations with the triiodide anions are within the *Van der Waals* range. The I(1)—I(3) bond distance [3.521 Å] is shorter than that found in the analogous 2-methyleneimidazoline triiodide compound [4.071(1)Å] [60].

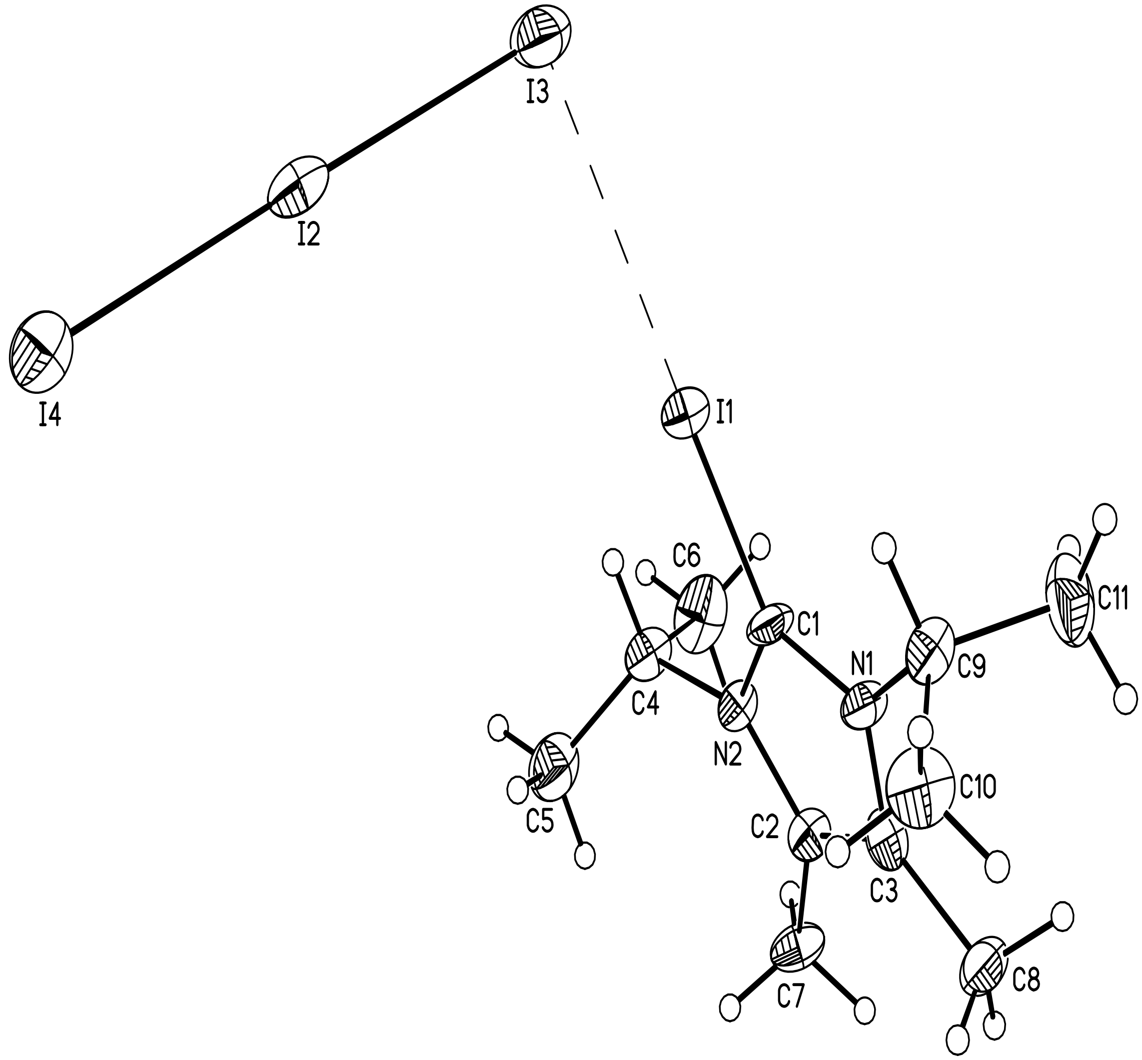
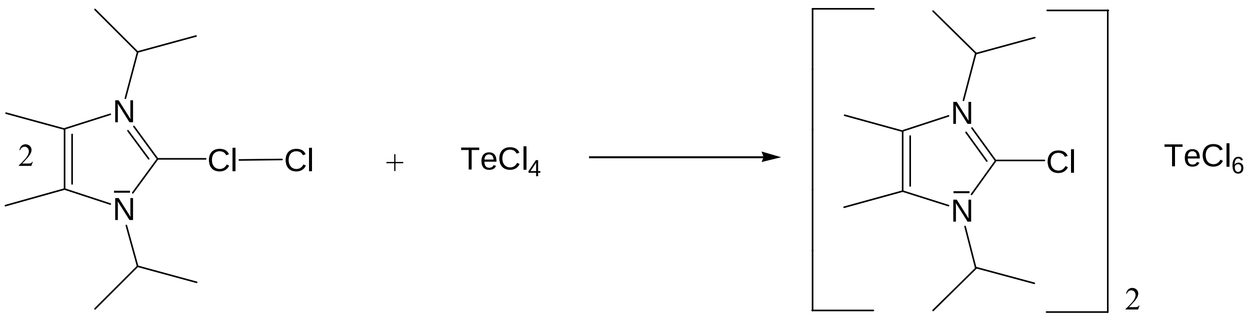


Fig.7 : The crystal structure of $C_{11}H_{20}N_2I_4$ (**32**).

3.3 Reactions of 2-haloimidazolium halides and imidazol-2-ylidenes with tetrahalotellurium

3.3.1 Synthesis of 2-chloro-1,3-diisopropyl-4,5-dimethylimidazolium hexachloro-tellurate(IV).

The reaction of **26** with $TeCl_4$ in CH_2Cl_2 gave the hexachlorotellurate compound **33** as a stable salt in excellent yield.



26 **33**

The crystal structure (fig. 8, tab. 36-40) reveals the presence of an ionic pair linked by weak Cl to Cl interactions, and indicates the presence of interionic secondary bonding in **33** including the bond distances and angles at the bridging halogen atoms characteristic of this type of interaction [Cl(2)—Cl(4) 3.196(1) Å; C(1)—Cl(4)—Cl(2) 161.9(1), Cl(2)—Cl(4)—Te(1) 113.7(2) °] [61]. On comparison with **26** a minor difference is observed [Cl(1)—Cl(2) 3.159 Å; C(1)—Cl(1)—Cl(2) 166.1°]. The compound crystallizes in the centrosymmetrical monoclinic P2(1)/n space group with similar arrangements of the ions to **34**. The Te atom lies on a crystallographic inversion center.

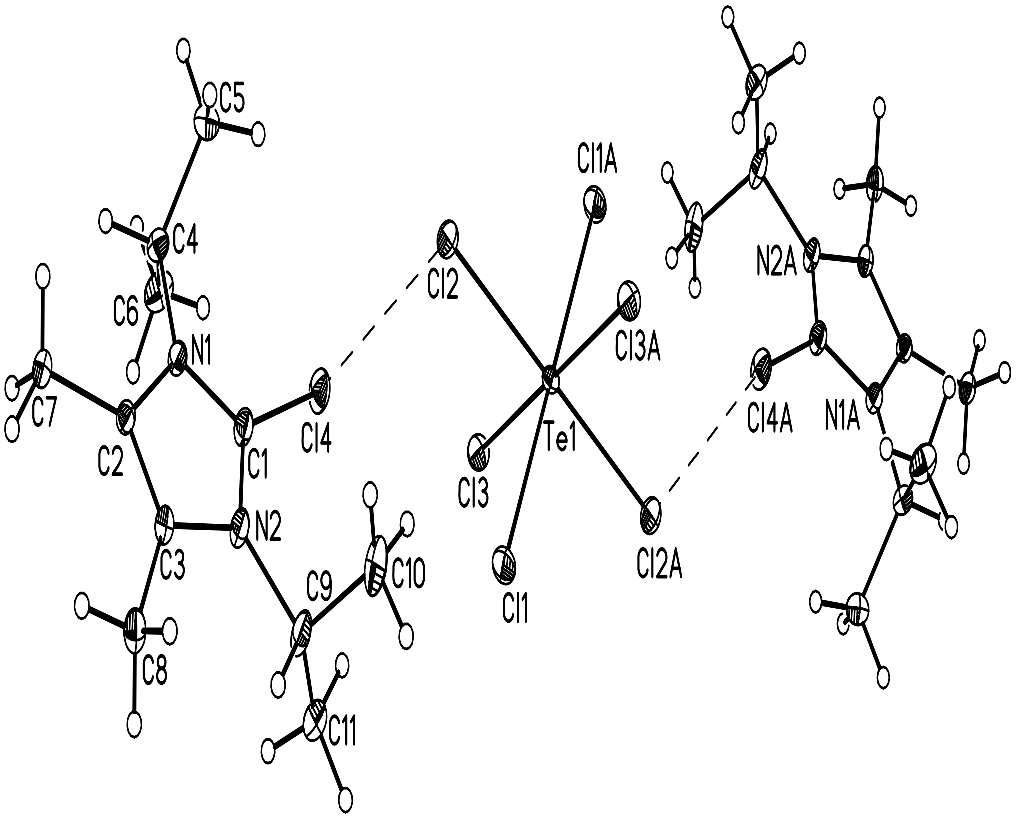
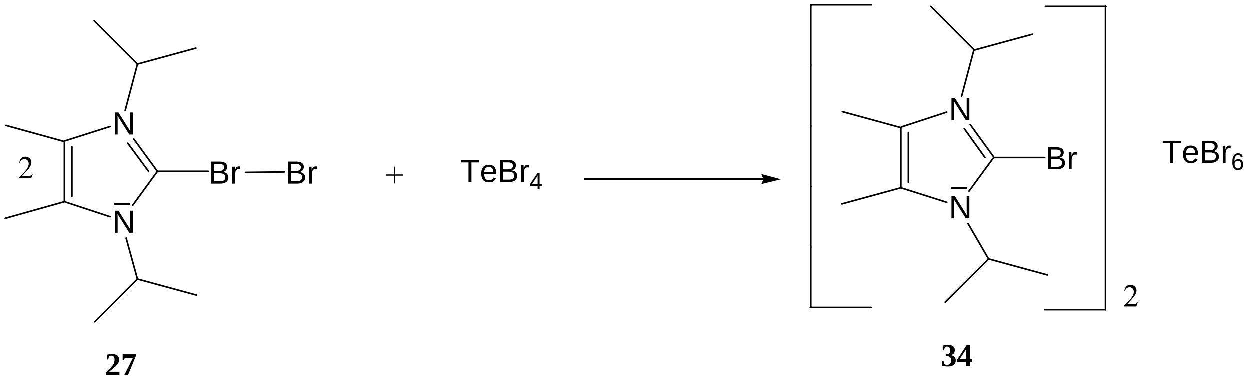


Fig.8 : The crystal structure of $C_{22}H_{40}N_4Cl_8Te$(**33**).

3.3.2 Synthesis of 2-bromo-1,3-diisopropyl-4,5-dimethylimidazolium hexabromo-tellurate(IV).

The reaction of **27** with $TeBr_4$ in CH_2Cl_2 the hexabromotellurate gave compound **34** as a stable salt in high yield.



The crystal structure (fig. 9, tab. 41-45) reveals the presence of an ionic pair linked by weak Br to Br interactions and indicates the presence of interionic secondary bonding in **34**. The bond distances and angles at the bridging halogen atoms are characteristic of this type of interaction [Br(1)—Br(3) 3.419(4) Å; C(1)—Br(1)—Br(3) 161.7(1), Br(1)—Br(3))—Te(1) 113.0(2) °] [61]. On comparison with **27** [Br(1)—Br(2) 3.102 Å; C(1)—Br(1)—Br(2) 176.3 °] a significantly stronger interaction is observed, and this confirm the nucleophilicity of the anion to be more relevant for the interaction in the softer bromine derivatives. The crystals contain two molecules of CH_2Cl_2 per formula unit and therefore are not isomorphous to **33**. Weak interactions are detected between the solvent molecules and the anions in **34** [H(12B)—Br (1) 2.769(2) Å]. The compound crystallizes in the centrosymmetrical triclinic space group P-1 with similar arrangements of the ions to **33**. The Te atom lies on a crystallographic inversion center.

No reaction could be detected between the compound **29** and TeI_4 apparently as a result of the solid state stability of both compounds.

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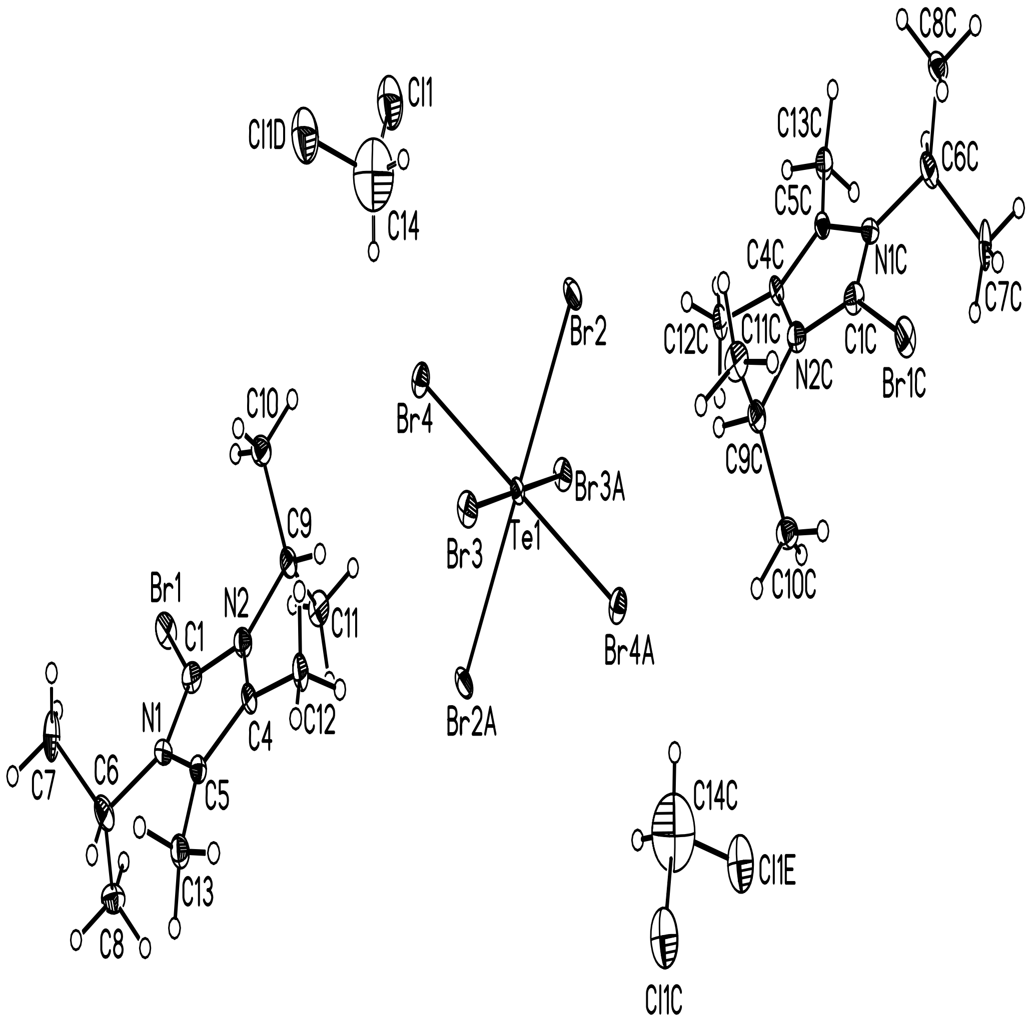
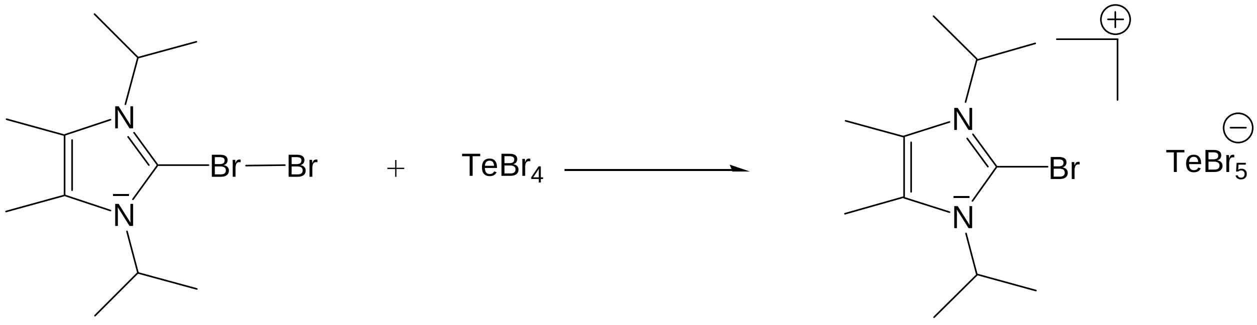


Fig.9 : The crystal structure of $C_{22}H_{40}N_4Br_8Te$. $2CH_2Cl_2$ (**34**).

3.2.3 Synthesis of 2-bromo-1,3-diisopropyl-4,5-dimethylimidazolium pentabromotellurate (IV).

The reaction of **27** with $TeBr_4$ in CH_2Cl_2 the pentabromotellurate gave compound **35** as a stable solid in high yield.



27 **35**

The crystal structure of **35** (fig 10, tab. 46-50) [38] indicates that the salt crystallizes in the triclinic space group P-1. The coordination of the bromide ion to the $TeBr_4$ unit lowers the nucleophilic character of the anion, causing a remarkable decrease of interaction in **35**. This finding is in agreement with the literature [62]. A significant lengthing of the closest interionic Br to Br distance [Br(4)— Br(6) 3.745(4) Å] is observed comparing this bond with the Br—Br distance in **27**. The structure of the $TeBr_5$ anion agrees with the reported data [63].

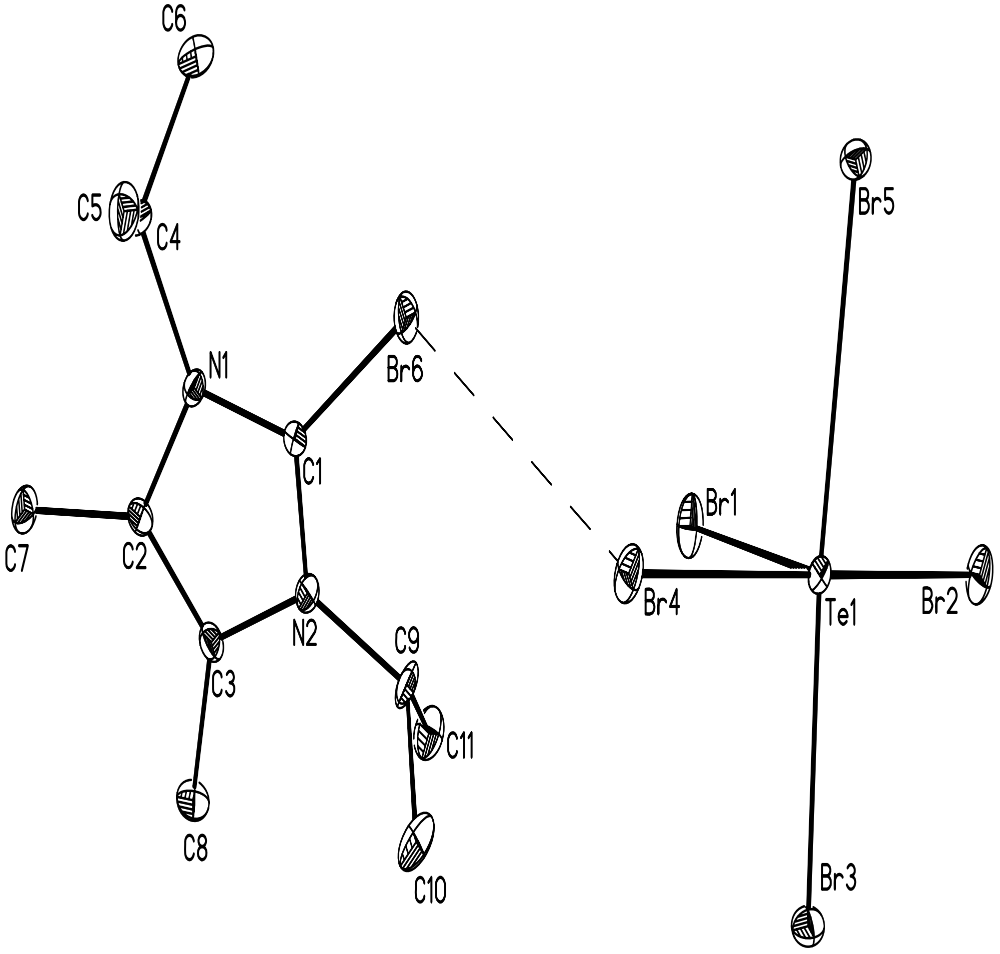


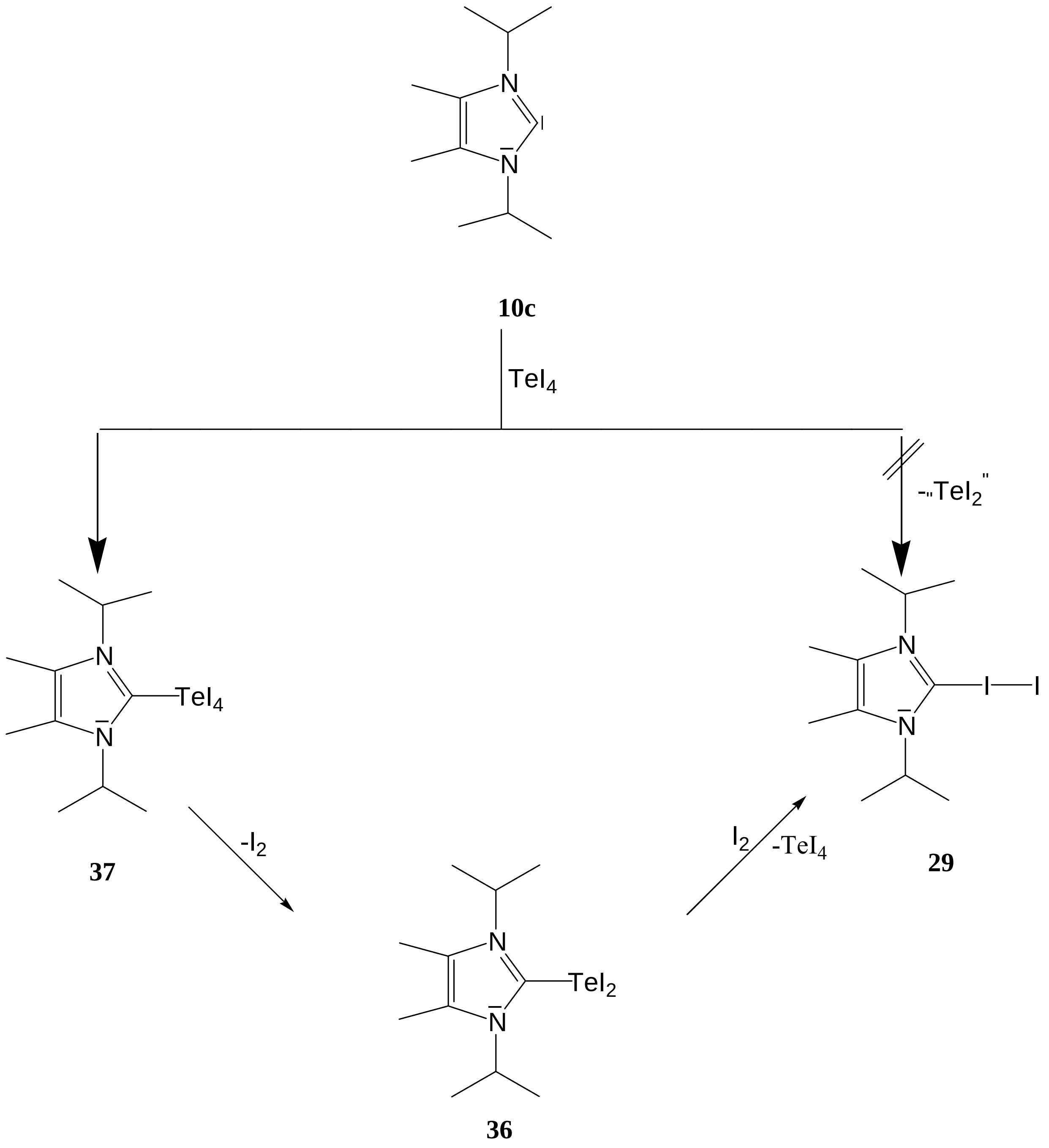
Fig.10 : The crystal structure of $C_{11}H_{20}N_2Br_6Te$ (**35**).

3.3.4 Synthesis of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene(diiodo)tellurium.

The carbene adduct **36** is obtained from the reaction of **10c** with TeI_4 as the sole product. Apparently, **36** is formed by the removal of iodine from the intermediate **37**. After prolonged heating of the reaction mixture the salt **29** is observed [38].

The crystal structure (fig. 11, tab. 51-55) [64] reveals a T chain structure in which the monomeric fragments are connected by weak Te to I interactions, which similarly have been found in ionic triorganotellurium iodides (3.6-3.9 Å) [65]. The iodo substituents are situated in the axial positions. The nonbonded Te…I distance is approximately 0.8 Å longer than the Te—I bonds (fig. 12) [Te(1b)—I(1) 3.714; Te(1b)—I(1b) 2.945 Å]. Both the structure of the monomeric unit [Te(1)-I(1) 2.945(1), Te(1)-I(2) 2.933(1), Te(1)-C(1) 2.105(4) Å; I(1)-Te(1)-I(2) 169.41(1), I(1)-Te(1)-C(1) 84.8(1), I(2)-Te(1)-C(1) 84.7(1) °] as well as the intermolecular angles and distances of the helical chain [Te(1)-I(1A) 3.714(1) Å, Te(1)-I(1A)-Te(1A) 133.7(1), I(1)-Te(1)-I(1A) 96.8(1) °] closely resemble to those found for the dimeric carbene adduct reported previously [66]. The Te—I distances in **36** are closely related to those found in $RTeI_2^-$ anions (ca. 2.9 Å) [67].

Reaction of **10c** with $TeCl_4$ or $TeBr_4$ results in complicated mixtures which may contain products of the types **36-37**, and **21**. The higher reactivity of the intermediates formed does not allow stopping the reactions sequence.


10c
TeI4
-"TeI2"
N
N
I
TeI4
37
-I2
I2
-TeI4
I—I
29
TeI2
36

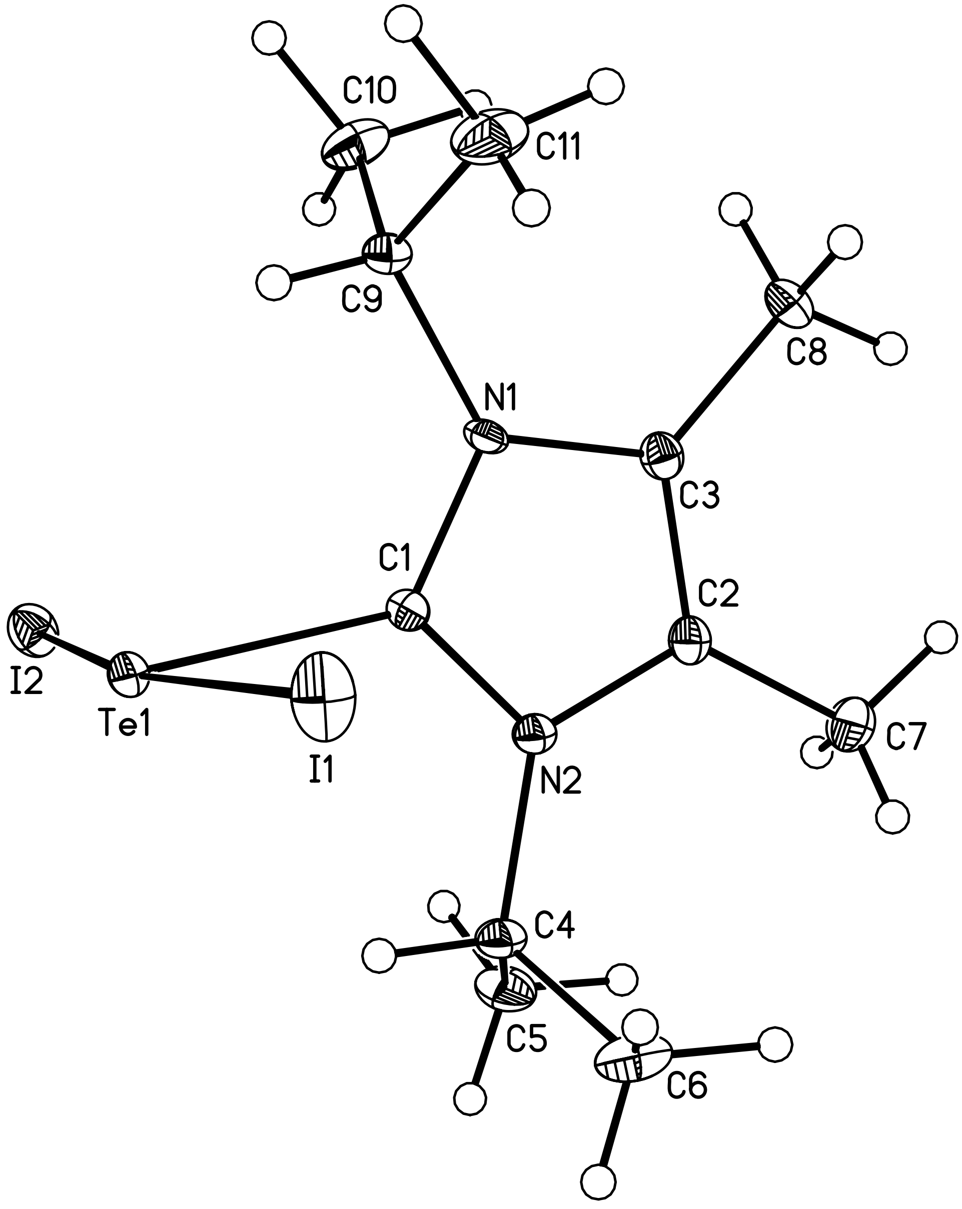


Fig.11 : The crystal structure of $C_{11}H_{20}N_2I_2Te$ (**36**).

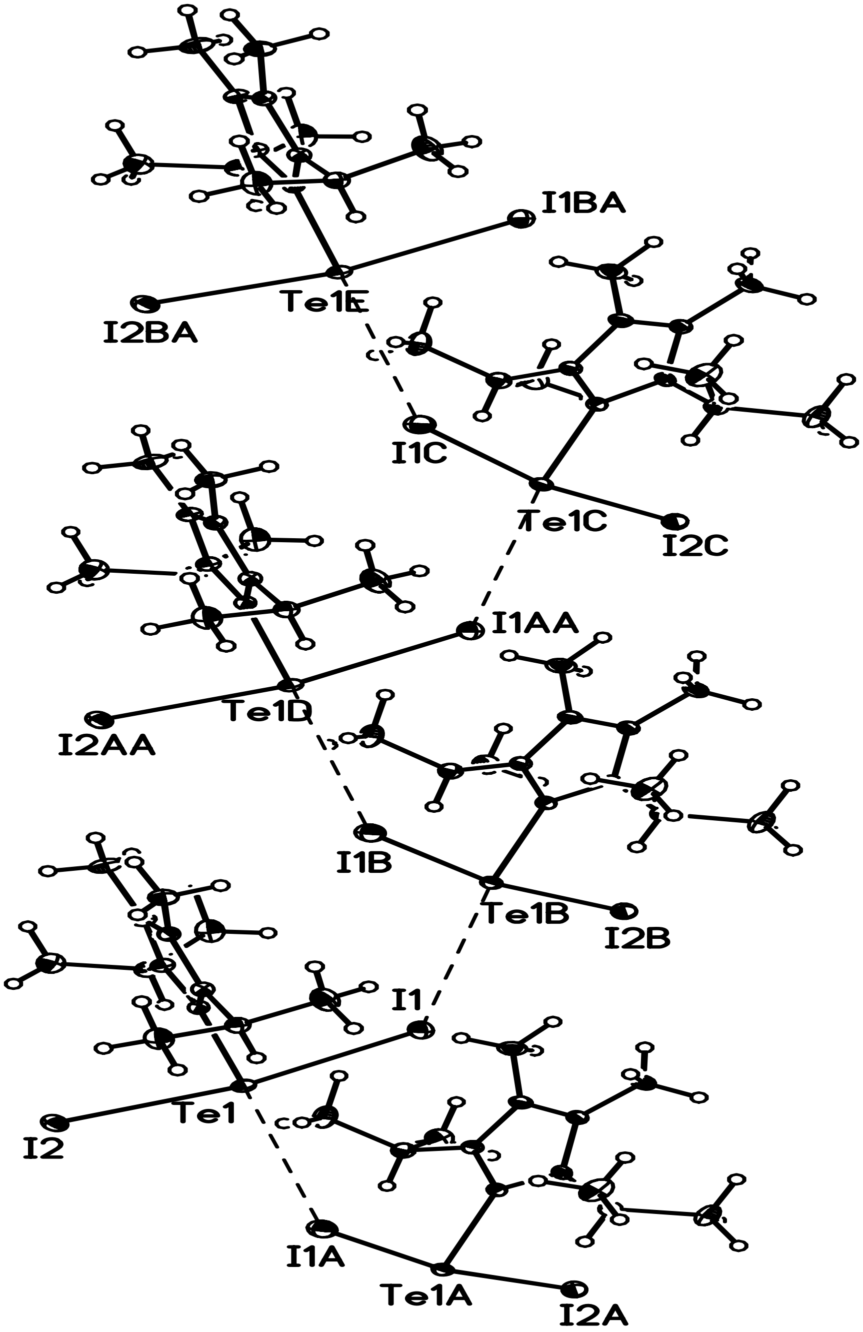
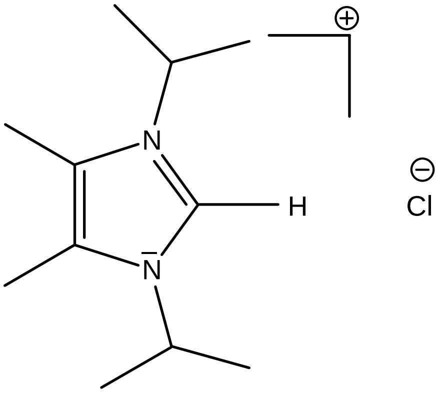


Fig.12 : The molecular packing of $C_{11}H_{20}N_2I_2Te$ (**36**) -

3.4 Nucleophilic reactions of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene.

3.4.1 Synthesis of 1,3-diisopropyl-4,5-dimethylimidazolium chloride.

The reaction of **10c** with NH_4Cl in Et_2O produced compound **38** as a stable solid in excellent yield.



. **38**

The crystal structure of **38** (fig. 13, tab. 56-60) is in agreement with other imidazolium salts which are reported [20, 68]. It proves interionic connection through hydrogen bridge and the crystal structure reveals the presence of a near linear C—H—Cl fragment [C(1)—H(1) 0.953, Cl(1)—H(1) 2.478 Å,C(1)—H(1)—Cl(1) 172.2(3) °][69].

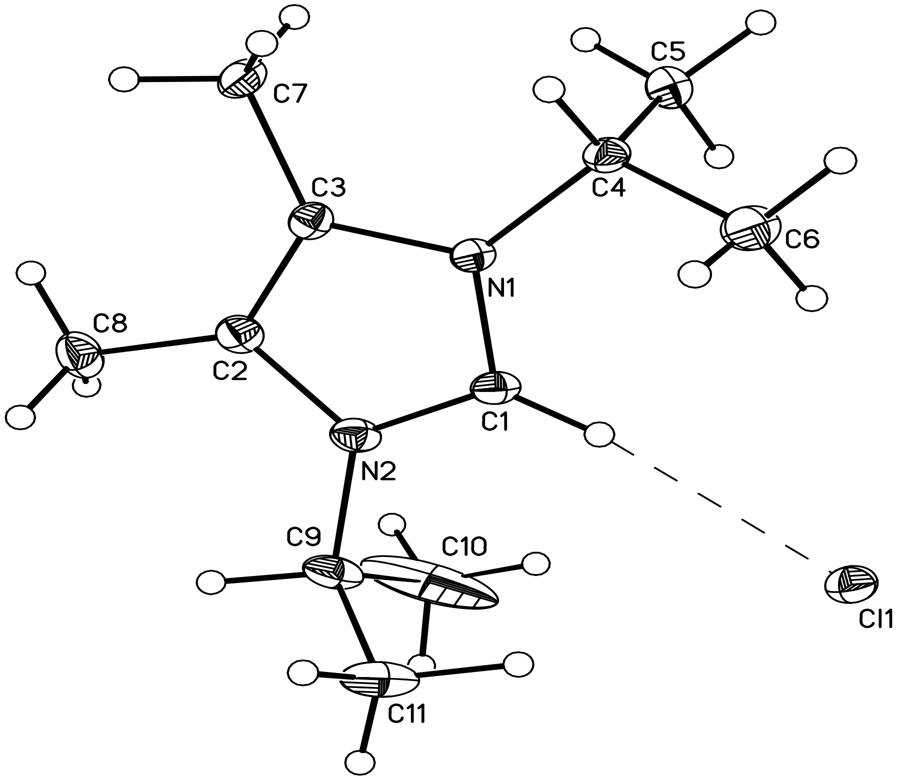
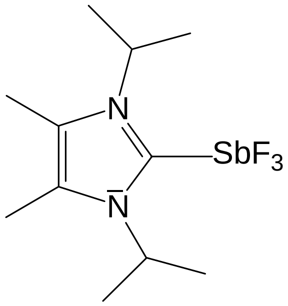


Fig.13 : The crystal structure of $C_{11}H_{21}N_2Cl$ (**38**).

3.4.2 The reaction of 1,3-diisopropyl-4,5dimethylimidazol-2-ylidene with antimonytrifluoride.

Carbene adducts of the heavy main group element centers [70] are easily split owing to the low stability of the carbon to element bond. The high basicity of the carbenes allows here the basic hydrolysis by the nucleophilic attack of the OH anion. Attempts to crystallize **39** from solvent led to **40** and **41** products [71].



39

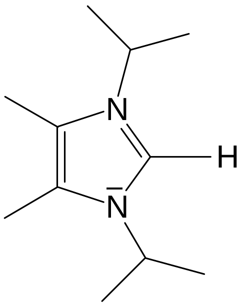
$M_2[Sb_2F_6O]$

40

$M_2[Sb_3F_7O_2]$

41

M =



The crystal structure of **40** (fig. 14, tab. 61-65) shows that the anion $[Sb_2F_6O]^{2-}$ consists of two SbF_2 fragments linked by a symmetrical oxygen bridge and two unsymmetrical fluorine bridges to from a distorted *Ψ*-octahedral coordination sphere at the antimony atoms. The cations are connected with two terminal fluorine substituents by hydrogen bonds. Interionic interaction of the anions over fluorine bridges does not occur.

The coordination environment of the antimony atoms in **40** is to be for Sb(1) more simple. An evaluation of the connection angles results in 353.5° for the four fluorine atoms connected with Sb(1) [F(1)—Sb(1)— F(2) 83.9(2), F(2)—Sb(1)—F(3) 86.1(2), F(3)—Sb(1)—F(6) 75.3(1), F(1)—Sb(1)—F(6) 108.2(1)°]. The structure can be described as a strongly distorted octahedron, in which the oxygen substituent and the lone pair of electrons are arranged in trans position to each other. The space requirement of the lone pair of electrons leads to noticeable contraction of the F—Sb—O angle with the sum of the angles 323.3 ° [F(1)—Sb(1)— O(1) 83.9(2), F(2)—Sb(1)—O(1) 91.8(2), F(3)—Sb(1)—O(1) 83.2(1), F(6)—Sb(1)—O(1) 64.4(1)°]. The Sb(2) fragment has similar finding. The clear deviation of the values of ideal geometry results from the integration of two unsymmetrical fluorine substituents into the cage structure, which additionally contains a symmetrical oxygen bridge [Sb(1)—F(3) 208.1(3), Sb(1)—F(6) 278.5(7), Sb(2)—F (3) 293.4(1), Sb(2)—F(6) 210.0(4), Sb(1)—O(1) 193.5(3), Sb(2)—O(1) 192.4(3) pm; Sb(1)—F(3)—Sb(2) 79.5(1), Sb(1)—F(6)—Sb(2) 82.8(1), Sb(1)—O(1)—Sb(2) 115.9(2)°]. The connection parameters of hydrogen bonds correspond to the expected structure of the organic fragment [H(1A)—F(1) 258 (4), H(1A)—C(1) 83(5), F(4)—H(12A) 215(6), H(12A)—C(12) 80(6) pm; F(1)—H(1A)—C(1) 117(4); F(4)—H(12A)—C(4) 174(6)°] [68, 69].

A comparison of **40** with the structure of the anion $[F_3Sb(\mu\text{-}OH)SbF_3]^-$ shows a clear difference of the bond lengths and bond angles at the oxygen atoms in the monoanion [Sb—O 227, 232 pm; Sb—O—Sb’ 124.7°][72]. The fluorine bridging ion in **40** here is replaced by interionic interaction. The structure of the neutral molecule $[F_2Sb—O—SbF_2]$ measured as an urea adduct exhibits comparable same Sb—O bond distances [Sb—O 196, Sb’—O 197 pm; Sb—O—Sb’ 125.9°] to **40**. The fluorine bridging does not occur [73].

In $[Sb_3F_7O_2]^{2-}$ **41** (fig. 15, tab. 66-70) the two symmetrical equivalent SbF_2 fragments are linked by a symmetrical fluorine bridge [Sb(1)—F(1) 226.8(2), Sb(3)—F(2) 198.7(2), Sb(1)—F(3) 192.5(3) pm; Sb(1)—F(1) —Sb(1A) 131.26(15) °]. Cations are also linked by CHF hydrogen bonding at F(2) [C(1)—H(1A) 75.9(8), H(1A)—F(1) 233.8(10) pm]. The third antimony atom Sb(2) is connected with the each SbF_2 by a symmetrical oxygen bridge and an unsymmetrical fluorine bridge [Sb(2)—O(1) 193.5(3), Sb(1)—O(1) 192.4(3); Sb(2)—F(4) 207.4(3); Sb(1)—F(4)1) 287.485) pm; Sb(1)—O(1)—Sb(2) 120.7(2); Sb(1)—F(4)—Sb(2) 83.7(1) °]. The Sb(2) does not have any terminal fluorine substituents.

The coordination polyhedron of the Sb(1) atom in **41** is derived from an octahedron and corresponds to a large extent to the findings described for **40**. Here the oxygen atom can also be assigned to the trans position to the lone pair of electrons of the antimony center [F(1)—Sb(1)—O(1) 82.1(1), F(2)—Sb(1)—O(1) 88.7(1), F(3)—Sb(1)—O(1) 93.1(1), F(4)—Sb(1)—O(1) 64.4(1)°]. In the same way clear bending of the equatorial axis is confirmed by installation of the fluorine atoms F(1) and F(4) into the cage structure.

The Sb(2) atom in **41** has four coordinating atoms. It's coordination geometry corresponds to trigonal-bipyramidal, the lone pair of electrons with oxygen atoms occupying the equatorial positions [O(1)—Sb(2)—O(1A) 100.0(1), F(4)—Sb(2)— F(4A) 160.6(1), O(1)—Sb(2)—F(4) 84.14(2), O(1A)—Sb(2)—F(4) 83.4(1)°].

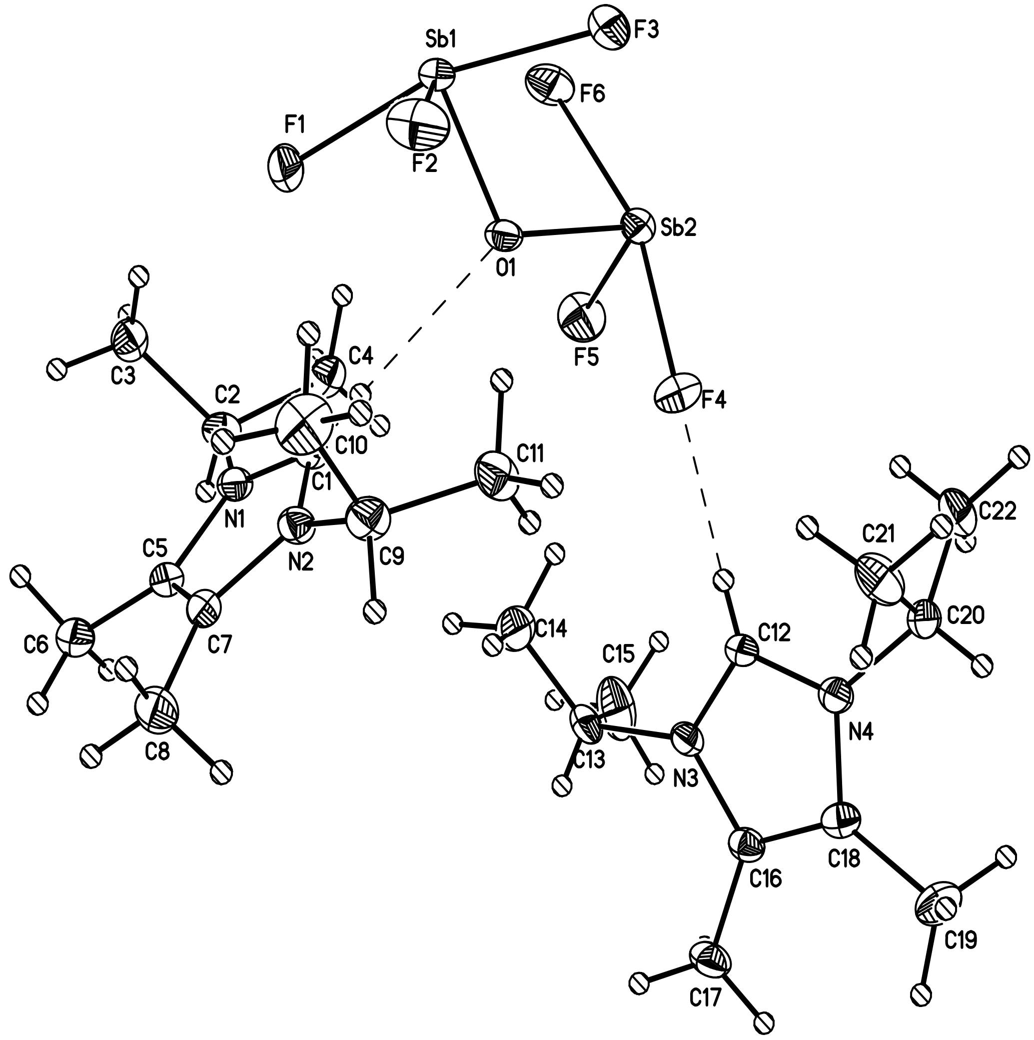


Fig.14 : The crystal structure of $C_{22}H_{42}N_4Sb_2F_6O$ (**40**).

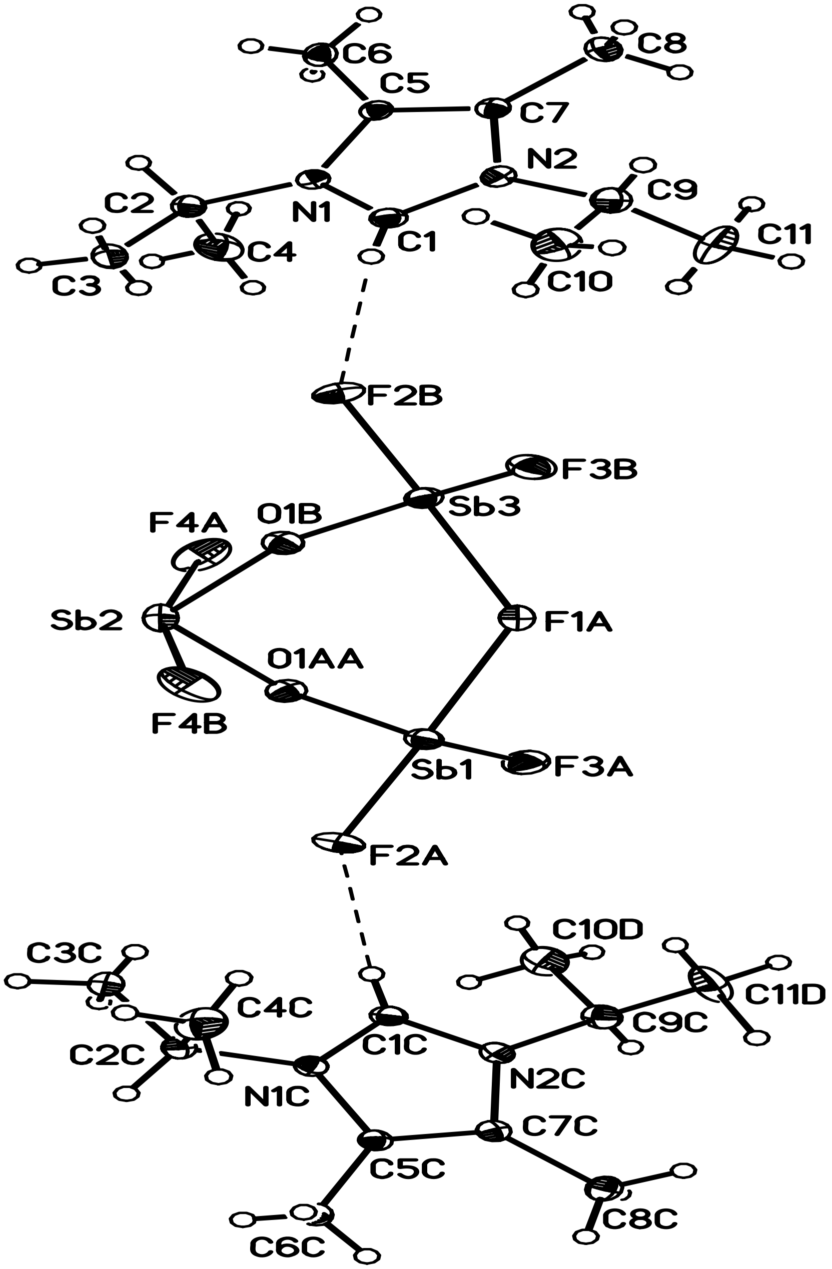
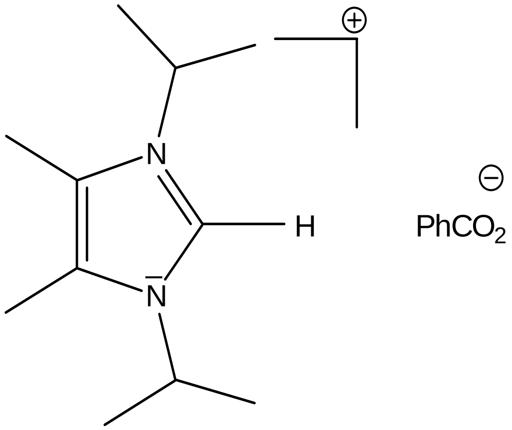


Fig.15 : The crystal structure of $C_{22}H_{42}N_4Sb_3F_7O_2$ (**41**).

3.4.3 Synthesis of 1,3-diisopropyl-4,5-dimethylimidazolium benzoate.

The reaction of **10c** with benzoic acid in Et_2O produced compound **42** as a stable solid in very high yield.



42

The crystal structure (fig. 16, tab.71-75) [74] indicates that the salt crystallizes in the monoclinic space group P2(1)/c and shows that the imidazole fragment is joined to the benzoate ion through hydrogen bonding. The crystal structure reveals the presence of a near linear C—H—O fragment [O(111)—H(1) 2.1588 Å, C(1)—H(1)—O(111) 179.52º]. The C—O bond distances are in excellent agreement with each other and their average value being 1.242 Å. The O(112)—C(111)—O(111) bond angle is 125.2(2) which is in agreement with the reported values [75].

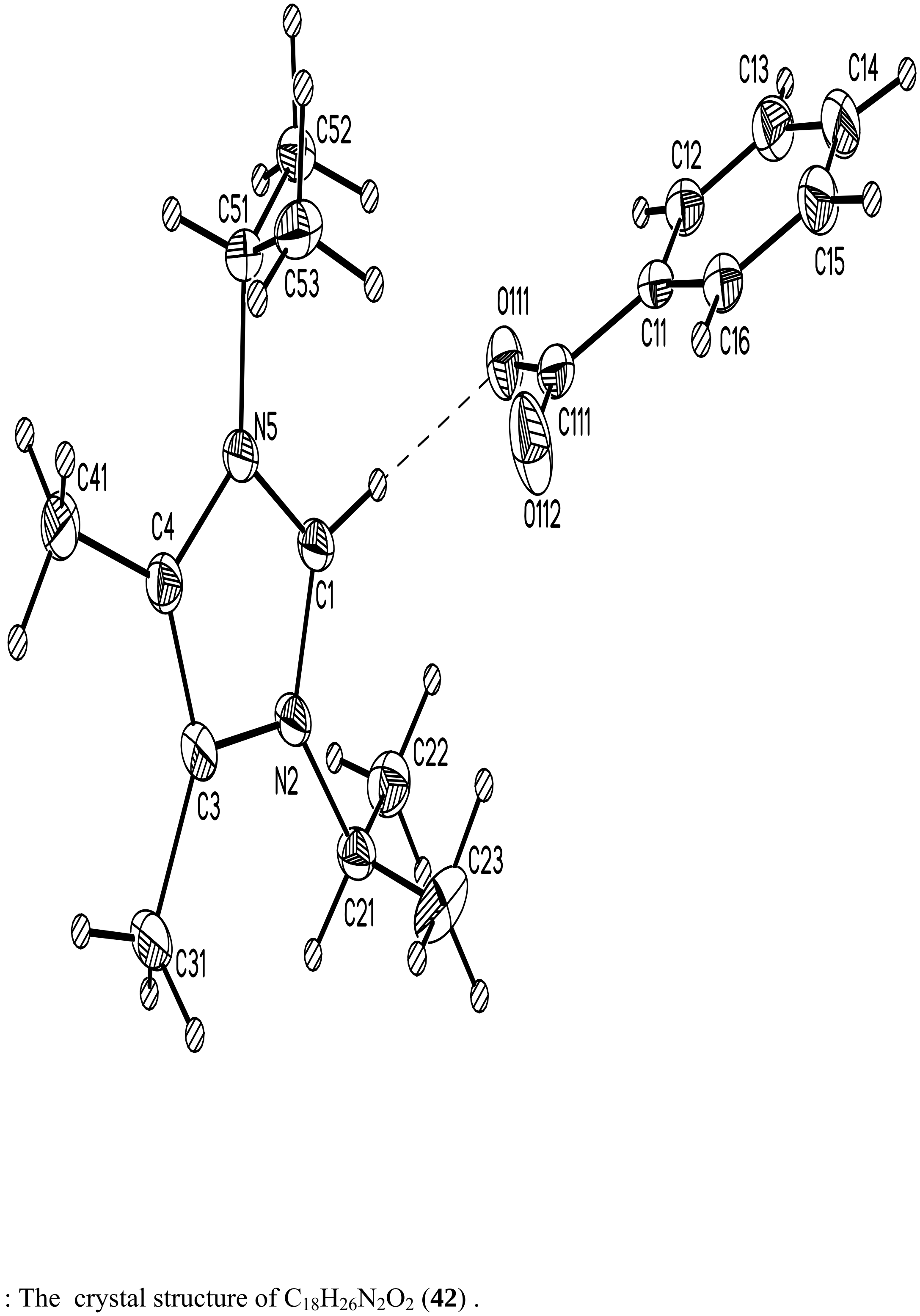
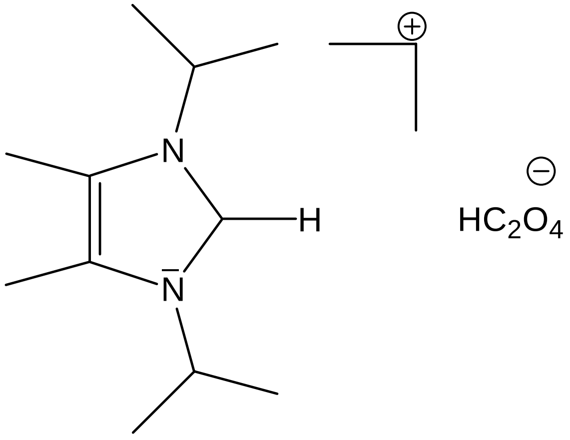


Fig. 16 : The crystal structure of $C_{18}H_{26}N_2O_2$ (**42**) .

3.4.4 Synthesis of 1,3-diisopropyl-4,5-dimethylimidazolium hydrogenoxalate

The reaction of **10c** with oxalic acid in Et_2O gave compound **43** as a stable solid in high yield.



43

The crystal structure indicates that the salt crystallizes in the monoclinic space group C2/c (fig.17, tab. 76-80) [76]. The crystal structure shows that the imidazole fragment is joined to the hydrogenoxalate ion through symmetrical hydrogen bridges [H—O(2A) 2.601, H—(O2) 2.601 Å; C1—H1—O2A 147.3, C1—H1—O2 147.3º]. The data for the hydrogenoxalate ion is in agreement with reported values [77].

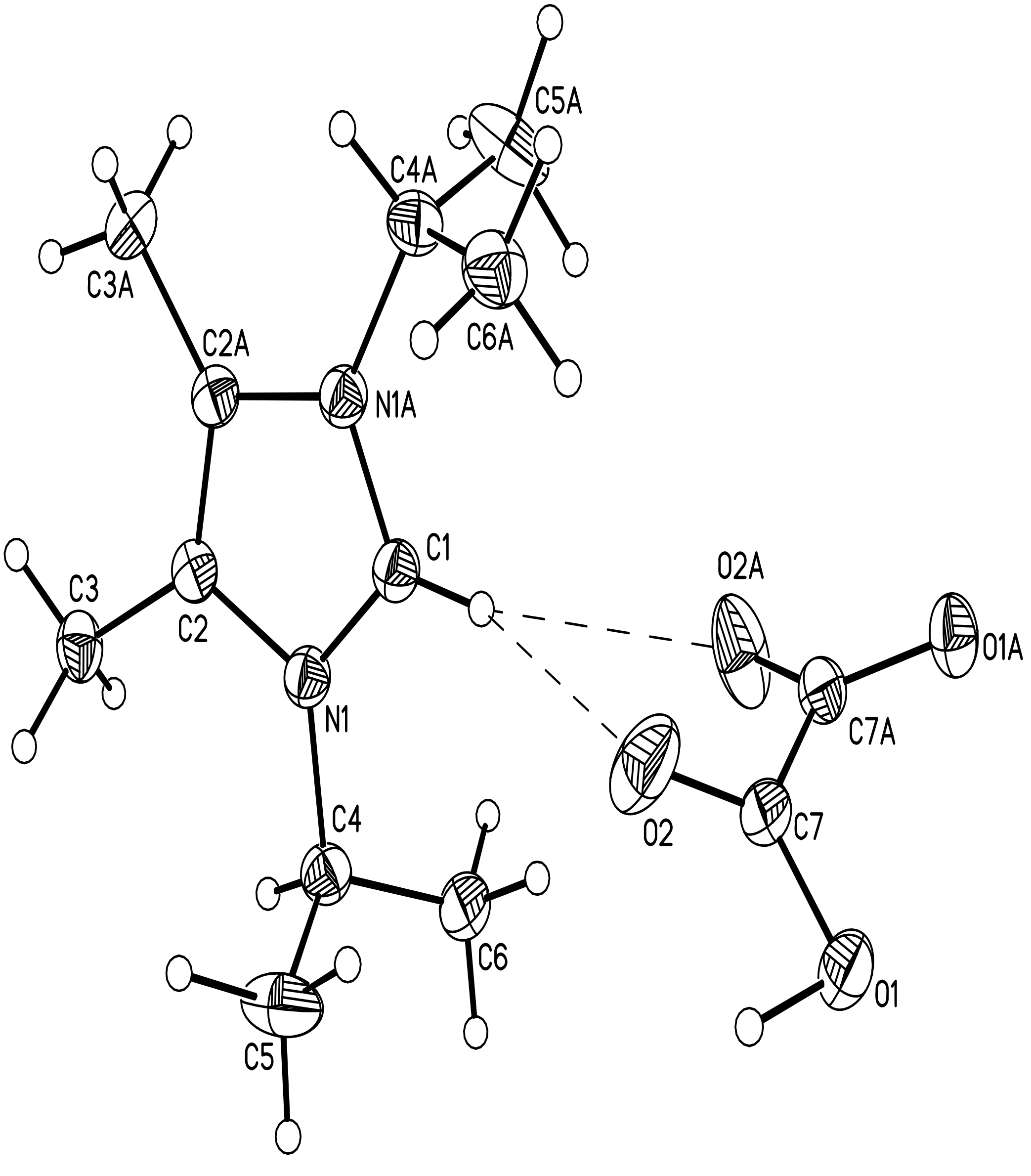
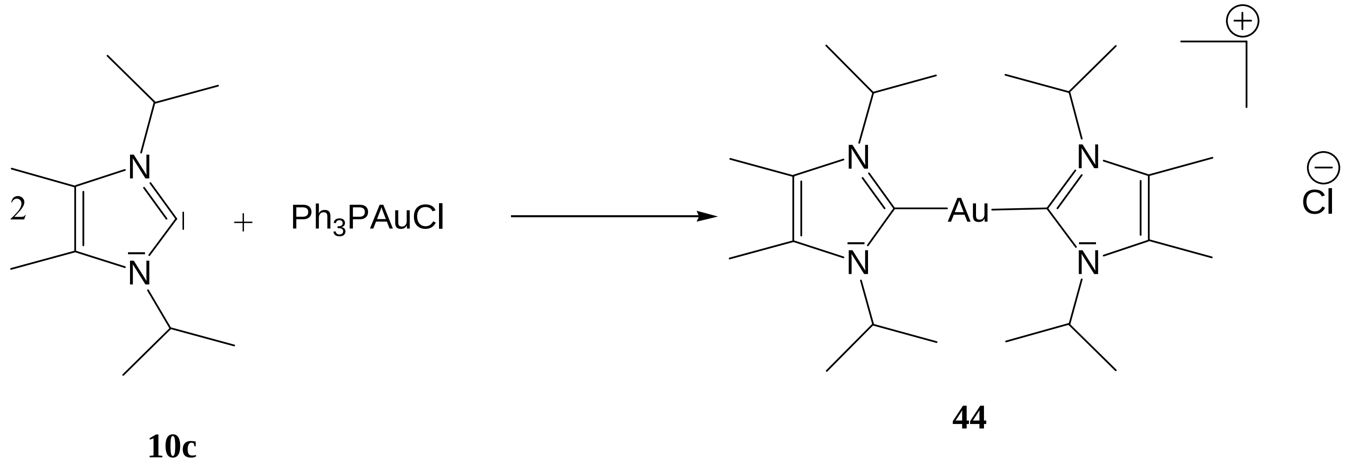


Fig. 17 : The crystal structure of $C_{11}H_{22}N_2O_4$ (**43**).

3.4.5 Synthesis of bis-(1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene)gold(I) chloride.

The reaction of **10c** with Ph_3PAuCl produced compound **44** as a stable solid in very high yield.



The gold carbene complex is very stable [78, 79]. The complex crystallizes in the orthorhombic space group P2(1)2(1)2(1). The gold atom is nearly linear linked with carbon atoms from the two imidazol rings. The deviation of the angle [C(11)—Au(1)—C(21) 177.1º] from 180º is to allow the gold atoms in neighboring cations to approach one another (fig. 18-19, tab. 81-85) [80]. The Au(1)—Au(1A) distance is [7.273 Å] and the bond angle Au(1)—Au(1A)—Au(1B) is [156.6 º].

The average Au—C distance is [2.0215(10) Å] which is very similar to Au(III)—CH_3 bonds [2.023 Å] [81], and longer than the distance observed in the compound where a single carbene is linked to the Au atom [1.9618(19) Å] [82].

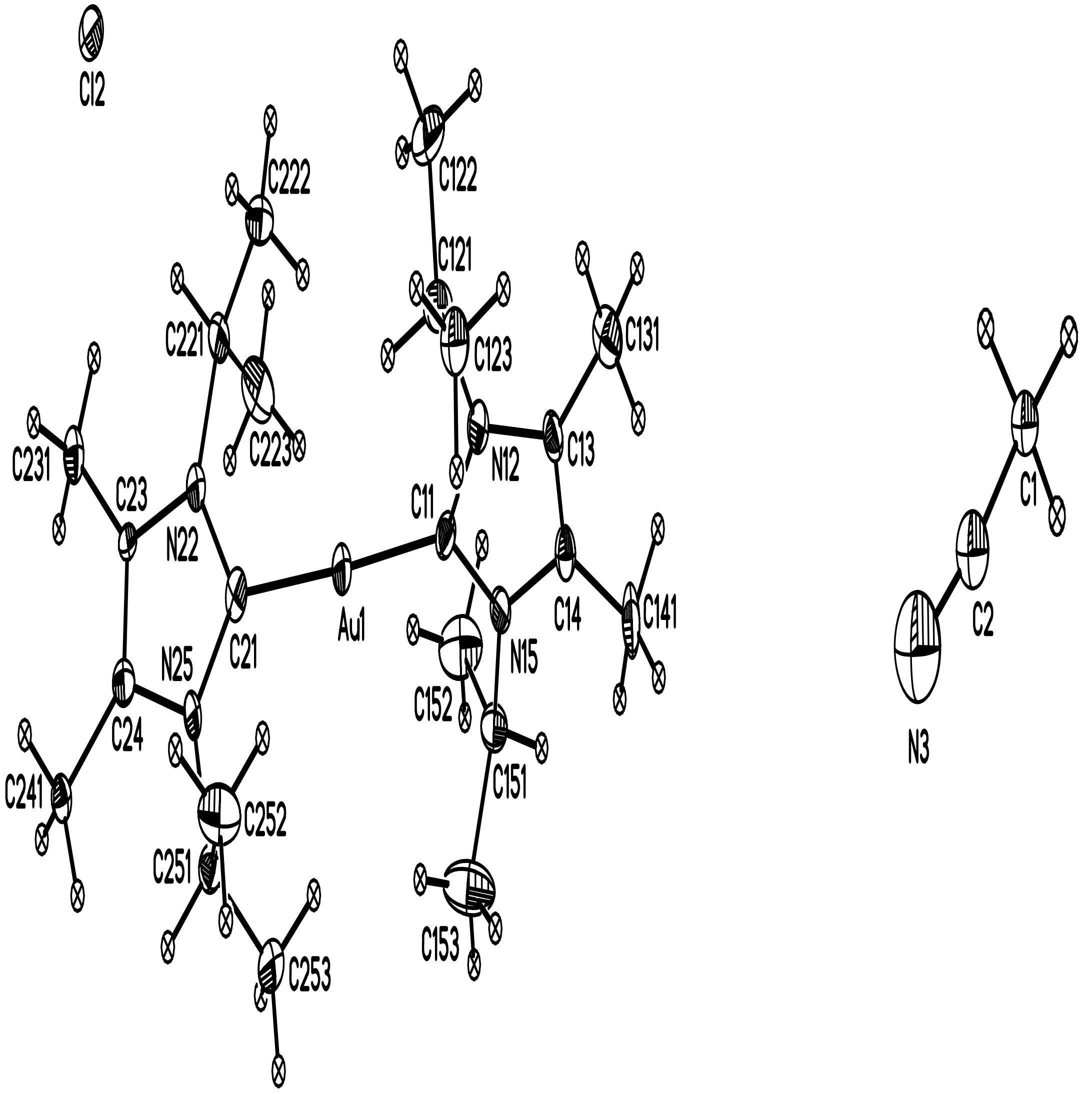


Fig. 18 : The crystal structure of $C_{22}H_{40}N_4ClAu$. CH_3CN (**44**).

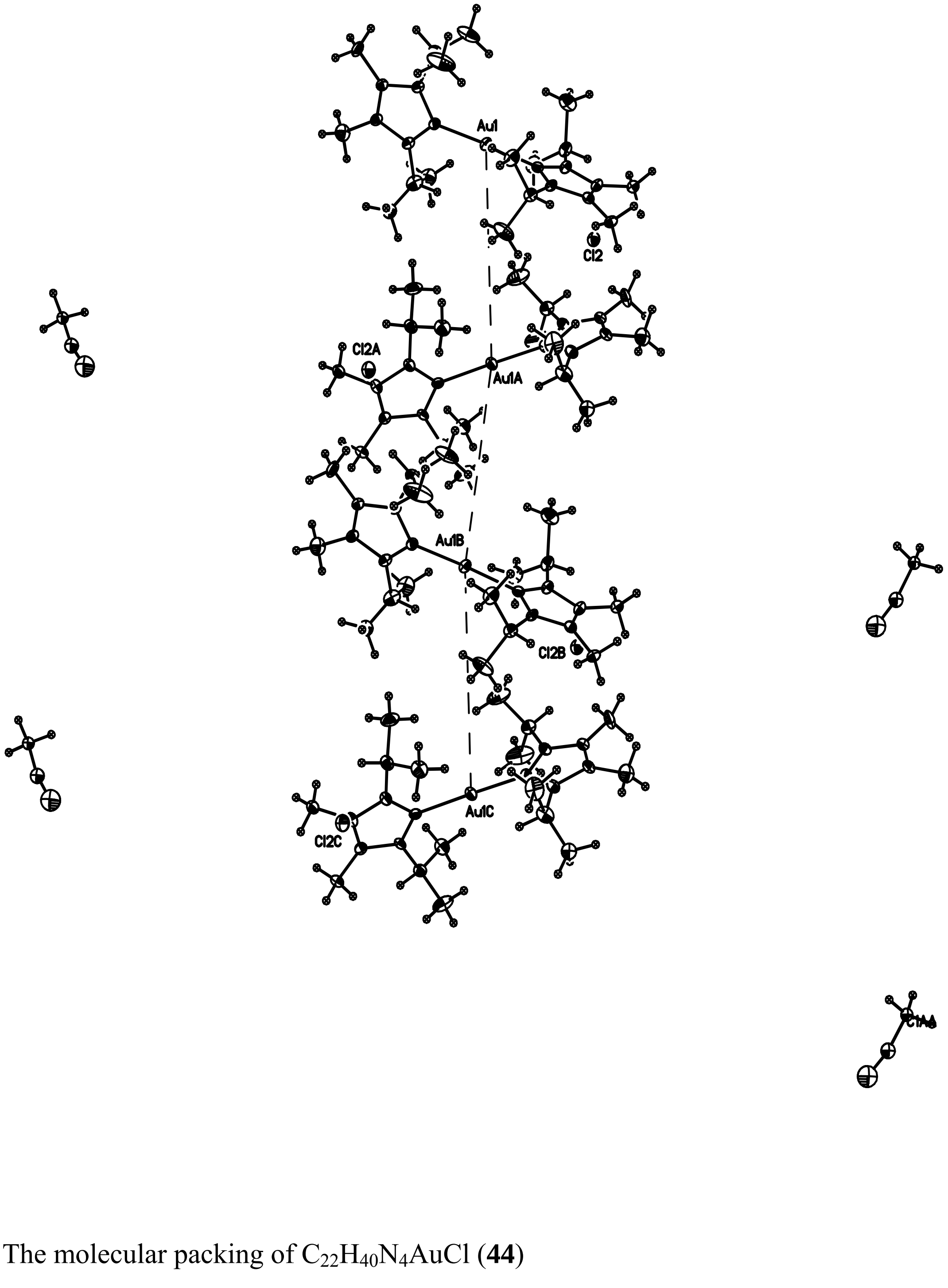
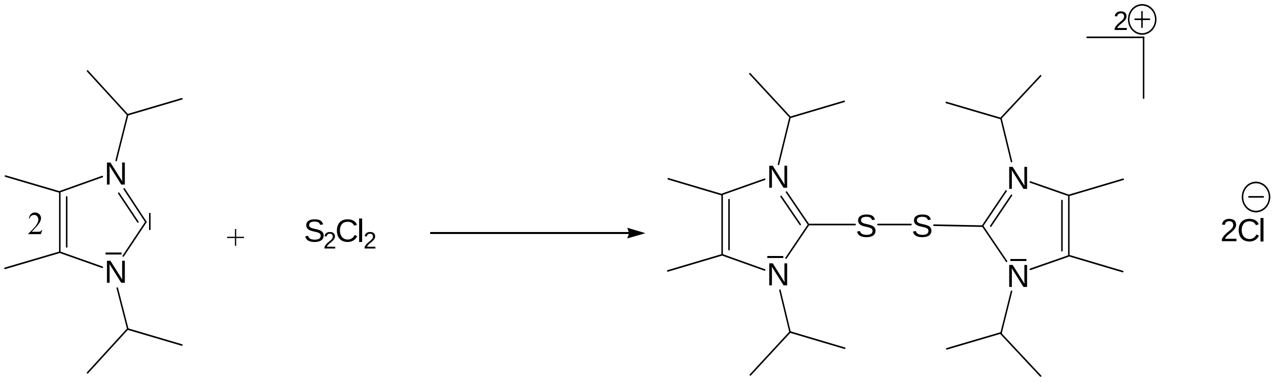


Fig. 19 : The molecular packing of $C_{22}H_{40}N_4AuCl$ (**44**)

3.4.6 Synthesis of bis(1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene)disulfide chloride.

The reaction of S_2Cl_2 with **10c** gave a stable crystalline solid **45** in very high yield.



10c **45**

A novel C—S bond has been found in the reaction of **10c** with S_2Cl_2. The crystal structure reveals that the compound crystallizes in the monoclinic space group P2(1)/n. The two imidazole fragments are bridged by S_2 (fig. 20, tab. 86-90). The C—S bond distances are [1.733(5), C(1)—S(4) 1.733(5), C(12)—S(3) 1.738(5) Å] [83] which are not significantly different and in agreement with the reported values [84].

The S—S bond distance was found to be [2.103 Å] which is longer than the S—S bond distance in orthorhombic sulfur [2.060 Å] [85] and other disulfide compounds [2.018 and 2.037 Å] [86, 87]. The S—S bond distance in disulfides is known to depend on both the electronegativity of the substituents attached to the sulfur atom and the dihedral angle. In disulfide groups the regular dihedral angle of about 90° corresponds to S—S bond distance of about 2.03 Å [87]. The C—S—S—C dihedral angle in **45** was found to be -72.75º, although values of dihedral angles outside the range 80-100º have been reported [88]. A small difference in the bond angles [C(1)—S(4)—S(3) 105.84(16), C(12)—S(3)—S(4) 101.11(17)º] has been observed which is also reported [87].

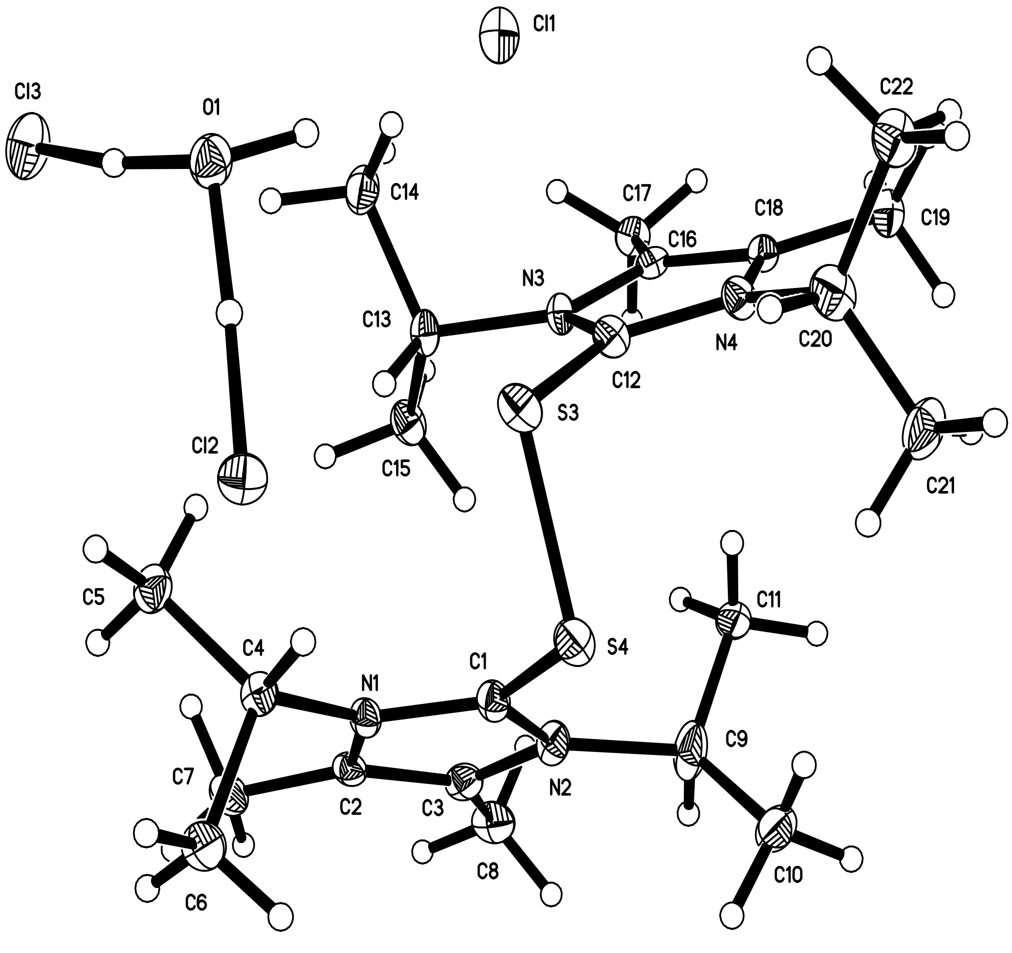
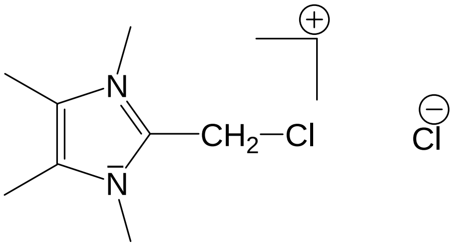


Fig 20 : The crystal structure of $C_{22}H_{40}N_4Cl_2S_2$. H_2O . HCl (**45**).

3.5 Reactions of 1,3,4,5-tetramethyl-2-methyleneimidazoline with halogen containing compounds.

3.5.1 Synthesis of 2-chloromethyl-1,2,3,4-tetramethylimidazolium chloride

The reaction of hexachloroethane with **17** produced compound **46** as a stable crystalline solid in high yield.



46

The use of elementary chlorine led to a mixture of products. Therefore organic chlorinating reagents were used for the synthesis of **46**. Compound **46** is obtained as an air stable salt in very high yield. The crystal structure of **46** (fig. 21, tab. 91-95) [89] showed isolated ions in the crystal, which is different to **25** (X = Br [89]; I [90]). The shortest interionic Cl—Cl distance is 4.064(2) Å observed for Cl(1)—Cl(2). The significant deviation of the coordination geometry at Cl(1) from the linearity [C (1)—Cl(1)—Cl(2) 148.8(2)º] gives little cause for the discussion of the *Van der Waals* interaction effect. The distances between cations [Cl(1)—Cl(1') 4.339(2) Å] and anions [Cl(2)—Cl(2') 5.496(2) Å] are in each case also outside of *Van der Waals* range. The structure of the cation lies in the standard range.

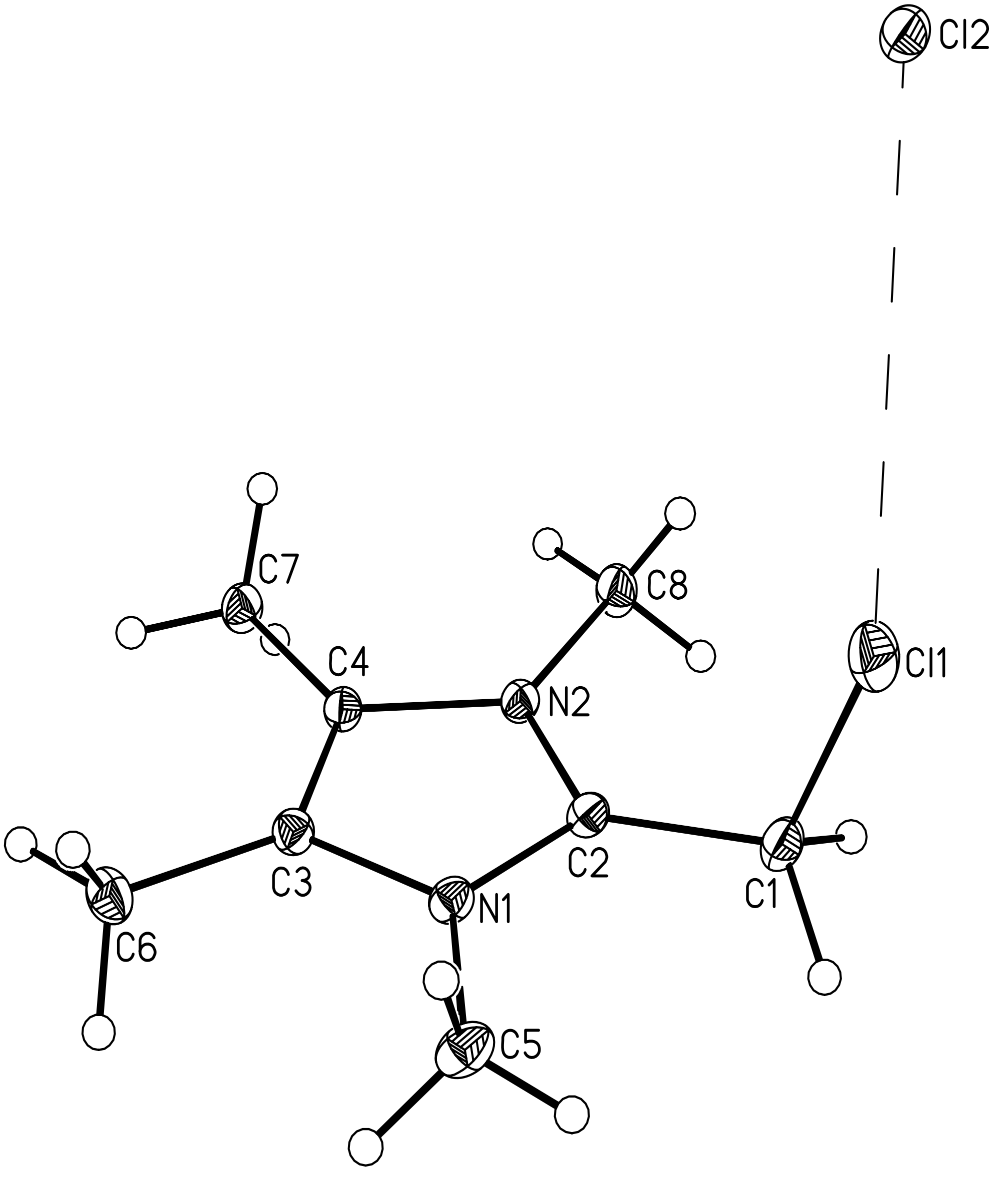
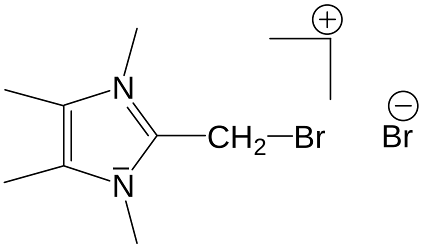


Fig. 21 : The crystal structure of $C_8H_{14}N_4Cl_2$ (**46**).

3.5.2 Synthesis of 2-bromomethyl-1,2,3,4-tetramethylimidazolium bromide

The reaction of Br_2 with **17** produced compound **47** as a stable crystalline solid in very high yield.



47

Recrystallization from dioxane/toluene affords solvent free single crystals suitable for X-ray structural analysis. The salt **47** crystallizes in the rhombohedra space group R-3 with 18 formula units in the unit cell (fig. 22, tab. 96-100) [89]. Here the bromine atoms of the cations from the trigonal anti prism axis lie in the crystallographic triple axis of the rotation. The Br—Br distance within the associated triangle surfaces [Br(2)—Br(2a) 3.767(7) Å] are slightly longer than those between the bromine atoms of neighbouring surfaces [Br(2)—Br(2c) 3.667(8) Å]. Both values are within the *Van der Waals* range (ca. 3.7 Å), and so we can say that the Br6 polyhedra may be held together by weak interactions [Br(2)—Br (2c)—Br(2a) 59.1º].

In contrast to this, the shortest interionic Br—Br distance is clearly outside of the *Van der Waals* range [Br(1)—Br(2) 4.484(7) Å]. The five membered ring fragments are oriented away on side of the triangle surfaces and form with this cavities, whose shape is similar to the building principle of the Calixarenes.

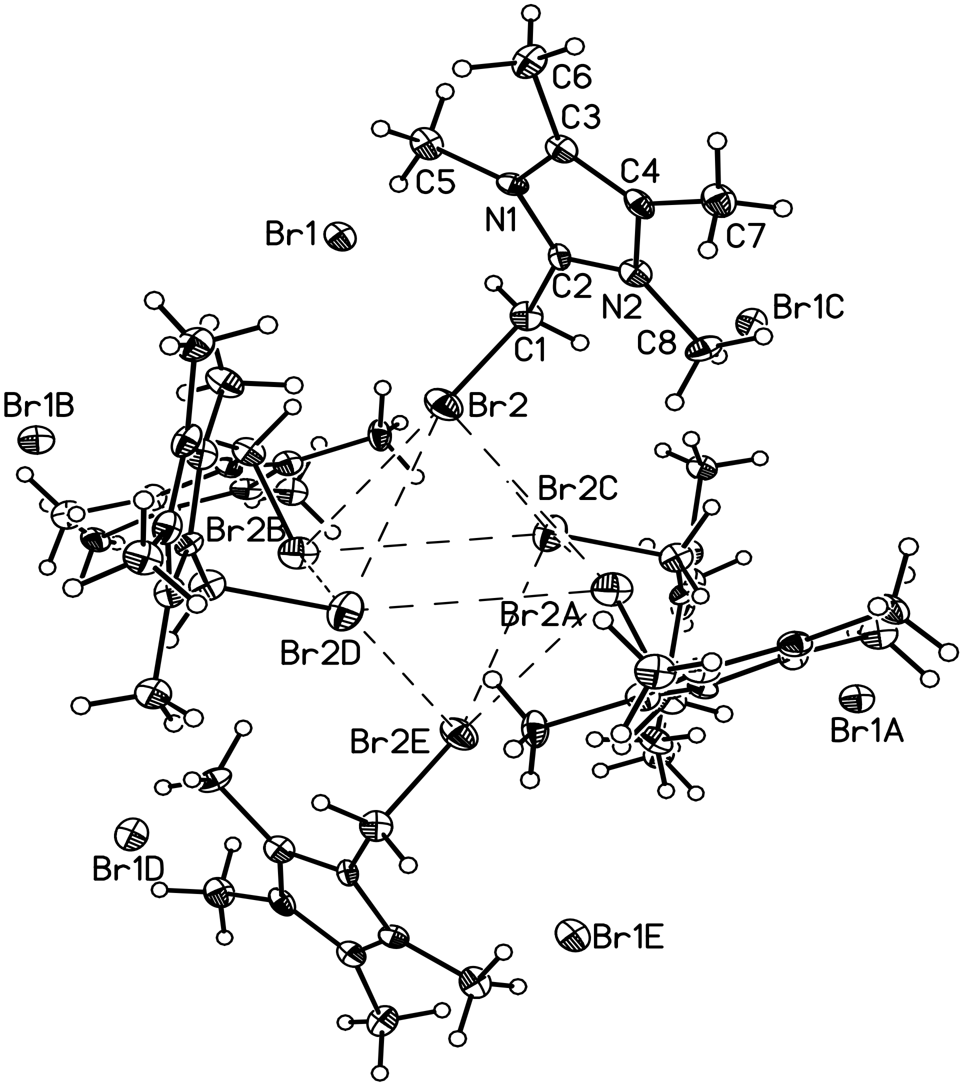
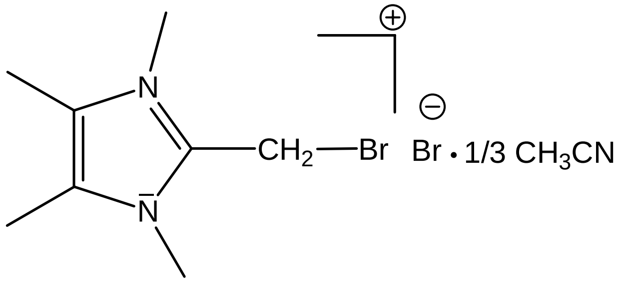
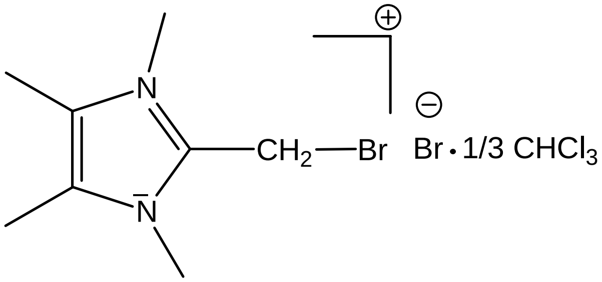


Fig. 22 : The crystal structure of $C_8H_{14}N_2Br_2$ (**47**).

After recrystallization of **47** from other solvents, the presence of solvent molecules in the crystal structure was observed. In the case of acetonitrile and chloroform, the cavities formed between the three five memebered fragments and associated Br_3 triangle surfaces are occupied by solvent molecules (fig. 23, 24) [89]. Here neither a change of the space group nor any other significant change of the unit cell parameters take place. The unit cell parameters do not actually correspond to the solvent molecule of the associated atoms of **48** and **49**. Weak halogen to halogen interaction effects are observed [**48** Br(2)—Br(2a) 3.75(1); Br(2)—Br(2c) 3.65(1); **49** Br(2)—Br(2a) 3.76 (1); Br(2)—Br(2c) 3.68(1) Å].



48



49

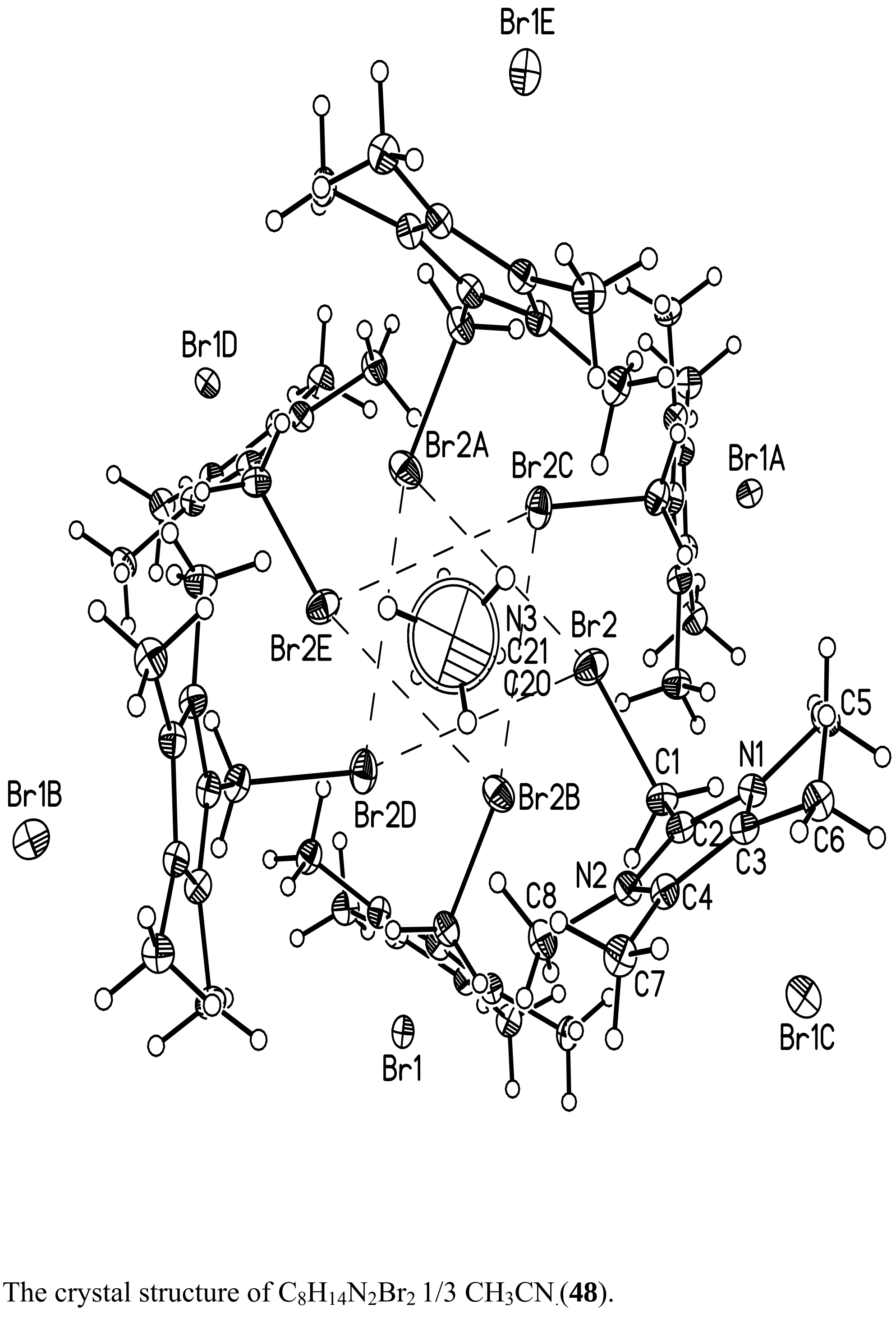


Fig 23 : The crystal structure of $C_8H_{14}N_2Br_2$ 1/3 CH_3CN.(**48**).

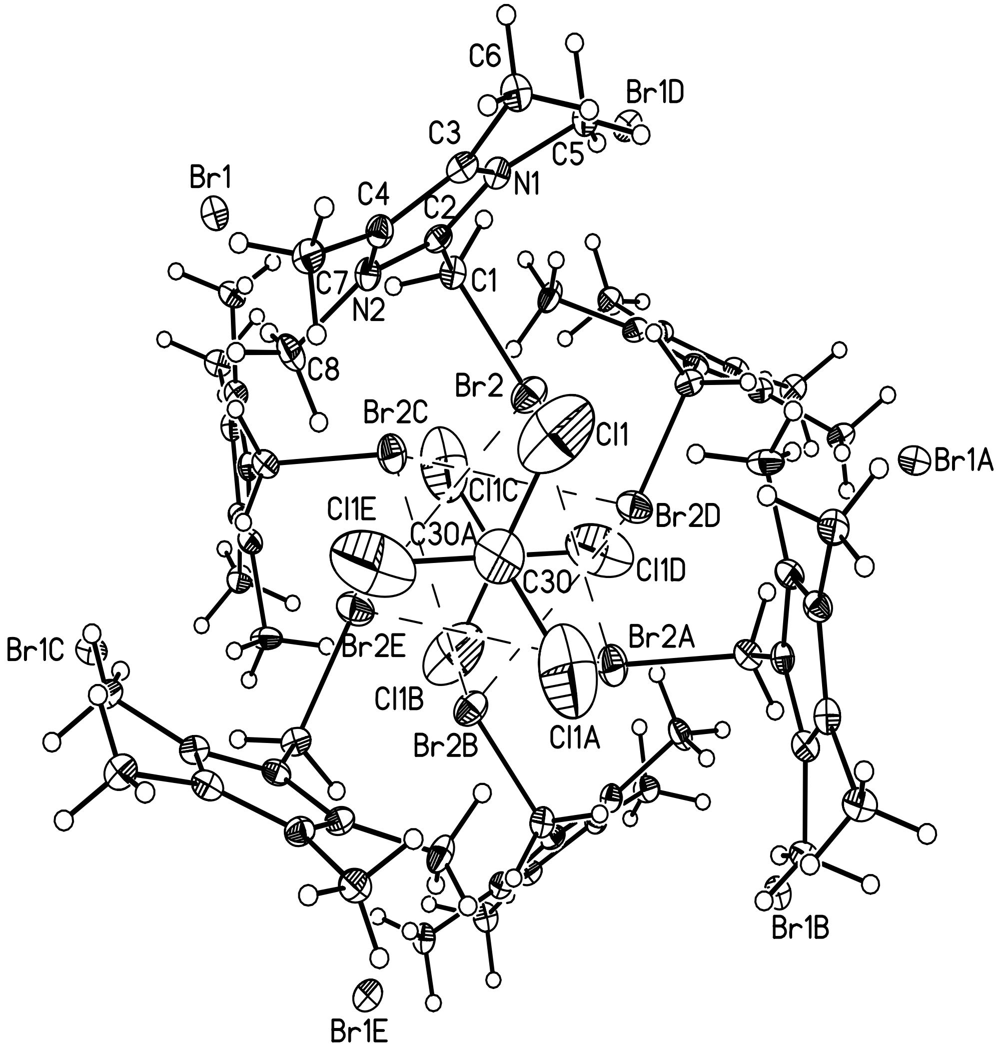
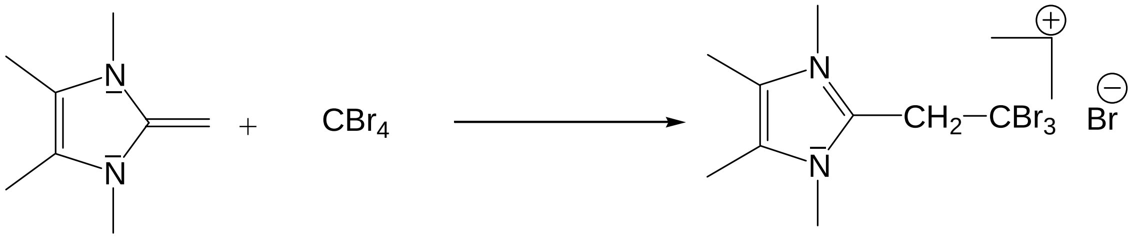


Fig. 24 : The crystal structure of $C_8H_{14}N_2Br_2 \cdot 1/3\ CHCl_3$ (**49**).

3.6 Nucleophilic reactions of 1,3,4,5-tetramethyl-2-methyleneimidazoline.

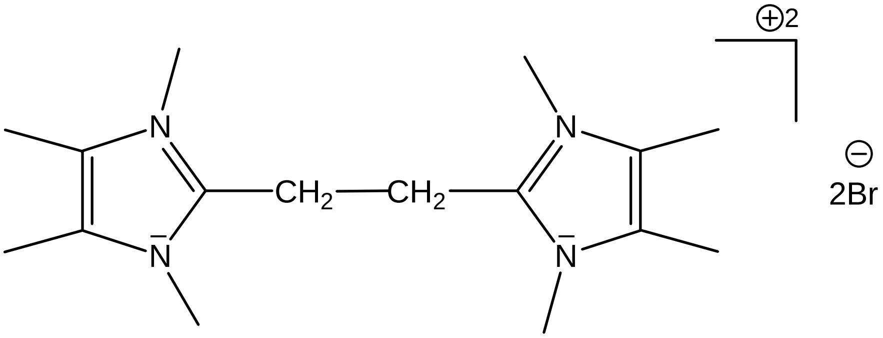
3.6.1 Synthesis of 2-(2,2,2-tribromoethyl)-1,3,4,5-tetramethylimidazolium bromide

Compound **50** was obtained from the reaction of **17** with CBr_4.



17 **50**

The crystal structure reveals that the compound crystallizes in the monoclinic space group P2(1)/n. The crystal structure of **50** (fig. 25, tab. 101-105) indicates a molecular structure in which the bromide ions are linked to the cations through weak halogen to halogen bonds generating a near linear coordination geometry at the central bromine atom [Br(2)—Br(4) 3.426 Å; C(1)—Br(4)—Br(2) 172.7°]. The C—Br bond distances are in excellent agreement with each other, their average value being 1.953 Å. The C(2)— C(1)—Br bond angles are also close to each other with average being [110.2 °] [91]. The crystals contain also the dimeric compound **51** (fig. 26). The Br(5)—Br(1) distance is [3.230 Å] which is smaller than the Br(2)—Br(4) bond distance and a near linear geometry at the central bromine atom [C(1)—Br(1)—Br(5) 173.9°] is observed.



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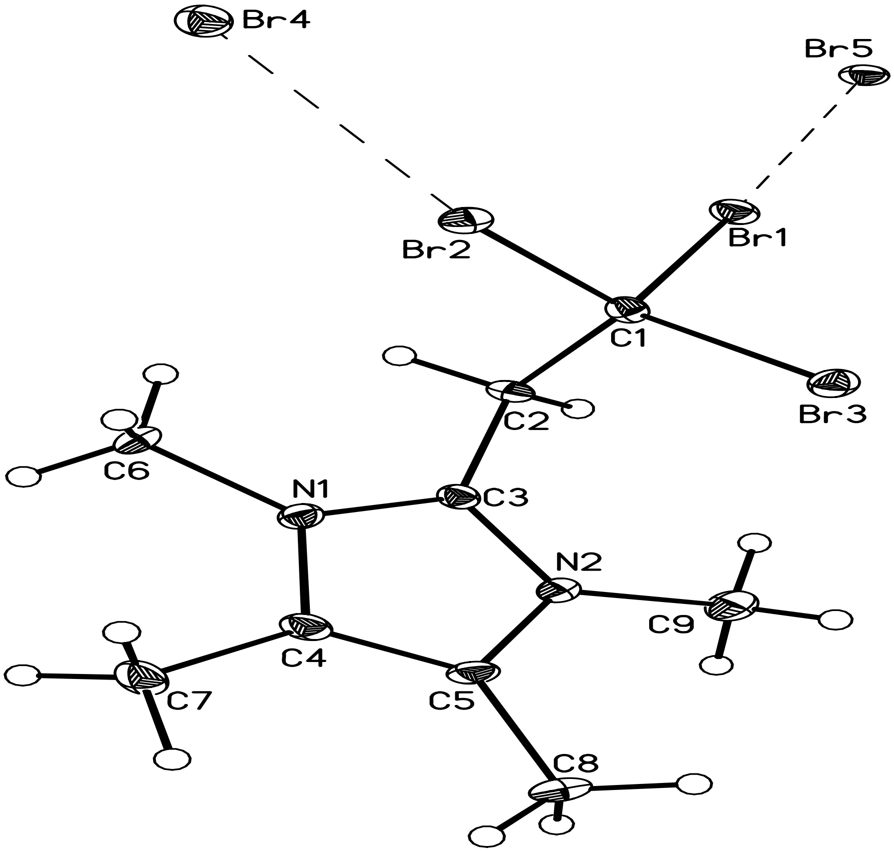


Fig. 25 : The crystal structure of $C_9H_{14}N_2Br_4$ (**50**).

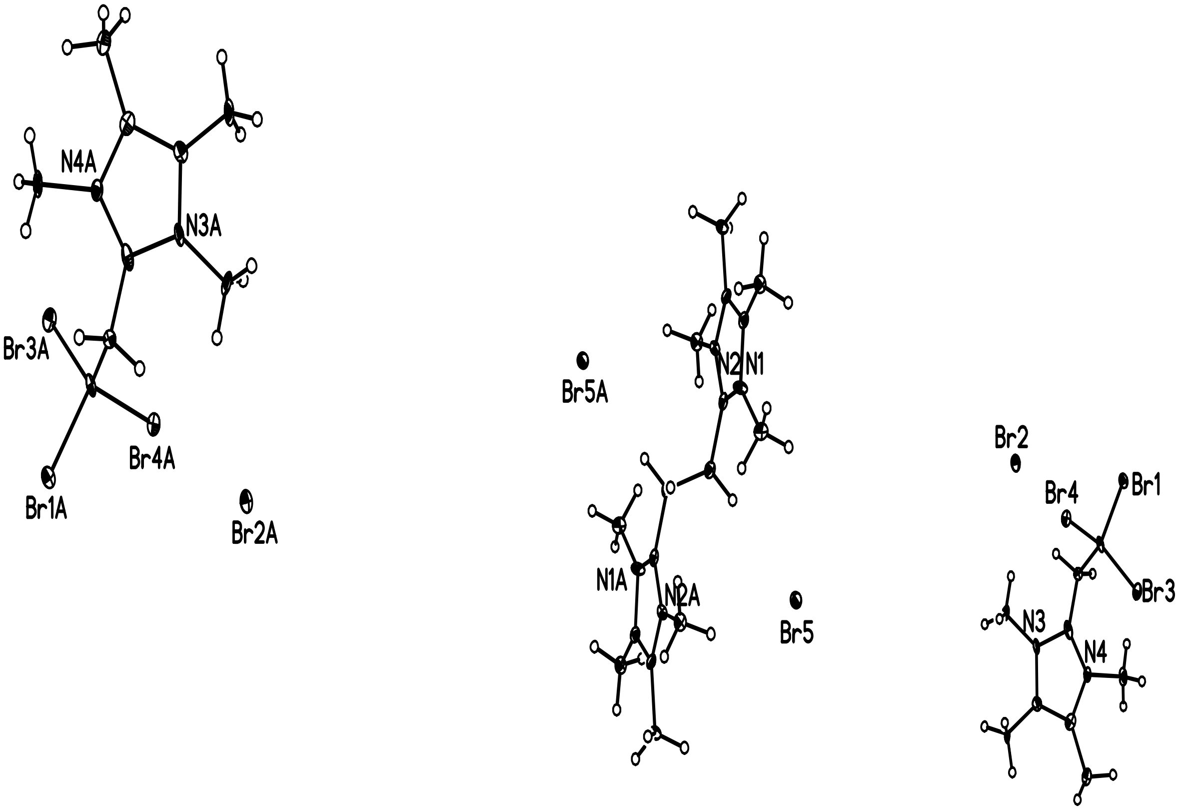
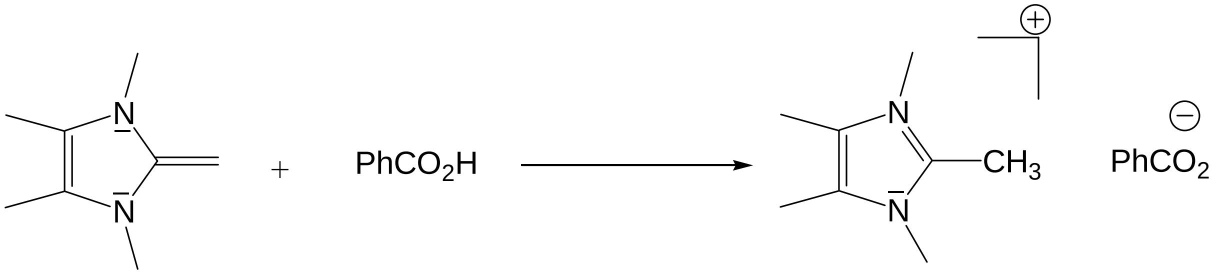


Fig. 26 : The crystal structure of $C_9H_{14}N_2Br_4$(**50**) and the dimeric compound.

3.6.2 Synthesis of 1,2,3,4,5-pentamethylimidazolium benzoate.

The reaction of benzoic acid with **17** gave compound **52** as a stable crystalline solid in high yield.



17

52

The crystal structure (fig. 27, tab. 106-110) [92] shows that the salt crystallizes in the tetragonal space group P4(1)2(1)2. For the pentamethylimidazolium cation both bond distances and angles are in agreement with the reported values [32]. The imidazole fragments joined to the benzoate ion through a hydrogen bridge [H(4A)—O(1) 2.36, H(7c)—O(2) 2.36 Å; C(4)—H(4A)—O(1) 168, C(7)—H(7C)—O(2) 176º]. The crystal structure for the benzoate ion is in agreement with documented results [75]. For the benzoate fragment itself the O(2)—C(15)—O(1) bond angle is [126.3(5)º] and the C—O bond distances are in good agreement with each other [C(15)—O(1) 1.254, and C(15)—O(2) 1.216(6) Å].

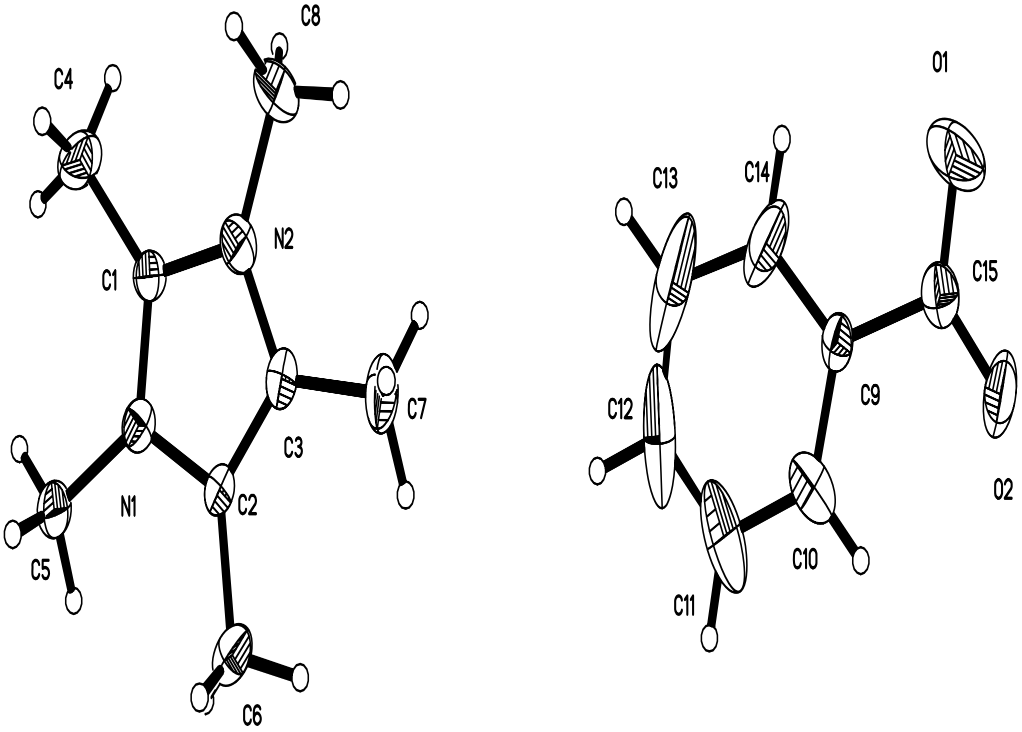
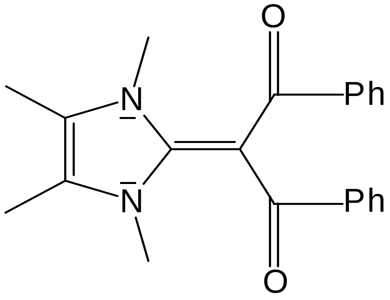


Fig. 27 : The crystal structure of $C_{15}H_{20}N_2O_2$ (**52**).

3.6.3 Synthesis of 1,3-diphenyl-2-(1,3,4,5-tetramethyl-2,3-dihydroimidazole-2-ylidene) propane-1,3-dione

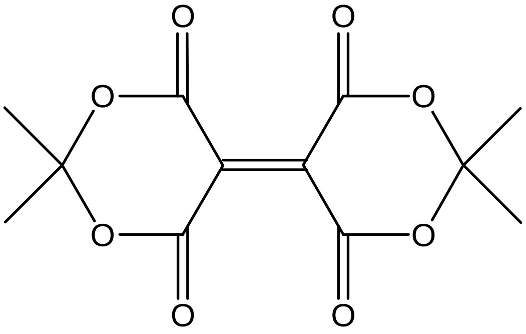
The reaction of benzoyl fluoride with **17** gave compound **53** as a stable compound in high yield.



53

The crystal structure reveals that the compound crystallizes in the monoclinic space group C2/c. Comparing the exocyclic C═C bond in **17** [1.357(3) Å] [31] with **53**, a relative elongation of the olefin C═C bond [C(1)—C(5) 1.467(3) Å] [93] is observed. The formally single bonds C(5)—C(6) and C(5)—C(6A) [1.432 Å] undergo significant shortening from the value of 1.464(18) Å reported for (C═)C_{sp2}—C_{sp2}—(C═O) conjugated bonds [84,94]. The two C═O bonds [1.251(2) Å] are elongated with respect to the value for conjugated (C═C)—C═O bonds [1.222(10) Å] [94]. The average two N—C(1) bond distances [1.344(2) Å] is slightly shorter than that reported ones for C═C—N_{sp2} [1.355(14) Å] [82] and shorter than the values reported for compound **17**.

The ^{13}C-NMR spectrum shows a downfield shift for the signal which corresponds to C═O (δ = 190.1 ppm). The C2 signal appears more downfield (δ = 148.6 ppm) than in the symmetrical olefin **54** (δ = 133.4 ppm) [95] which confirms the influence of the C(O)Ph substituents.



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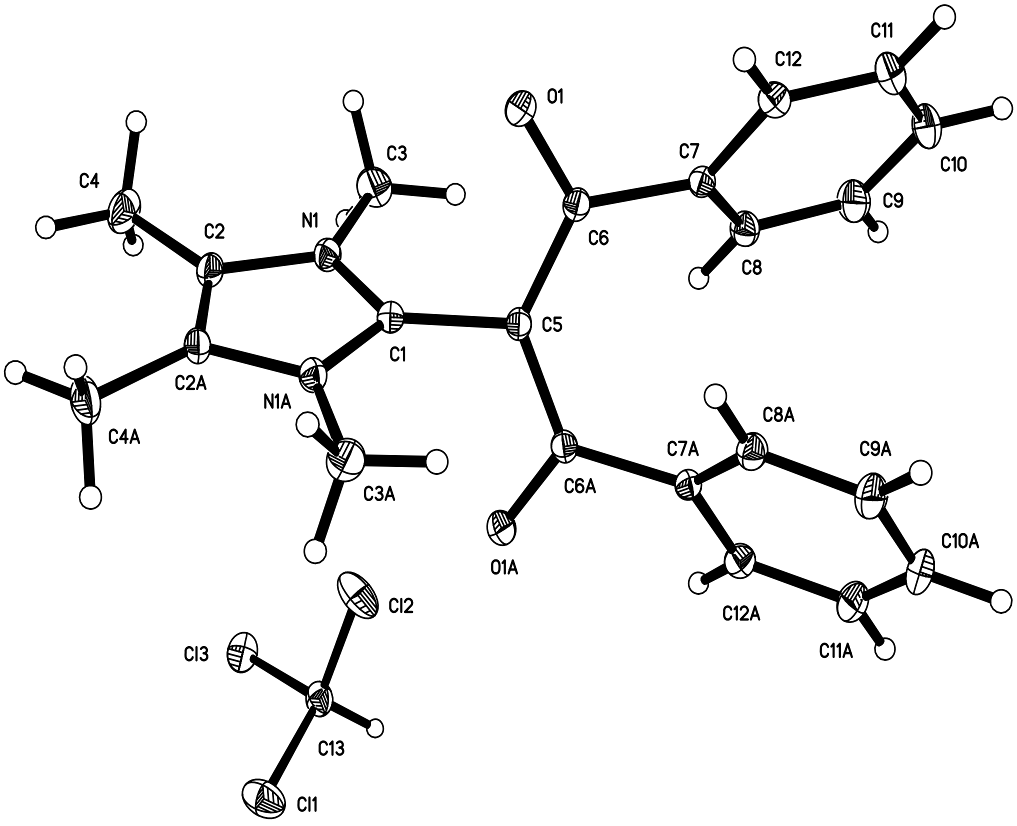
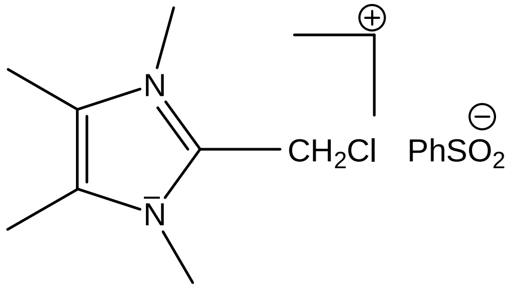


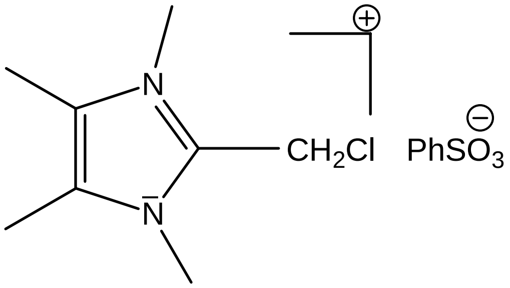
Fig .28 : The crystal structure of $C_{22}H_{22}N_2O_2 . CHCl_3$ (**53**).

3.6.4 Crystal structure analysis of 2-chloromethyl-1,,3,4,5-tetramethyl-2-imidazolium benzenesulfonate.

The reaction of benzenesulfonyl chloride with **17** produced compound **55** as a stable solid in high yield. Crystallization of compound **55** led to compound **56** due to the effect of wet solvent or air.



55



56

The salt crystallizes in the orthorhombic space group P2(1)2(1)2(1) [96]. The bond distances and angles within the sulfonate group itself agree well with those in other aromatic sulfonates (fig. 29, tab. 116-120) [97]. The average phenyl C—C bond distance is 1.3785 Å with extreme values of 1.359 and 1.396 Å, the longer bonds are on the sulfonate side of the ring [C(1)— C(6) 1.396(4), C(1)— C(2) 1.387(4) Å]. The C—S bond distance is 1.776(3) Å, no significant difference from the parent compound is observed [98]. The S—O bond distances are in excellent agreement with each other and their average value being 1.439 Å. The C— S—O bond angles are also close to each other with the average being 105.65°, while the average of the

O—S—O bond angles is 113º. The C(17)—Cl(1) bond distance is 1.808 Å which is similar to compound **46** .

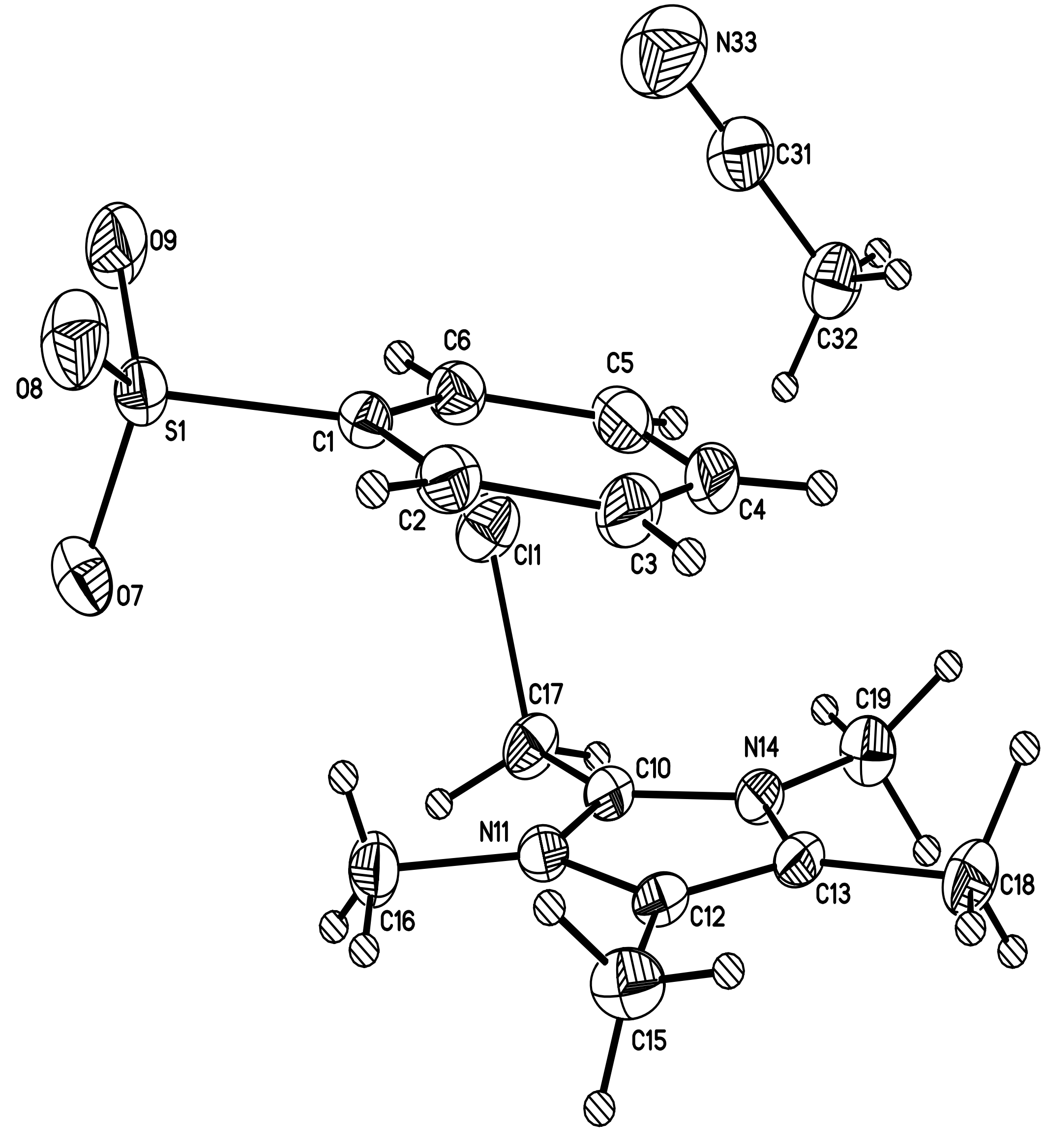


Fig. 29 : The crystal structure of $C_{14}H_{19}N_2SO_3$ (**55**)

4 Experimental Section

4.1 Methods of analysis

4.1.1 Elemental analysis

The elemental analyses were measured by an elemental analyzer from Carlo Erba Company, Model 1106.

The accuracy was attached

Carbon:	± 0,3%
Hydrogen:	± 0,3%
Nitrogen:	± 0,3%
Chlorine:	± 0,3%

4.1.2 Melting point determination

The melting point device is from Büchi Company, type Büchi 510. All melting points are not corrected.

4.1.3 Mass spectra

The EI-Mass spectra were acquired on a Finnigan TQS 70, by 70 eV at 200 °C. The FAB-Mass spectra were acquired on a Finnigan TQS 70, by 70 eV in Nitrobenzylalcohol-Matrix at 30°C. Instrument modified by AMD and reported as mass / charge (*m/z*).

4.1.4 NMR spectra

The high resolution NMR spectra were acquired by Bruker DRX 250 NMR spectrometer operating at ^{1}H: 250,13 MHz; ^{13}C: 62,90 MHz; ^{31}P: 101,20 MHz; ^{19}F: 235,34 MHz. The spectra were measured relative to TMS (^{1}H, ^{13}C); C_6F_6 (^{19}F); 85% H_3PO_4 (^{31}P) as internal standard.

The following abbreviations were used in the description of the spectra:

s	Singlet
d	Doublet
t	Triplet
q	Quartet
m	Multiplet

4.1.5 IR spectra

The FT-IR spectra were acquired by a Bruker IFS 25 IR spectrometer. The measuring range was from 4000 to 225 cm^{-1}. The sample preparations were taken place in the form of KBr pressing or films.

4.1.6 Crystal structure analyses

The crystals were mounted on a glass fiber and transferred to a P4 Siemens diffractometer (**26, 27, 29, 30, 32, 33, 34, 35, 36, 38, 45, 46, 47, 48, 49, 53, 50**), a Nonius Kappa CCD diffractometer (**28, 42, 44, 52**), and a Stoe IPDS diffractometer (**31, 40, 41**) using graphite-monochromated Mo–K_α radiation. The lattice constants were determined by 25 precisely centered high-angle reflections and refined by least-squares methods. The structures were solved by direct methods and refined by full-matrix least-squares on F^2 using SHELXTL-93 or SHELXTL-97.

4.2 General comments

All experiments were carried out under an atmosphere of argon.

4.3 Solvents

The solvents needed for the reactions were dried by conventional procedures and were stored over molecular sieves under argon.

4.4 Starting materials

1,3-diisopropyl-4,5-dimethyl-2-thione	[9c]
1,3-diisopropyl-4,5-dimethyl-2-ylidene	[10c]
1,3,4,5-tetrametyl-2-methyleneimidazoline	[17]

4.5 Preparation of the Compounds

4.5.1 Preparation of 2-chloro-1,3-diisopropyl-4,5-dimethylimidazolium chloride (**26**).

1.863 g (7.869 mmol) of C_2Cl_6 was added to a solution of 1.422 g (7.88 mmol) of **10c** in Et_2O at -10 °C. After stirring for 1 h at room temperature, the precipitate was filtered off, washed with Et_2O (2 x 20 ml) and dried in vacuo. Yield after recrystallization from CH_2Cl_2 / Et_2O : 1.89 g (96 %).

^{1}H-NMR (CD_2Cl_2):

$\delta = 1.62$ [d, 12 H,1,3- CH*Me*$_2$ $^3J = 6.9$ Hz]

$\delta = 2.39$ [s, 6 H, 4,5-Me]

$\delta = 4.90$ [sept, 2 H, C*H*Me$_2$]

^{13}C-NMR (CD_2Cl_2):

$\delta = 10.5$ [4,5-Me]

$\delta = 20.8$ [1,3-CH*Me*$_2$]

$\delta = 53.3$ [1,3-*C*HMe$_2$]

$\delta = 128.6$ [C4,5]

$\delta = 127.4$ [C2]

Elemental analysis for $C_{11}H_{20}Cl_2N_2$ (M = 251.18 g / mol).

Found (cal) : C 52.28 (52.59), H 8.51 (8.03), N 10.99 (11 .15) %.

MS (FAB) : m/z (%) = 215 [100, M^+], 173 [12, M^+ - C_3H_7], 136 [12, M^+ -Cl, - C_3H_7], and further fragments.

4.5.2 Preparation of 2-bromo-1,3-diisopropyl-4,5-dimethylimidazolium bromide (**27**)

0.88 g (5.55 mmol) of Br_2 was added to a solution of 1.0 g (5.55 mmol) of **10c** in 30 ml of THF at 0 °C. After stirring for 2 h at room temperature, the precipitate was filtered off, washed with THF (3 x 10 ml) and dried in vacuo. Yield after recrystallization from CH_2Cl_2/ Et_2O 1.56 g (82 %).

^{1}H-NMR (CD_2Cl_2):

δ = 1.60	[d, 12 H,1,3- CH*Me₂* $^3J = 6.9$ Hz]
δ = 2.37	[s, 6 H, 4,5-Me]
δ = 4.9	[sept, 2 H, C*H*Me$_2$]

^{13}C-NMR (CD_2Cl_2):

δ = 10.9	[4,5-Me]
δ = 20.9	[1,3-CH*Me*$_2$]
δ = 53.2	[1,3-*C*HMe$_2$]
δ = 129.0	[C4,5]
Not observed	[C2]

Elemental analysis for $C_{11}H_{20}Br_2N_2$ (M = 340.0 g / mol).

Found (cal.) : C 38.64 (cal. 38.85), H 5.76 (5.93), N 8.16 (8.24) %.

MS (FAB) : m/z (%) = 259 [100, M^+], 216 [16, M^+ - C_3H_7], 273 [16, M^+, -2 (C_3H_7)], and further fragments.

4.5.3 Preparation of 2-iodo-1,3-diisopropyl-4,5-dimethylimidazolium iodide (**29**).

0.88 g (3.44 mmol) of I_2 was added to a solution of 0.62 g (3.44 mmol) of **10c** in 30 ml of THF at 0 °C. After stirring for 2 h at room temperature, the precipitate was filtered off, washed with THF (3 x 10 ml) and dried in vacuo. Yield after recrystallization from CH_2Cl_2 / Et_2O : 1.2 g (80 %).

^{1}H-NMR (CD_2Cl_2):

$\delta = 1.59$	[d, 12 H,1,3- CH*Me₂* $^3J = 6.9$ Hz]
$\delta = 2.38$	[s, 6 H, 4,5-Me]
$\delta = 4.88$	[sept, 2 H, C*H*Me$_2$]

^{13}C-NMR (CD_2Cl_2):

$\delta = 10.7$	[4,5-Me]
$\delta = 21.1$	[1,3-CH*Me*$_2$]
$\delta = 53.5$	[1,3-*C*HMe$_2$]
$\delta = 129.4$	[C4,5]
$\delta = 128.3$	[C2]

Elemental analysis for $C_{11}H_{20}I_2N_2$ (M = 433.8 g / mol).

Found (cal.) : C 30.24 (30.43), H 4.55 (4.61), N 6.32 (6.45) %.

MS (FAB) : m/z (%) = 307 [100, M^+], 265 [18, M^+ - C_3H_7], 242 [14, M^+ -2 (C_3H_7)], 181 [12, Im-H^+], 136 [12, -I, - C_3H_7], and further fragments.

4.5.4 Preparation of bis (2-iodo-1,3-diisopropyl-4,5-dimethylimidazolium bromide) 1-bromo-2-iodotetrafluoroethane adduct (**30**)

2.97 g (9.66 mmol) of 1-Bromo-2-iodotetraflouroethane were added to a solution of 0.58 g (3.22 mmol) of **10c** in 30 Et_2O at -78 °C. After stirring for 2 h at room temperature, the precipitate was filtered off, washed with Et_2O (2 x 10 ml) and dried in vacuo. Yield after recrystallization from CH_2Cl_2 / Et_2O : 3.13 g (90 %).

^{1}H-NMR (CD_2Cl_2):

$\delta = 1.58$	[d, 24 H,1,3- CH*Me₂* $^3J = 7.2$ Hz]
$\delta = 2.25$	[s, 12 H, 4,5-Me]
$\delta = 5.27$	[sept, 4 H, C*H*Me$_2$]

^{13}C-NMR (CD_2Cl_2):

$\delta = 11.5$	[4,5-Me]
$\delta = 21.6$	[1,3-CH*Me*$_2$]
$\delta = 55.1$	[1,3-*C*HMe$_2$]
$\delta = 128.6$	[C4,5]
Not observed	[C2]
Not observed	[CF2]

Elemental analysis for $C_{24}H_{40}Br_3F_4I_3N_4$ (M = 1081.04 g / mol).

Found (cal.) : C 26.65 (26.67), H 3.47 (3.73), N 5.22 (5.18) %.

MS (FAB) : m/z (%) = 307 [100, M^+], 265 [12, M^+ - C_3H_7], 242 [12, M^+ -2 (C_3H_7)], 181 [16, Im-H^+], 136 [16, -I, -C_3H_7], and further fragments.

4.5.5 Preparation of 2-chloro-1,3-diisopropyl-4,5-dimethylimidazolium dichlorophosphate (**31**).

0.62 g (4.0 mmol) of $POCl_3$ was added to a solution of 1.01 g (4.0 mmol) of **26** in 20 ml of dichloromethane. The resulting mixture was refluxed for 6 h and evaporated to dryness. The resulting pale yellow solid was dissolved in 2 ml of $POCl_3$, and the solution was saturated with gaseous Et_2O over period of 5 d. The formed crystals were collected and dried in vacuo. Yield 54 mg (27 %)

^{1}H-NMR (CD_2Cl_2):

$\delta = 1.58$	[d, 12 H,1,3- $\mathrm{CH}\mathit{Me_2}$ $^3J = 7.2$ Hz]
$\delta = 2.33$	[s, 6 H, 4,5-Me]
$\delta = 4.82$	[sept, 2 H, $\mathrm{C}\mathit{H}\mathrm{Me_2}$]

^{13}C-NMR (CD_2Cl_2):

$\delta = 10.6$	[4,5-Me]
$\delta = 20.8$	[1,3-$\mathrm{CH}\mathit{Me}_2$]
$\delta = 53.4$	[1,3-$\mathit{C}\mathrm{HMe_2}$]
$\delta = 127.6$	[C4,5]
not observed	[C2]

^{31}P-NMR (CD_2Cl_2):

$\delta = -8.0$	[PO_2Cl_2]

Elemental analysis for $C_{11}H_{20}Cl_3N_2O_2P$(M = 349.62 g / mol).

Found (cal.) : C 38.09 (37.79), H 5.50 (5.77), N 8.04 (8.07) %.

MS (FAB) : m/z (%) = 215 [100, M^+], 181 [20, ImH^+].

4.5.6 Preparation of 2-iodo-1,3-diisopropyl-4,5-dimethylimidazolium triiodide (**32**)

0.38 g (1.50 mmol) of I_2 was added to a solution of 0.65 g (1.50 mmol) of **29** in 30 ml of CH_2Cl_2. The resulting mixture was refluxed for 6 h filtered, and evaporated in vacuo. Yield after recrystallization from CH_2Cl_2 / Et_2O : 0.85 g (82 %).

^{1}H-NMR (CD_2Cl_2):

$\delta = 1.58$	[d, 12 H,1,3- CH*Me₂* $^3J = 7.2$ Hz]
$\delta = 2.30$	[s, 6 H, 4,5-Me]
$\delta = 4.90$	[sept, 2 H, C*H*Me$_2$]

^{13}C-NMR (CD_2Cl_2):

$\delta = 11.4$	[4,5-Me]
$\delta = 21.30$	[1,3-CH*Me*$_2$]
$\delta = 53.6$	[1,3-*C*HMe$_2$]
$\delta = 128.4$	[C4,5]
$\delta = 127.2$	[C2]

Elemental analysis for $C_{11}H_{20}I_4N_2$ (M = 687.6 g / mol).

Found (cal.) : C 19.08 (19.20), H 2.83 (2.90), N 3.98 (4.07) %.

MS (FAB) : m/z (%) = 307 [100, M$^+$], 265 [18, M$^+$ -C3H7], 242 [14, M$^+$ -2 (C_3H_7)], 181 [12, Im-H$^+$], 136 [12, -I, -C_3H_7], and further fragments.

4.5.7 Preparation of 2-chloro-1,3-diisopropyl-4,5dimethylimidazolium hexachloro-tellurate (IV) (**33**).

0.33 g (1.21 mmol) of $TeCl_4$ was added to a solution of 0.61 g (2.43 mmol) of **26** in 30 ml of CH_2Cl_2. The resulting mixture was refluxed for 4 h filtered, and evaporated in vacuo. Yield after recrystallization from CH_2Cl_2 / Et_2O : 0.78 g (84 %) as yellow crystals.

^{1}H-NMR ($CDCl_3$):

$\delta = 1.62$	[d, 24 H, CH*Me₂* ,^3J = 7.0 Hz]
$\delta = 2.34$	[s, 6 H,4,5- Me]
$\delta = 4.80$	[sept, 2H, C*H*Me$_2$]

^{13}C-NMR ($CDCl_3$):

$\delta = 11.08$	[4,5-Me]
$\delta = 21.30$	[1,3-CH*Me*$_2$]
$\delta = 53.43$	[1,3-*C*HMe$_2$]
$\delta = 128.8$	[C4,5]
$\delta = 126.4$	[C2]

Elemental analysis for $C_{22}H_{40}Cl_8N_4Te$(M = 771.80 g / mol).

Found (cal.) : C 34.08 (34.24), H 5.18 (5.22), N 7.12 (7.26) %.

MS (FAB) : m/z (%) = 215 [80, M^+], 181 [20, ImH^+], 136 [100, -Cl., -C_3H_7], negative FAB : 343 [20, $TeCl_6$], 307 [60, -Cl], 271 [100, -2Cl],] and further fragments.

4.5.8 Preparation of 2-bromo-1,3-diisopropyl-4,5dimethylimidazolium hexabromotellurate (IV) (**34**).

0.36 g (0.81 mmol) of $TeBr_4$ was added to a solution of 0.55 g (2.43 mmol) of **27** in 30 ml of CH_2Cl_2. The resulting mixture was refluxed for 4 h filtered, and evaporated in vacuo. Yield after recrystallization from CH_2Cl_2 / Et_2O : 0.81 g (89 %) as red crystals

^{1}H-NMR (CD_2Cl_2):

δ = 1.58	[d, 24 H,1,3- CH*Me₂* ^3J = 7.2 Hz]
δ = 2.30	[s, 6 H, 4,5-Me]
δ = 4.87	[sept, 2 H, C*H*Me₂]

^{13}C-NMR (CD_2Cl_2):

δ = 11.2	[4,5-Me]
δ = 21.15	[1,3-CH*Me*₂]
δ = 53.8	[1,3-*C*HMe₂]

δ = 127.6	[C4,5]
δ = 126.2	[C2]

Elemental analysis for $C_{22}H_{40}Br_8N_4Te$ (M = 1127.41 g / mol).

Found (cal) : C 23.23 (23.44), H 3.50 (3.58), N 4.89 (4.97) %.

MS (FAB): m/z (%) = 259 [80, M^+], 181 [20, ImH^+], 136 [100, -Br., -C_3H_7] negative FAB : 608 [20, $TeBr_6$], 528 [60, -Br], 448 [60, -2Br], 368 [100, -3Br], and further fragments.

4.5.9 Preparation of 2-bromo-1,3-diisopropyl-4,5dimethylimidazolium pentabromo-tellurate (IV) (**35**).

0.60 g (1.34 mmol) of $TeBr_4$ was added to a solution 0.46 g (1.34 mmol) of **27** in 30 ml of CH_2Cl_2. The resulting mixture was refluxed for 4 h filtered, and evaporated in vacuo. Yield after recrystallization from CH_2Cl_2 / Et_2O : 0.76 g (72 %) as red crystals.

^{1}H-NMR (CD_2Cl_2):

δ = 1.58	[d, 12 H,1,3- CH*Me₂* ^{3}J = 7.2 Hz]
δ = 2.33	[s, 6 H, 4,5-Me]
δ = 4.82	[sept, 2 H, C*H*Me₂]

^{13}C-NMR (CD_2Cl_2):

δ = 11.2	[4,5-Me]

$\delta = 21.1$	[1,3-CHMe_2]
$\delta = 53.4$	[1,3-CHMe$_2$]
$\delta = 128.6$	[C4,5]
not observed	[C2]

Elemental analysis for $C_{11}H_{20}Br_6N_2Te$ (M = 787.31 g / mol).

Found (cal.) : C 16.61 (16.77), H 2.36 (2.54), N 3.42 (3.56) %.

MS (FAB): m/z (%) = 259 [80, M$^+$], 181 [20, ImH$^+$], 136 [100, -Br., -C_3H_7] negative FAB: [20, 528 [60, $TeBr_5$], 448 [60, -Br], 368 [100, -2Br], 288 [20, -3Br], and further fragments.

4.5.10 Preparation of 1,3-diisopropyl-4,5-dimethyl-2-imidazol-2-yliden (diiodo)tellurium(II) (**36**).

1.24 g (1.94 mmol) of TeI_4 was added to a solution of 0.35 g (1.94 mmol) of **10c** in 30 ml of THF. The resulting mixture was refluxed for 5 h and filtered. The precipitate was dissolved in cold CH_2Cl_2, stirred for 2 h filtered off and evaporated to dryness in vacuo. Yield after recrystallization from CH_2Cl_2 /Et_2O : 0.72 g (66 %) as red crystals.

^{1}H-NMR ($CDCl_3$):

$\delta = 1.62$	[d, 24 H,1,3- CHMe_2 $^3J = 7.0$ Hz]
$\delta = 2.34$	[s, 12 H, 4,5-Me]
$\delta = 4.80$	[sept, 4 H, CHMe$_2$]

^{13}C-NMR ($CDCl_3$):

δ = 11.1	[4,5-Me]
δ = 21.3	[1,3-CH*Me*$_2$]
δ = 53.43	[1,3-*C*HMe$_2$]
δ = 128.8	[C4,5]
δ = 126.4	[C2]

Elemental analysis for $C_{11}H_{20}N_2TeI_2$ (M = 561.70 g / mol).

Found (cal.) : C 22.95 (23.53), H 3.43 (3.56), N 4.99 (4.63) %.

MS (FAB) : m/z (%) = 434 [4, ImTeI], 307 [100, ImTe], 181 [16, Im-H$^+$], 136 [94, -Te, -C_3H_7], and further fragments.

4.5.11 Preparation of 1,3-diisopropyl-4,5-dimethylimidazolium benzoate (**42**)

0.42 g (3.44 mmol) of Benzoic acid in 20 ml Et_2O was added to a solution of 0.62 g (3.44 mmol) of **10c** in 30 Et_2O at -78 °C. After stirring for 4 h at room temperature, the precipitate was filtered off, washed with Et_2O (2 x 10 ml) and dried in vacuo. Yield after recrystallization from CH_3CN / Et_2O : 0.92 g (89 %).

^{1}H-NMR (CD_2Cl_2):

δ = 1.58	[d, 12 H,1,3- CH*Me*$_2$ ^{3}J = 7.2 Hz]

$\delta = 2.29$ [s, 6 H, 4,5-Me]

$\delta = 4.48$ [sept, 2 H, C*H*Me$_2$]

$\delta = 5.27$ [m, 5 H, ph]

^{13}C-NMR (CD_2Cl_2):

$\delta = 7.26$ [4,5-Me]

$\delta = 21.38$ [1,3-CH*Me*$_2$]

$\delta = 49.86$ [1,3-*C*HMe$_2$]

$\delta = 128.3$ [C4,5]

$\delta = 131.3$ [C2]

$\delta = 126.6$ [C2,6 Ph]

$\delta = 128.7$ [C3,5 Ph]

$\delta = 140.2$ [C1 Ph]

$\delta = 125.7$ [C4 Ph]

$\delta = 169.4$ [CO]

Elemental analysis for $C_{18}H_{26}N_2O_2$ (M = 302.41 g / mol).

Found (cal) : C 71.41 (71.49), H 8.61 (8.67), N 9.13 (9.26) %.

4.5.12 Preparation of 1,3-Diisopropyl-4,5-dimethylimidazolium hydrogenoxalate (**43**)

0.38 g (3.06 mmol) of oxalic acid in 30 ml THF was added to a solution of 0.55 g (3.06 mmol) of **10c** in 20 THF at -50 °C . After stirring for 4 h at room temperature, the precipitate was filtered off, washed with Et_2O (2 x 10 ml) and dried in vacuo. Yield after recrystallization from CH_3CN / Et_2O : 0.76 g (92 %).

^{1}H-NMR (CD_2Cl_2):

$\delta = 1.58$ [d, 12 H,1,3- CH*Me*$_2$ ^{3}J = 7.2 Hz]

$\delta = 2.29$ [s, 6 H, 4,5-Me]

$\delta = 4.49$ [sept, 2 H, C*H*Me$_2$]

$\delta = 8.06$ [s, 1 H, OH]

$\delta = 8.61$ [s, 1H, C2]

^{13}C-NMR (CD_2Cl_2):

$\delta = 7.26$ [4,5-Me]

$\delta = 21.35$ [1,3-CH*Me*$_2$]

$\delta = 49.183$ [C4,5]

$\delta = 129.41$ [C2]

$\delta = 162.45$ [CO]

Elemental analysis for $C_{13}H_{22}N_2O_4$ (M = 270.16 g / mol).

Found (cal.) : C 57.54 (57.76), H 8.05 (8.20), N 10.16 (10.36) %.

4.5.13 Preparation of bis (1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene) gold(I) chloride (**44**).

0.89 g (1.79 mmol) Ph_3PAuCl was added to a solution of 0.65 g (3.61 mmol) of **10c** in 20 ml of THF at -60 °C. After the reaction mixture was warmed to room temperature, the precipitate was filtered off and dried in vacuo. Yield after recrystallization from CH_3CN /Et_2O : 0.93 g (88 %) as white crystals

^{1}H-NMR (CD_2Cl_2):

δ = 1.58	[d, 24 H,1,3- CH*Me₂* ^{3}J = 7.2 Hz]
δ = 2.13	[s, 12 H, 4,5-Me]
δ = 4.75	[sept, 4 H, C*H*Me$_2$]

^{13}C-NMR (CD_2Cl_2):

δ = 9.8	[4,5-Me]
δ = 23.2	[1,3-CH*Me*$_2$]
δ = 51.7	[1,3-*C*HMe$_2$]
δ = 125.1	[C4,5]
δ = 178.0	[C2]

Elemental analysis for $C_{22}H_{40}N_4AuCl$ (M = 593.0 g / mol).

Found (cal.) : C 44.42 (44.56), H 6.58 (6.80), N 9.39 (9.45) %.

MS (FAB) : m/z (%) = 557 [100, M$^+$], 377 [20, -Im], 335 [8, -Im, -C_3H_7], 293 [6, -Im, -2(C_3H_7)], 181[4, Im].

4.5.14 Preparation of bis 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene disulfide chloride (**45**).

A 0.34 g (2.5 mmol) S_2Cl_2 was added to a solutionof 0.90 g (5 mmol) of **10c** in 20 ml of Et_2O at -70°C overnight. After that the reaction mixture was warmed to room temperature, the precipitate was filtered off and dried in vacuo. Yield after recrystallization from CH_3Cl /Et_2O : 1.12 g (91 %) as pale yellow crystals.

^{1}H-NMR ($CDCl_3$):

$\delta = 1.58$	[d, 24 H,1,3- $\mathrm{CH}\mathit{Me}_2$ $^3J = 7.2$ Hz]
$\delta = 2.25$	[s, 12 H, 4,5-Me]
$\delta = 4.82$	[sept, 4 H, $\mathrm{C}\mathit{H}\mathrm{Me}_2$]

^{13}C-NMR ($CDCl_3$):

$\delta = 10.8$	[4,5-Me]
$\delta = 21.5$	[1,3-$\mathrm{CH}\mathit{Me}_2$]
$\delta = 53.3$	[1,3-$\mathit{C}\mathrm{HMe}_2$]
$\delta = 128.3$	[C4,5]
Not observed	[C2]

Elemental analysis for $C_{22}H_{40}N_4S_2Cl_2$ (M = 495.61 g / mol).

Found (cal.) : C 53.22 (53.31), H 7.96 (8.13), N 11.06 (11.30) %.

MS (FAB) : m/z (%) = 424 [100, M^+], 212 [20, -Im, -S], 181 [8, -Im, -2S].

4.5.15 Preparation of 2-chloromethyl-1,2,3,4-tetramethylimidazolium chloride (**46**).

0.55 g (2.32 mmol) of C_2Cl_6 was added to a solution of 0.32 g (2.32 mmol) of **17** in 30 ml Et_2O at -20°C. After stirring the mixture for 2 h at room temperature, the precipitate was filtered off, washed with Et_2O (3x 10 ml) and dried vacuo. Yield after recrystallization from CH_2Cl_2 /Et_2O : 0.39 g (81 %) as white crystals.

^{1}H-NMR ($CDCl_3$):

$\delta = 2.12$	[s, 6 H, 4,5-Me]
$\delta = 2.30$	[s, 2 H, CH_2Cl]
$\delta = 3.65$	[s, 6 H, .1,3-Me]

^{13}C-NMR ($CDCl_3$):

$\delta = 8.8$	[4,5-Me]
$\delta = 21.0$	[CH_2Cl]
$\delta = 33.3$	[1,3-Me]
$\delta = 127.2$	[C4,5]
$\delta = 142.3$	[C2]

Elemental analysis for $C_8H_{14}N_2Cl_2$ (M = 209.12 g / mol).

Found (cal.) : C 45.78 (45.93), H 6.62 (6.70), N 13.28 (13.40) %.

MS (FAB) : m/z (%) = 173 [100, M^+], 138 [20, M^+, -Cl], and further fragments.

4.5.16 Preparation of 2-bromomethyl-1,2,3,4-tetramethylimidazolium bromide(**47**)

0.36 g (2.25 mmol) of Br_2 was added to a solution of 0.31 g (2.24 mmol) of **17** in 30 ml Et_2O at 0°C. After stirring the mixture for 2 h at room temperature, the precipitate was filtered off, washed with Et_2O (3x 10 ml) and dried vacuo. Yield after recrystallization from CH_2Cl_2 /Et_2O : 0.56 g (84 %) as white-yellow crystals.

^{1}H-NMR ($CDCl_3$):

δ = 2.15 [s, 6 H, 4,5-Me]

δ = 2.60 [s, 2 H, CH_2Br]

δ = 3.74 [s, 6 H, .1,3-Me]

^{13}C-NMR ($CDCl_3$):

δ = 8.7 [4,5-Me]

δ = 21.1 [CH_2Br]

δ = 32.5 [1,3-Me]

δ = 126.6 [C4,5]

δ = 143.7 [C2]

Elemental analysis for $C_8H_{14}N_2Br_2$ (M = 298.2 g / mol)

Found (cal.) : C 32.02 (32.26), H 4.65 (4.70), N 6.23 (9.40) %

MS (FAB) : m/z (%) = 217 [100, M^+], 138 [20, M^+, -Br], and further fragments.

4.5.17 Preparation of 2-(2,2,2-tribromoethyl)-1,2,3,4-tetramethylimidazolium bromide (**50**).

0.60 g (1.81 mmol) of CBr_4 was added to a solution of 0.25 g (1.81 mmol) of **17** in 30 ml Et_2O at -70°C. After stirring the mixture for 2 h at room temperature, the precipitate was filtered off, washed with Et_2O (3x 10 ml)and dried vacuo. Yield after recrystallization from CH_2Cl_2 /Et_2O : 0.56 g (69 %) as white crystals.

^{1}H-NMR ($CDCl_3$):

$\delta = 2.13$	[s, 6 H, 4,5-Me]
$\delta = 2.71$	[s, 2 H, CH_2CBr_3]
$\delta = 3.74$	[s, 6 H, 1,3-Me]

^{13}C-NMR ($CDCl_3$):

$\delta = 10.2$	[4,5-Me]
$\delta = 23.1$	[$\mathit{CH_2}CBr_3$]
$\delta = 56.3$	[$CH_2\mathit{CBr_3}$]
$\delta = 34.5$	[1,3-Me]
$\delta = 129.6$	[C4,5]
$\delta = 146.7$	[C2]

MS (FAB) : m/z (%) = 388 [68, M^+], 308 [100, M^+, -Br], 228 [100, M^+, -2Br], and further fragments.

4.5.18 Preparation of 1,2,3,4.5-pentamethylimidazolium benzoate (**52**).

0.28 g (2.32 mmol) of benzoic acid was added to a solution of 0.32 g (2.32 mmol) of **17** in 30 ml Et_2O at -70°C. After stirring the mixture for 4 h at room temperature, the precipitate was filtered off, washed with Et_2O (3x 10 ml) and dried in vacuo. Yield after recrystallization from CH_3CN/Et_2O : 0.55 g (91 %) as white crystals.

^{1}H-NMR ($CDCl_3$):

$\delta = 2.13$	[s, 6 H, 4,5-Me]
$\delta = 2.71$	[s, 3 H, CH_3]
$\delta = 3.74$	[s, 6 H, .1,3-Me]

^{13}C-NMR ($CDCl_3$):

$\delta = 8.32$	[4,5-Me]
$\delta = 9.96$	[CH_3]
$\delta = 31.88$	[1,3-$\mathit{C}H_3$]
$\delta = 129.07$	[C4,5]
$\delta = 142.32$	[C2]
$\delta = 126.99$	[C2,6 Ph]
$\delta = 127.96$	[C3,5 Ph]

$\delta = 142.25$ [C1 Ph]

$\delta = 125.22$ [C4 Ph]

$\delta = 168.14$ [CO]

4.5.19 preparation of 1,3-diphenyl -2-(1,3,4,5- tetramethyl-2,3-dihydroimidazole-2-ylidene) propane-1,3-dione (**53**).

0.58 g (4.64 mmol) of benzoyl fluoride was added to a solution of 0.32 g (2.32 mmol) of **17** in 30 ml Et_2O at -40° C. After stirring the mixture for 4 h at room temperature, the precipitate was filtered off, washed with Et_2O (3x 10 ml) and dried in vacuo. Yield after recrystallization from $CHCl_3/Et_2O$: 0.51 g (64 %) as white crystals.

^{1}H-NMR ($CDCl_3$)

$\delta = 2.18$ [s, 6 H, 4,5-Me]

$\delta = 3.55$ [s, 6 H, 1,3-Me]

$\delta =7.33-7.51$ [m, 10 H, .Ph]

^{13}C-NMR ($CDCl_3$):

$\delta = 7.6$ [4,5-Me]

$\delta = 31.2$ [1,3-Me]

$\delta = 124.3$ [C4,5]

$\delta = 148.6$ [C2]

$\delta = 126.8$ [C2,6 Ph]

$\delta = 128.2$ [C3,5 Ph]

$\delta = 134.8$ [C1 Ph]

$\delta = 130.3$	[C4 Ph]
$\delta = 190.1$	[CO]

Elemental analysis for $C_{22}H_{22}N_2O_2$ (M = 346.33 g / mol).

Found (cal) : C 75.85 (76.30), H 6.18 (6.36), N 7.85 (8.09) %.

IR (KBr): $\upsilon_{CO} = 1665\ cm^{-1}$.

MS (EI) : m/z (%) = 347 [100, M^+], 242 [60, M^+, -PhCO], 138 [30, M^+, - 2PhCO], and further fragments

5. Summary

Objectives of the present work were the synthesis of different types of imidazol-2-yildene derivatives (scheme 1-2) and the synthesis of different types of 2-methyleneimidazoline derivatives (scheme 3).

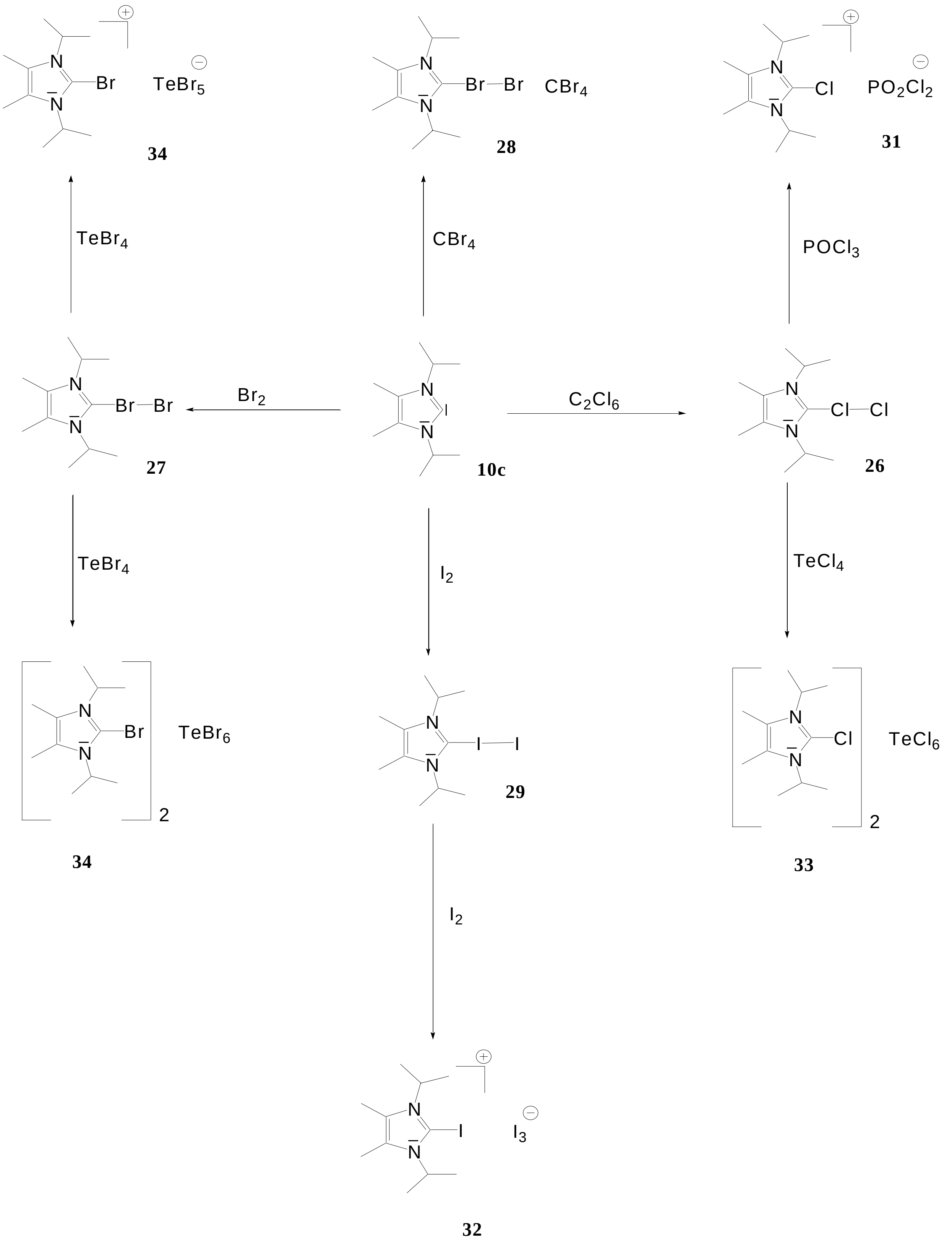
The resulting derivatives from the previous two compounds are classified into halogen charge transfer adducts, salts, and neutral compounds.

The halogen adducts **26**, **27**, **28**, **29**, **30**, **32**, **46**, **47**, and **50** were obtained by the reactions of imidazol-2-yildene or 2-methyleneimidazoline with halogen containing compounds.

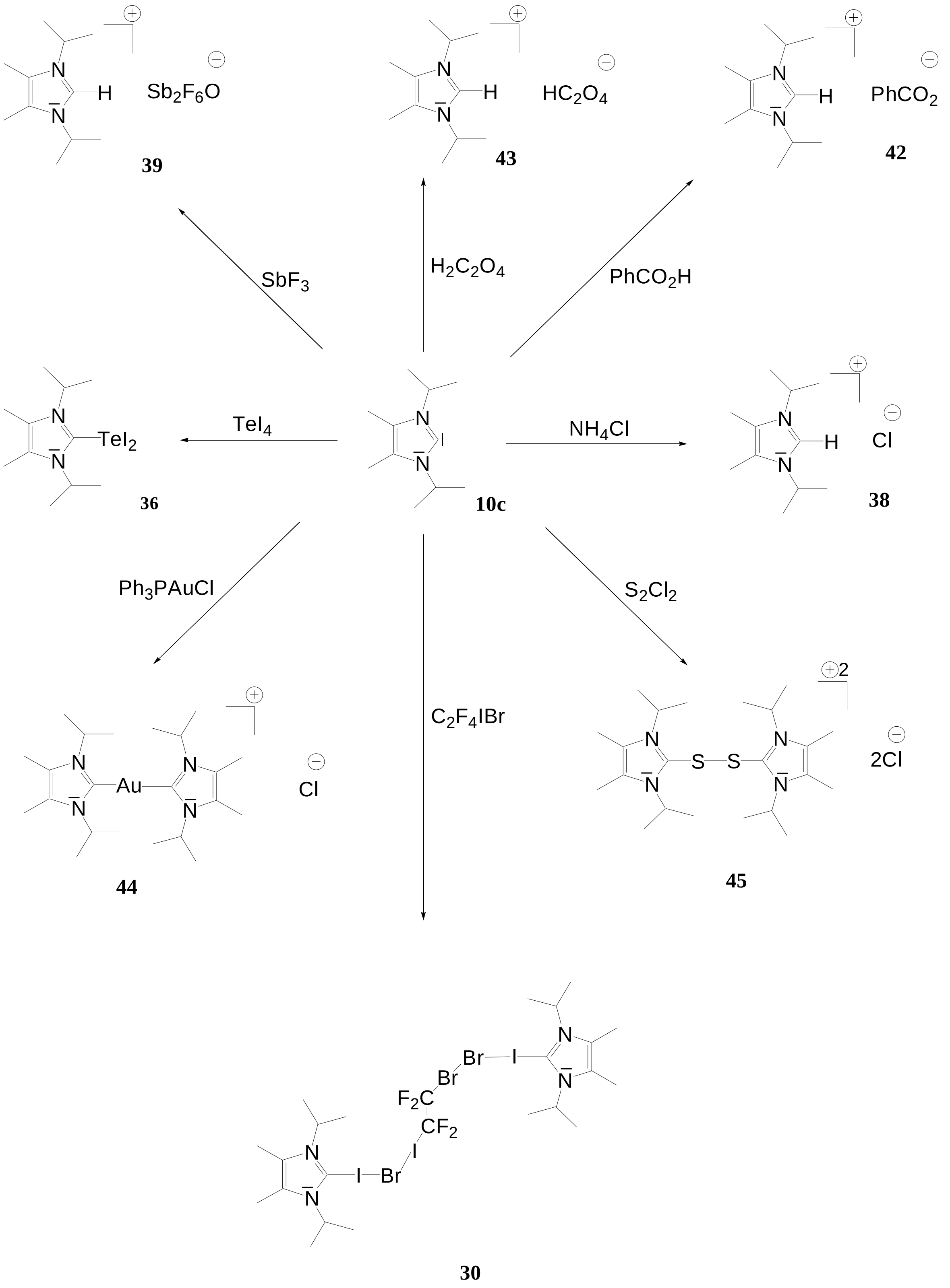
The salts **38**, **40**, **41**, **42**, **43**, **44**, and **45** were obtained by the nucleophilic reactions of imidazol-2-ylidene. While the salts **52** and **55** were obtained from the nucleophilic reactions of 2-methyleneimidazoline.

The salts **33**, **34**, and **35** were obtained from the reaction of 2-haloimidazolium halide with tetrahalotellurium.

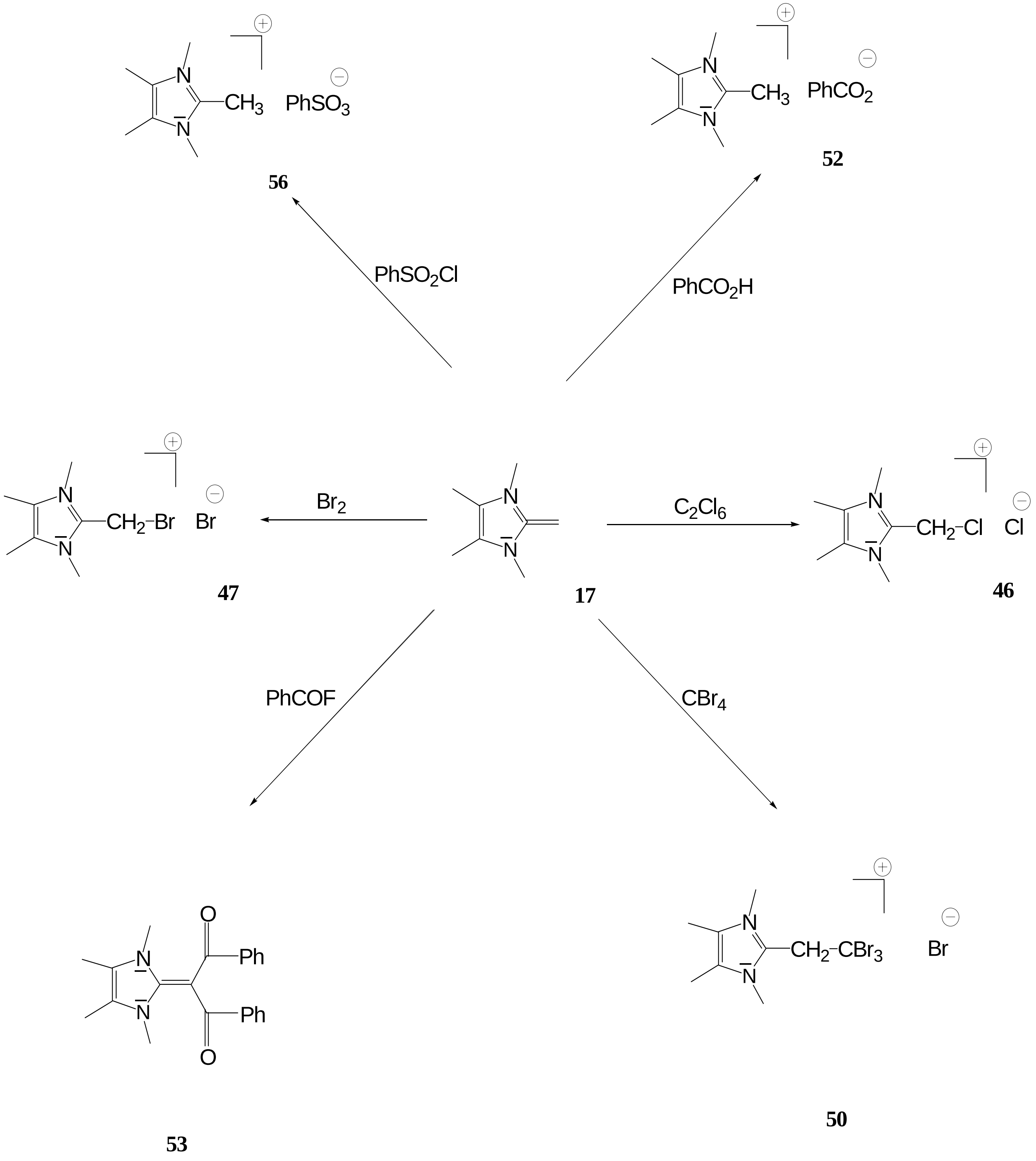
The neutral compound **53** was obtained from the reaction of 2-methyleneimidazoline with benzoyl fluoride.


TeBr$_5$
34
Br—Br CBr$_4$
28
PO$_2$Cl$_2$
31
TeBr$_4$
CBr$_4$
POCl$_3$
Br$_2$
C$_2$Cl$_6$
27
10c
26
TeBr$_4$
I$_2$
TeCl$_4$
TeBr$_6$
34
29
TeCl$_6$
33
I$_2$
I$_3$
32

Scheme 1


Sb2F6O
39
HC2O4
43
PhCO2
42
SbF3
H2C2O4
PhCO2H
TeI2
36
TeI4
10c
NH4Cl
Cl
38
Ph3PAuCl
C2F4IBr
S2Cl2
Au
Cl
44
2Cl
45
Br
F2C
CF2
30

Scheme 2



Scheme 3

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7 Appendix

7.1 Abbreviations andDefinitions

Å	Angstrom (10^{-10})
δ	Chemical shift
h	Hour
EI	Electron ionization (mass spectroscopy)
eV	Electron Volt
FAB	Fast atom bombordment (mass spectroscopy)
FT	Fourier transformation
g	Grams
d	Day
Hz	Hertz
Im	Imidazole
i-Pr	Isopropyl
Me	Methyl
Et	Ethyl
IR	Infrared spectroscopy
CH_3CN	Acetonitrile
mmol	Millimol
MS	Mass spectroscopy
º	Degree
NMR	Nuclear magnetic resonance spectroscopy
Ph	Phenyl
pm	Pico meter

Tab.	Table
THF	Tetrahydrofuran
Et_2O	Diethyl ether
CH_2Cl_2	Dichloromethane
TMS	Tetramethylsilane
ν	Frequency

7.2 Numbering of Compounds

1	Carbene
1a	Singlet methylenecarbene
1b	Triplet difluorocarbene
2	Fischer carbene complex
3	2,3-Diphenylcycloprop-2-en pentacarbonylchromium
4	1,3-Diphenyl-2-trichloromethylimidazlidine
5	1,3-Diphenylimidazlidin-2-ylidene
6	1,3,1’,3’-Tetraphenyl-[2,2’] biimidazolidinylidene
7a	1,3-Diphenylimidazolium chloride
7b	I,3,4,5-Tetraphenylimidazolium chloride
7c	1,3-Diadamantylimidazolium chloride
8a	1,3-Diphenylimidazol-2-ylidene
8b	I,3,4,5-tetraphenylimidazol-2-ylidene
8c	1,3-Diadamantylimidazol-2-ylidene
9a	1,3,4,5-Tetramethylimidazol-2-thione

9b	1,3-Diethyl-4,5-dimetylimidazol-2-thione
9c	1,3-Diisopropyl-4,5-dimethylimidazol-2-thione
10a	1,3,4,5-Tetramethylimidazol-2-ylidene
10b	1,3-Diethyl-4,5-dimetylimidazol-2-ylidene
10c	1,3-Diisopropyl-4,5-dimethylimidazol-2-ylidene
11	Imidazol-2-ylidene adduct
12	2-Haloimidazolium salt
13a	1,3-Diisopropyl-4,5-dimethylimidazol-2-carboxylate
13b	1,3-Diisopropyl-4,5-dimethylimidazol-2-carbothioate
14	2-Halo imidazolium halide
15	Enamine resonance form
16a	1,3-Diisopropyl-4,5-dimethylimidazol-2-selone
16b	1,3-Diisopropyl-4,5-dimethylimidazol-2-tellone
17	1,3,4,5-Tetramethy-2-methyleneimidazoline
18	1,2,3,4,5-Pentametylimidazolium iodide
19	2,4,5-Trimethylimidazol
20	2,4,5-Dimethylimidazolium chloride
21	2-Halo-1,3-Diisopropyl-4,5-dimethylimidazolium halide
22	2-Halo-1,3-Diisopropyl-4,5-dimethylimidazolium salt
23	1,3-Diisopropyl-4,5-dimethylimidazolium salt
20	4,5-Dimethylimidazolium chloride
21	1,3-Diisopropyl-4,5-dimethylimidazolium salt
22	Electroneutral alkylborates
23	2-Dithioethanoate –1,3,4,5-tetramethyl-2,3-dihydroimidazol

24a	2-Halo-1,3-diisopropyl-4,5-dimethylimidazolium salt
24b	1,3-diisopropyl-4,5-dimethylimidazolium salt
25	2-Halomethyl-1,3,4,5-teramethylimidazolium halide
26	2-Chloro-1,3-diisopropyl-4,5-dimethylimidazolium chloride
27	2-Bromo-1,3-diisopropyl-4,5-dimethylimidazolium bromide
28	2-Bromo-1,3-diisopropyl-4,5-dimethylimidazolium bromide tetrabromomethane
29	2-Iodo-1,3-diisopropyl-4,5-dimethylimidazolium iodide……..
30	2-Iodo 1,3-diisopropyl-4,5-dimethylimidazolium bromide
31	2-Chloro-1,3-diisopropyl-4,5-dimethylimidazolium dichlorophosphate
32	2-Iodo-1,3-diisopropyl-4,5-dimethylimidazolium triidide
33	2- Chloro-1,3-diisopropyl-4,5dimethylimidazolium hexachlorotellurate(IV)
34	2- Bromo-1,3-diisopropyl-4,5dimethylimidazolium hexabromotellurate(IV)
35	2- Bromo-1,3-diisopropyl-4,5dimethylimidazolium pentabromotellurate(IV)
36	1,3-Diisopropyl-4,5-dimethyl-2-imidaz-2-yliden(diodo)tellurium(II)
37	1,3-Diisopropyl-4,5-dimethyl-2-imidaz-2-yliden(tetraiodo)tellurium(IV)
38	1,3-Diisopropyl-4,5-dimethylimidazolium chloride
39	1,3-Diisopropyl-4,5-dimethyl-2-imidazol-2-yliden trifluoroantimony(III)
40	1,3-Diisopropyl-4,5-dimethylimidazolium hexafluoro-μ-oxodiantimonate(III)
41	1,3-Diisopropyl-4,5-dimethylimidazolium tetrafluoro-μ-trifluoro-μ-dioxodiantimonate(III)………
42	1,3-Diisopropyl-4,5-dimethylimidazolium benzoate
43	1,3-Diisopropyl-4,5-dimethylimidazolium hydrogenoxalate
44	Bis-(1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene)gold(I) chloride
45	Bis-(1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene)disulfide dichloride

46	2-Chloromethyl-1,3,4,5-teramethylimidazolium chloride
47	2-Bromomethyl-1,3,4,5-teramethylimidazolium bromide
48	2-Bromomethyl-1,3,4,5-teramethylimidazolium bromide
49	2-Bromomethyl-1,3,4,5-teramethylimidazolium bromide
50	2-(2,2,2-Tribromoethyl)-1,2,3,4-tetramethylimidazolium bromide
51	Bis (1,3,4,5-tetramethylimidazol) ethylene dibromide
52	1,2,3,4.5- Pentamethyl-2-imidazolium benzoate
53	1,3-Diphenyl-2-(1,3,4,5-tetramethyl-2,3-dihydroimidazole-2-ylidene) propane-1,3-dione
54	Diisoprpylidene ethylenetetracarboxlyte
55	2-Chloromethyl-1,3,4,5-teramethylimidazolium benzenesulfinate
56	2-Chloromethyl-1,3,4,5-teramethylimidazolium benzenesulfonate

7.3 Crystal Structure's Data

7.3.1 Crystal data for $C_{11}H_{20}N_2Cl_2$ (26)

Table 1 : Crystal data and structure refinement for $C_{11}H_{20}N_2Cl_2$.

Empirical formula	$C_{11}H_{20}N_2Cl_2$	
Formula weight	502.38	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 15.044(4) Å	alpha= 90°.
	b = 10.159(3) Å	beta= 96.37(3)°.

	c = 17.765(10) Å gamma = 90°.
Volume	2698.4(18) Å^3
Z	8
Density (calculated)	1.237 Mg/m^3
Absorption coefficient	0.455 mm^{-1}
F(000)	1072
Crystal size	0.35 x 0.50 x 0.35 mm^3
Theta range for data collection	2.31 to 29.06°.
Index ranges	-20<=h<=11, -13<=k<=13, -23<=l<=23
Reflections collected	11506
Independent reflections	3509 [R(int) = 0.0520]
Completeness to theta = 29.06°	97.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3509 / 0 / 217
Goodness-of-fit on F^2	2.052
Final R indices [I>2sigma(I)]	R1 = 0.0377, wR2 = 0.1001
R indices (all data)	R1 = 0.0463, wR2 = 0.1113
Extinction coefficient	0.0051(9)
Largest diff. peak and hole	0.416 and -0.497 e.Å^{-3}

Table 2 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{11}H_{20}N_2Cl_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	151(1)	8988(1)	1523(1)	33(1)
Cl(2)	1957(1)	9017(1)	3562(1)	50(1)
N(1)	1743(1)	7934(1)	1298(1)	28(1)
N(2)	1674(1)	10073(1)	1157(1)	27(1)
C(1)	1228(1)	9002(1)	1338(1)	28(1)
C(2)	2561(1)	8346(1)	1075(1)	28(1)
C(3)	2516(1)	9674(1)	984(1)	28(1)
C(4)	1524(1)	6539(1)	1460(1)	32(1)
C(5)	750(1)	6035(2)	919(1)	46(1)
C(6)	1382(2)	6346(2)	2280(1)	47(1)
C(7)	1365(1)	11466(1)	1131(1)	31(1)
C(8)	722(1)	11704(2)	430(1)	41(1)
C(9)	1005(1)	11875(2)	1863(1)	45(1)
C(10)	3308(1)	7427(2)	962(1)	36(1)
C(11)	3168(1)	10601(2)	700(1)	37(1)

Table 3 : Bond lengths [Å] and angles [°] for $C_{11}H_{20}N_2Cl_2$.

Cl(1)-C(1)	1.6883(13)
N(1)-C(1)	1.3397(16)
N(1)-C(2)	1.3978(15)
N(1)-C(4)	1.4908(17)
N(2)-C(1)	1.3363(16)
N(2)-C(3)	1.3959(15)
N(2)-C(7)	1.4894(17)
C(2)-C(3)	1.3596(19)
C(2)-C(10)	1.4914(18)
C(3)-C(11)	1.4876(18)
C(4)-C(6)	1.508(2)
C(4)-C(5)	1.514(2)
C(7)-C(8)	1.508(2)
C(7)-C(9)	1.522(2)
(1)-N(1)-C(2)	107.77(11)
C(1)-N(1)-C(4)	128.16(11)
C(2)-N(1)-C(4)	124.07(11)
C(1)-N(2)-C(3)	108.00(11)
C(1)-N(2)-C(7)	128.20(10)
C(3)-N(2)-C(7)	123.80(10)
N(2)-C(1)-N(1)	109.72(11)
N(2)-C(1)-Cl(1)	124.89(10)
N(1)-C(1)-Cl(1)	125.24(10)

C(3)-C(2)-N(1)	107.28(11)
C(3)-C(2)-C(10)	129.35(12)
N(1)-C(2)-C(10)	123.36(12)
C(2)-C(3)-N(2)	107.23(11)
C(2)-C(3)-C(11)	129.97(12)
N(2)-C(3)-C(11)	122.66(12)
N(1)-C(4)-C(6)	111.58(13)
N(1)-C(4)-C(5)	111.70(12)
C(6)-C(4)-C(5)	112.84(15)
N(2)-C(7)-C(8)	110.38(12)
N(2)-C(7)-C(9)	111.82(13)
C(8)-C(7)-C(9)	113.89(14)

Symmetry transformations used to generate equivalent atoms:

Table 4 : Anisotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{20}N_2Cl_2$. The anisotropic displacement factor exponent takes the form: $-2\square^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	22(1)	36(1)	41(1)	0(1)	10(1)	0(1)
Cl(2)	31(1)	28(1)	92(1)	-5(1)	13(1)	-2(1)
N(1)	22(1)	29(1)	35(1)	2(1)	7(1)	1(1)
N(2)	21(1)	28(1)	32(1)	-1(1)	5(1)	1(1)
C(1)	22(1)	30(1)	31(1)	0(1)	5(1)	0(1)
C(2)	22(1)	31(1)	31(1)	1(1)	5(1)	2(1)

C(3)	21(1)	31(1)	32(1)	-1(1)	5(1)	0(1)
C(4)	28(1)	28(1)	38(1)	4(1)	7(1)	1(1)
C(5)	52(1)	35(1)	48(1)	-3(1)	-4(1)	-7(1)
C(6)	61(1)	43(1)	36(1)	9(1)	6(1)	-6(1)
C(7)	28(1)	26(1)	40(1)	-1(1)	7(1)	1(1)
C(8)	42(1)	40(1)	42(1)	6(1)	2(1)	8(1)
C(9)	52(1)	42(1)	45(1)	-10(1)	14(1)	6(1)
C(10)	27(1)	38(1)	45(1)	5(1)	10(1)	9(1)
C(11)	28(1)	35(1)	50(1)	-1(1)	14(1)	-4(1)

Table 5 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{11}H_{20}N_2Cl_2$.

	x	y	z	U(eq)
H(4A)	2039(18)	6050(20)	1318(16)	44(6)
H(5A)	150(20)	6450(30)	999(19)	59(7)
H(5B)	720(20)	5090(30)	963(19)	67(8)
H(5C)	880(30)	6220(40)	370(30)	95(13)
H(6A)	850(20)	6710(40)	2360(20)	72(9)
H(6B)	1370(20)	5410(30)	2384(18)	60(8)
H(6C)	1870(30)	6790(40)	2630(20)	84(11)
H(7A)	1915(17)	11970(20)	1090(15)	41(5)
H(8A)	1006(19)	11490(30)	-25(17)	49(6)
H(8B)	190(20)	11220(30)	456(17)	50(7)

H(8C)	543(18)	12610(30)	423(16)	50(6)
H(9A)	1330(20)	11480(40)	2290(20)	73(9)
H(9B)	400(20)	11640(30)	1856(17)	53(7)
H(9C)	1030(20)	12770(40)	1900(20)	71(9)
H(10A)	3450(20)	6810(30)	1367(19)	62(8)
H(10B)	3170(20)	6870(30)	540(20)	70(9)
H(10C)	3840(20)	7960(30)	910(20)	67(9)
H(11A)	3696(19)	10150(30)	713(17)	53(7)
H(11B)	2970(20)	10880(30)	191(19)	53(7)
H(11C)	3287(16)	11380(30)	1030(15)	39(5)

7.3.2 Crystal data of the compounds $C_{11}H_{20}Br_2N_2$ (**27**)

Table 6 : Crystal data and structure refinement for $C_{11}H_{20}Br_2N_2$

Empirical formula	$C_{11}H_{20}Br_2N_2$	
Formula weight	340.11	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 7.4292(16) Å	a= 90°.
	b = 9.2057(13) Å	b= 90°.
	c = 20.878(4) Å	g = 90°.
Volume	1427.9(5) Å^3	
Z	4	

Density (calculated)	1.582 Mg/m^3
Absorption coefficient	5.653 mm^{-1}
F(000)	680
Crystal size	0.2 x 0.5 x 0.2 mm^3
Theta range for data collection	2.42 to 27.48°.
Index ranges	-1<=h<=9, -1<=k<=11, -1<=l<=27
Reflections collected	2519
Independent reflections	2332 [R(int) = 0.0307]
Completeness to theta = 27.48°	99.6 %
Max. and min. transmission	0.5159 and 0.3905
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2332 / 0 / 143
Goodness-of-fit on F^2	1.037
Final R indices [I>2sigma(I)]	R1 = 0.0340, wR2 = 0.0701
R indices (all data)	R1 = 0.0459, wR2 = 0.0746
Absolute structure parameter	-0.008(19)
Extinction coefficient	0.0052(5)
Largest diff. peak and hole	0.493 and -0.493 e.Å^{-3}

Table 7 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3)for $C_{11}H_{20}Br_2N_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	6556(1)	6684(1)	1223(1)	35(1)
Br(2)	8866(1)	3877(1)	1236(1)	40(1)
N(1)	5018(5)	9390(5)	798(2) 3	1(1)
N(2)	3704(5)	8532(4)	1648(2)	34(1)
C(1)	5027(6)	8310(5)	1225(2)	32(1)
C(2)	3625(7)	10344(5)	951(2)	35(1)
C(3)	2830(7)	9803(6)	1486(2)	37(1)
C(4)	6317(7)	9631(5)	251(2)	33(1)
C(5)	6289(7)	8384(6)	-227(2)	39(1)
C(6)	8176(8)	9987(6)	503(3) 4	4(1)
C(7)	3251(8)	7647(7)	2227(2)	46(1)
C(8)	2349(11)	6235(8)	2049(3)	85(3)
C(9)	4779(8)	7537(8)	2679(2)	66(2)
C(10)	3318(8)	11729(6)	600(2)	50(1)
C(11)	1253(7)	10380(6)	1857(2)	45(1)

Table 8 : Bond lengths [Å] and angles [°] for $C_{11}H_{20}Br_2N_2$.

Br(1)-C(1)	1.879(5)
Br(1)-Br(2)	3.1017(8)
N(1)-C(1)	1.336(6)
N(1)-C(2)	1.394(6)

N(1)-C(4)	1.513(6)
N(2)-C(1)	1.338(6)
N(2)-C(3)	1.380(6)
N(2)-C(7)	1.496(6)
C(2)-C(3)	1.358(7)
C(2)-C(10)	1.488(7)
C(3)-C(11)	1.502(7)
C(4)-C(6)	1.514(7)
C(4)-C(5)	1.520(6)
C(7)-C(9)	1.480(7)
C(7)-C(8)	1.509(9)
C(1)-Br(1)-Br(2)	176.36(14)
C(1)-N(1)-C(2)	108.7(4)
C(1)-N(1)-C(4)	127.6(4)
C(2)-N(1)-C(4)	123.7(4)
C(1)-N(2)-C(3)	108.2(4)
C(1)-N(2)-C(7)	128.0(4)
C(3)-N(2)-C(7)	123.7(4)
N(1)-C(1)-N(2)	108.9(4)
N(1)-C(1)-Br(1)	126.5(3)
N(2)-C(1)-Br(1)	124.6(3)
C(3)-C(2)-N(1)	106.3(4)
C(3)-C(2)-C(10)	130.7(5)
N(1)-C(2)-C(10)	122.7(5)

C(2)-C(3)-N(2)	108.0(4)
C(2)-C(3)-C(11)	129.3(5)
N(2)-C(3)-C(11)	122.7(5)
N(1)-C(4)-C(6)	110.5(4)
N(1)-C(4)-C(5)	112.1(4)
C(6)-C(4)-C(5)	113.8(4)
C(9)-C(7)-N(2)	112.3(5)
C(9)-C(7)-C(8)	116.1(6)
N(2)-C(7)-C(8)	111.7(4)

Table 9 : Anisotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{20}Br_2N_2$. The anisotropicdisplacement factor exponent takes the form: -2p^2[h^2 a*^{2}U^{11} + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	33	U^{23}	U^{13}	U^{12}
Br(1)	38(1)	31(1)	38(1)	6(1)	5(1)	9(1)
Br(2)	53(1)	29(1)	40(1)	4(1)	12(1)	10(1)
N(1)	33(2)	28(2)	31(2)	1(2)	-3(2)	8(2)
N(2)	29(2)	40(2)	32(2)	4(2)	0(2)	8(2)
C(1)	35(2)	31(2)	30(2)	2(2)	-1(2)	9(2)
C(2)	38(3)	34(3)	34(2)	-4(2)	-7(2)	10(3)
C(3)	32(3)	48(3)	32(2)	-4(2)	-4(2)	7(3)
C(4)	39(3)	32(3)	30(2)	3(2)	3(2)	7(3)
C(5)	44(3)	41(3)	33(2)	-3(2)	-2(2)	6(3)

C(6)	38(3)	37(3)	56(3)	-7(3)	2(3)	-7(3)
C(7)	49(3)	53(3)	36(3)	11(3)	7(3)	7(3)
C(8)	106(6)	90(6)	59(4)	17(4)	30(4)	-30(6)
C(9)	60(4)	103(6)	34(3)	5(3)	-2(3)	23(4)
C(10)	63(4)	46(3)	42(3)	11(3)	4(3)	28(4)
C(11)	37(3)	55(4)	43(3)	-7(3)	-1(2)	19(3)

Table 10 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{11}H_{20}Br_2N_2$.

	x	y	z	U(eq)
H(4)	5888	10512	15	40
H(5A)	7022	8639	-600	59
H(5B)	5048	8200	-363	59
H(5C)	6781	9206	787	66
H(6B)	8132	10903	742	66
H(6C)	9016	10083	143	66
H(7)	2310	8218	2459	55
H(8A)	1174	6438	1855	127
H(8B)	2184	5642	2434	127
H(8C)	3104	5709	1742	127
H(9A)	5617	6788	2531	98
H(9B)	4328	7277	3105	98
H(9C)	5403	8474	2702	98

H(10A)	4374	12357	647	75
H(10B)	2258	12220	776	75
H(10C)	3119	11521	145	75
H(11A)	873	11311	1674	68
H(11B)	1602	10520	2306	68
H(11C)	256	9685	1834	68

7.3.3 Crystal data and structure refinement for $C_{12}H_{20}Br_6N_2$ (**28**)

Table 11 : Crystal data and structure refinement for $C_{12}H_{20}Br_6N_2$

Empirical formula	$C_{12}H_{20}Br_6N_2$	
Formula weight	669.74	
Temperature	293(2) K	
Wavelength	1.54184 Å	
Unit cell dimensions	a = 8.1245(6) Å	alpha= 90°.
	b = 12.5558(12) Å	beta= 90.726(9)°.
	c = 19.971(4) Å	gamma = 90°.
Volume	2037.1(4) $Å^3$	
Z	4	
Density (calculated)	2.184 Mg/m^3	
Absorption coefficient	14.184 mm^{-1}	
F(000)	1256	
Theta range for data collection	5.66 to 64.83°.	
Index ranges	-1<=h<=9, 0<=k<=14, -23<=l<=23	

Reflections collected	4214
Independent reflections	3415 [R(int) = 0.0760]
Completeness to theta = 64.83°	98.8 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3415 / 0 / 182
Goodness-of-fit on F^2	0.982
Final R indices [I>2sigma(I)]	R1 = 0.0691, wR2 = 0.1248
R indices (all data)	R1 = 0.2039, wR2 = 0.1654
Extinction coefficient	0.00009(6)
Largest diff. peak and hole	0.905 and -0.854 e.Å^{-3}

Table 12 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for $C_{12}H_{20}Br_6N_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	6515(3)	7215(2)	3729(1)	62(1)
Br(2)	9775(2)	5494(1)	3442(1)	50(1)
Br(3)	12655(2)	7166(1)	3530(1)	53(1)
Br(4)	8703(3)	6816(2)	2144(1)	68(1)
Br(5)	4299(3)	8942(2)	4445(1)	85(1)
Br(6)	4761(3)	9008(1)	2865(1)	56(1)
N(1)	6474(19)	7078(10)	1067(7)	54(4)
N(2)	8270(17)	8352(9)	1103(6)	43(4)

C(1)	6110(20)	7730(13)	515(9)	47(4)
C(2)	7750(20)	7469(13)	1396(8)	46(5)
C(3)	7240(20)	8554(13)	563(8)	47(4)
C(4)	9660(20)	9072(13)	1288(8)	49(5)
C(5)	5520(30)	6106(17)	1232(12)	98(9)
C(6)	6130(30)	5146(16)	980(13)	110(9)
C(7)	4210(30)	6320(20)	1659(15)	134(11)
C(8)	11270(20)	8508(19)	1129(10)	89(7)
C(9)	9620(20)	9423(14)	2016(8)	64(6)
C(10)	4570(20)	8084(13)	3636(8)	54(5)
C(11)	7560(20)	9449(14)	88(8)	62(6)
C(12)	4820(20)	7564(15)	21(9)	62(6)

Table 13 : Bond lengths [Å] and angles [°] for $C_{12}H_{20}Br_6N_2$.

Br(1)-C(10)	1.926(18)	N(2)-C(4)	1.491(19)
Br(3)-C(10)#1	1.946(16)	C(1)-C(3)	1.39(2)
Br(4)-C(2)	1.864(15)	C(1)-C(12)	1.45(2)
Br(5)-C(10)	1.957(17)	C(3)-C(11)	1.50(2)
Br(6)-C(10)	1.935(16)	C(4)-C(9)	1.52(2)
N(1)-C(2)	1.316(19)	C(4)-C(8)	1.52(3)
N(1)-C(1)	1.40(2)	C(5)-C(7)	1.40(3)
N(1)-C(5)	1.49(2)	C(5)-C(6)	1.40(3)
N(2)-C(2)	1.32(2)	C(10)-Br(3)#2	1.946(15)
N(2)-C(3)	1.380(19)	C(2)-N(1)-C(1)	109.3(15)

C(2)-N(1)-C(5)	127.3(17)	C(1)-C(3)-C(11)	129.5(16)
C(1)-N(1)-C(5)	123.4(17)	N(2)-C(4)-C(9)	112.6(15)
C(2)-N(2)-C(3)	107.8(13)	N(2)-C(4)-C(8)	108.4(14)
C(2)-N(2)-C(4)	130.0(13)	C(9)-C(4)-C(8)	111.6(15)
C(3)-N(2)-C(4)	122.1(13)	C(7)-C(5)-C(6)	132(2)
C(3)-C(1)-N(1)	104.5(14)	C(7)-C(5)-N(1)	112(2)
C(3)-C(1)-C(12)	128.9(17)	C(6)-C(5)-N(1)	116(2)
N(1)-C(1)-C(12)	126.5(17)	Br(1)-C(10)-Br(6)	110.1(9)
N(1)-C(2)-N(2)	110.2(14)	Br(1)-C(10)-Br(3)#2	109.2(8)
N(1)-C(2)-Br(4)	123.6(14)	Br(6)-C(10)-Br(3)#2	109.9(7)
N(2)-C(2)-Br(4)	126.2(12)	Br(1)-C(10)-Br(5)	109.4(7)
N(2)-C(3)-C(1)	108.0(14)	Br(6)-C(10)-Br(5)	109.7(8)
N(2)-C(3)-C(11)	122.0(15)	Br(3)#2-C(10)-Br(5)	108.5(9)

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z #2 x-1,y,z

Table 14 : Anisotropic displacement parameters (Å^{2}x 10^3) for $C_{12}H_{20}Br_6N_2$. The anisotropic displacement factor exponent takes the form: $-2\square^{2}[\, h^{2} a^{*2}U^{11} + ... + 2\, h\, k\, a^{*}\, b^{*}\, U^{12}\,]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	54(1)	63(1)	69(1)	11(1)	-10(1)	-5(1)
Br(2)	51(1)	53(1)	47(1)	1(1)	-4(1)	-5(1)
Br(3)	49(1)	58(1)	53(1)	1(1)	3(1)	-12(1)
Br(4)	86(2)	57(1)	60(1)	11(1)	-4(1)	9(1)

Br(5)	92(2)	93(2)	71(2)	-34(1)	20(1)	-23(2)
Br(6)	53(1)	50(1)	64(1)	8(1)	2(1)	-5(1)
N(1)	65(11)	46(8)	51(9)	-4(7)	9(9)	3(8)
N(2)	50(10)	32(7)	46(8)	2(6)	0(8)	0(7)
C(1)	31(10)	45(9)	64(12)	-22(9)	6(9)	17(9)
C(2)	36(11)	64(11)	37(9)	-6(8)	-13(9)	14(9)
C(3)	47(12)	56(10)	39(10)	-5(8)	18(9)	1(9)
C(4)	52(12)	46(9)	48(10)	-6(8)	-11(10)	0(9)
C(5)	130(20)	55(13)	110(20)	-16(13)	62(18)	-42(15)
C(6)	130(30)	66(15)	140(20)	-4(14)	0(20)	-5(16)
C(7)	100(20)	120(20)	180(30)	70(20)	70(20)	28(19)
C(8)	57(15)	130(20)	79(16)	18(14)	-15(13)	-7(15)
C(9)	78(15)	61(11)	52(11)	-19(9)	-26(11)	-5(11)
C(10)	44(11)	65(11)	51(10)	4(9)	-17(9)	-23(10)
C(11)	79(15)	64(11)	42(10)	-6(9)	-25(10)	14(11)
C(12)	40(12)	86(14)	61(12) -	15(10)	3(10)	11(10)

Table 15 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{12}H_{20}Br_6N_2$.

	x	y	z	U(eq)
H(6A)	7031	5292	685	166
H(6B)	5278	4779	737	166
H(6C)	6516	4709	1344	166

H(7A)	4130	7079	1726	201
H(7B)	4403	5978	2082	201
H(7C)	3205	6064	1462	201
H(8A)	11255	8292	668	133
H(8B)	11386	7892	1410	133
H(8C)	12174	8984	1209	133
H(9A)	8590	9775	2100	96
H(9B)	10509	9904	2106	96
H(9C)	9718	8811	2302	96
H(11A)	8446	9879	256	92
H(11B)	6582	9878	44	92
H(11C)	7839	9166	-342	92
H(12A)	4221	6930	129	94
H(12B)	5302	7486	-413	94
H(12C)	4093	8164	19	94

7.3.4 Crystal data for $C_{11}H_{20}I_2N_2$ (**29**)

Table 16 : Crystal data and structure refinement for $C_{11}H_{20}\ I_2N_2$.

Empirical formula	$C_{11}H_{20}\ I_2N_2$
Formula weight	434.09
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	Pnma

Unit cell dimensions	a = 12.706(3) Å	alpha= 90°.
	b = 15.103(4) Å	beta= 90°.
	c = 7.846(2) Å	gamma = 90°.
Volume	1505.7(6) $Å^3$	
Z	4	
Density (calculated)	1.915 Mg/m^3	
Absorption coefficient	4.154 mm^{-1}	
F(000)	824	
Crystal size	0.35 x 0.35 x 0.35 mm^3	
Theta range for data collection	2.93 to 29.48°.	
Index ranges	-17<=h<=17, -20<=k<=20, -10<=l<=-1	
Reflections collected	6850	
Independent reflections	2020 [R(int) = 0.0355]	
Completeness to theta = 29.48°	93.2 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2020 / 0 / 77	
Goodness-of-fit on F^2	1.054	
Final R indices [I>2sigma(I)]	R1 = 0.0356, wR2 = 0.0954	
R indices (all data)	R1 = 0.0396, wR2 = 0.0986	
Extinction coefficient	0.0054(6)	
Largest diff. peak and hole	1.905 and -2.361 $e.Å^{-3}$	

Table 17 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)for $C_{11}H_{20}I_2N_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	8878(1)	2500	577(1)	36(1)
I(2)	7436(1)	7500	-725(1)	31(1)
N(1)	8528(2)	8220(2)	2524(3)	29(1)
C(1)	8237(4)	7500	1647(5)	30(1)
C(2)	9014(3)	7950(2)	4025(4)	27(1)
C(3)	9421(3)	8549(3)	5387(5)	41(1)
C(4)	8331(3)	9144(2)	1950(4)	37(1)
C(5)	7511(5)	9586(4)	3007(8)	80(2)
C(6)	9352(5)	9667(4)	1865(9)	75(2)

Table 18 : Bond lengths [Å] and angles [°] for $C_{11}H_{20}I_2N_2$

I(2)-C(1)	2.121(4)	N(1)-C(1)-N(1)#1	108.7(3)
C(1)-N(1)	1.339(3)	N(1)-C(1)-I(2)	125.67(17)
C(1)-N(1)#1	1.339(3)	N(1)#1-C(1)-I(2)	125.67(17)
C(2)-C(2)#1	1.360(6)	C(2)#1-C(2)-N(1)	107.05(16)
C(2)-N(1)	1.391(4)	C(2)#1-C(2)-C(3)	127.32(19)
C(2)-C(3)	1.492(4)	N(1)-C(2)-C(3)	125.6(3)
C(4)-N(1)	1.487(4)	N(1)-C(4)-C(5)	111.7(3)
C(4)-C(5)	1.491(6)	N(1)-C(4)-C(6)	110.9(3)
C(4)-C(6)	1.521(7)	C(5)-C(4)-C(6)	112.8(5)
		C(1)-N(1)-C(2)	108.6(2)

C(1)-N(1)-C(4)	124.1(2)	C(2)-N(1)-C(4)	127.3(2)

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+3/2,z

Table 19 : Anisotropic displacement parameters ($Å^2$x 10^3) for $C_{11}H_{20}I_2N_2$ .The anisotropic displacement factor exponent takes the form: $-2\square^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I(1)	38(1)	47(1)	24(1)	0	-1(1)	0
I(2)	37(1)	33(1)	24(1)	0	-4(1)	0
N(1)	37(1)	24(1)	25(1)	0(1)	-4(1)	2(1)
C(1)	37(2)	29(2)	24(2)	0	-7(2)	0
C(2)	29(1)	28(2)	24(1)	-1(1)	-2(1)	-1(1)
C(3)	50(2)	37(2)	36(2)	-7(1)	-13(1)	1(2)
C(4)	55(2)	26(1)	29(1)	2(1)	-5(1)	2(1)
C(5)	83(4)	63(3)	95(4)	32(3)	36(3)	45(3)
C(6)	74(4)	44(3)	108(5)	30(3)	10(3)	-9(2)

Table 20 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10 ^{3}) for $C_{11}H_{20}I_2N_2$.

	x	y	z	U(eq)
H(3A)	8892	8978	5671	61

H(3B)	10040	8848	4985	61
H(3C)	9592	8207	6380	61
H(4)	8055	9109	786	44
H(5A)	6896	9218	3060	121
H(5B)	7330	10146	2505	121
H(5C)	7778	9680	4137	121
H(3A)	8892	8978	5671	61
H(3B)	10040	8848	4985	61
H(3C)	9592	8207	6380	61
H(6A)	9555	9845	2993	113
H(6B)	9252	10183	1169	113
H(6C)	9895	9304	1379	113

7.3.5 Crystal data for $C_{24}H_{40}Br_3F_4I_3N_4$ (**30**)

Table 21 : Crystal data and structure refinement for $C_{24}H_{40}Br_3F_4I_3N_4$.

Empirical formula	$C_{24}H_{40}Br_3F_4I_3N_4$	
Formula weight	1081.04	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 8.808(1) Å	alpha= 90°.

	b = 15.570(4) Å	beta= 105.41(1)°.
	c = 13.484(3) Å	gamma = 90°.
Volume	1782.6(6) Å^3	
Z	2	
Density (calculated)	2.014 Mg/m^3	
Absorption coefficient	6.033 mm^{-1}	
F(000)	1024	
Crystal size	0.45 x 0.45 x 0.25 mm^3	
Theta range for data collection	2.04 to 27.54°.	
Index ranges	-11<=h<=9, -20<=k<=20, -17<=l<=17	
Reflections collected	15394	
Independent reflections	4108 [R(int) = 0.0621]	
Completeness to theta = 27.54°	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4108 / 2 / 189	
Goodness-of-fit on F^2	1.029	
Final R indices [I>2sigma(I)]	R1 = 0.0385, wR2 = 0.0965	
R indices (all data)	R1 = 0.0484, wR2 = 0.1028	
Extinction coefficient	0.0038(3)	
Largest diff. peak and hole	1.176 and -0.842 e.Å^{-3}	

Table 22 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for $C_{24}H_{40}Br_3F_4I_3N_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	7041(1)	10624(1)	5954(1)	36(1)
I(2)	6871(10)	3800(6)	5215(8)	50(1)
Br(2)	6989(16)	3874(8)	5336(11)	46(1)
Br(3)	8590(1)	2030(1)	4918(1)	45(1)
F(2)	6396(7)	5621(3)	4676(6)	131(3)
F(1)	6041(5)	5262(3)	6246(3)	86(1)
N(1)	4854(4)	9733(2)	7105(3)	37(1)
N(2)	6345(4)	8804(2)	6604(3)	33(1)
C(1)	5994(5)	9632(3)	6616(3)	34(1)
C(2)	4496(5)	8926(3)	7423(4)	38(1)
C(3)	5413(5)	8338(3)	7112(3)	34(1)
C(4)	4113(7)	10569(3)	7242(5)	53(1)
C(5)	2329(8)	10541(5)	6790(6)	80(2)
C(6)	4629(8)	10844(5)	8354(6)	79(2)
C(7)	3328(6)	8740(4)	8023(5)	56(1)
C(8)	5411(6)	7402(3)	7244(4)	48(1)
C(9)	7610(5)	8440(3)	6185(4)	40(1)
C(10)	8956(6)	8118(5)	7046(5)	67(2)
C(11)	6997(6)	7781(4)	5354(4)	51(1)
C(12)	5810(10)	4998(6)	5272(5)	93(3)

Table 23 : Bond lengths [Å] and angles [°] for $C_{24}H_{40}Br_3F_4I_3N_4$

Br(2)-C(12)	2.025(14)	C(2)-C(7)	1.497(6)
I(2)-C(12)	2.097(11)	C(3)-C(8)	1.469(7)
I(1)-C(1)	2.114(4)	C(4)-C(6)	1.508(10)
N(1)-C(1)	1.349(5)	C(4)-C(5)	1.528(9)
N(1)-C(2)	1.390(6)	C(9)-C(10)	1.508(8)
N(1)-C(4)	1.490(6)	C(9)-C(11)	1.510(7)
N(2)-C(1)	1.325(6)	F(2)-C(12)	1.440(9)
N(2)-C(3)	1.403(5)	F(1)-C(12)	1.339(7)
N(2)-C(9)	1.489(5)	C(12)-C(12)#1	1.420(16)
C(2)-C(3)	1.358(6)		
C(1)-N(1)-C(2)	107.7(4)	C(2)-C(3)-N(2)	106.0(4)
C(1)-N(1)-C(4)	124.8(4)	C(2)-C(3)-C(8)	127.7(4)
C(2)-N(1)-C(4)	127.5(4)	N(2)-C(3)-C(8)	126.4(4)
C(1)-N(2)-C(3)	109.2(3)	N(1)-C(4)-C(6)	110.3(5)
C(1)-N(2)-C(9)	125.1(4)	N(1)-C(4)-C(5)	111.3(5)
C(3)-N(2)-C(9)	125.6(4)	C(6)-C(4)-C(5)	114.2(5)
N(2)-C(1)-N(1)	109.0(4)	N(2)-C(9)-C(10)	110.4(4)
N(2)-C(1)-I(1)	125.1(3)	N(2)-C(9)-C(11)	112.6(4)
N(1)-C(1)-I(1)	125.9(3)	C(10)-C(9)-C(11)	113.4(5)
C(3)-C(2)-N(1)	108.1(4)	F(1)-C(12)-F(2)	110.9(8)
C(3)-C(2)-C(7)	126.0(5)	F(1)-C(12)-C(12)#1	112.0(8)
N(1)-C(2)-C(7)	125.9(4)	F(2)-C(12)-C(12)#1	99.7(8)

F(1)-C(12)-Br(2)	106.3(6)	F(1)-C(12)-I(2)	110.8(6)
F(2)-C(12)-Br(2)	110.5(6)	F(2)-C(12)-I(2)	110.4(5)
C(12)#1-C(12)-Br(2)	117.3(10)	C(12)#1-C(12)-I(2)	112.4(9)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1

Table 24 : Anisotropic displacement parameters (Å^{2}x 10^3) for $C_{24}H_{40}Br_3F_4I_3N_4$. The anisotropicdisplacement factor exponent takes the form: $-2□^2[h^2 a^{*2}U^{11} + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(2)	49(2)	45(1)	37(3)	-5(2)	0(2)	7(2)
I(2)	46(1)	59(2)	41(2)	-10(1)	3(1)	4(1)
Br(3)	50(1)	45(1)	44(1)	8(1)	20(1)	-2(1)
I(1)	39(1)	36(1)	35(1)	-2(1)	12(1)	-4(1)
N(1)	35(2)	36(2)	43(2)	-1(2)	16(2)	0(2)
N(2)	33(2)	36(2)	31(2)	-4(1)	10(1)	-1(1)
C(1)	34(2)	37(2)	31(2)	-1(2)	10(2)	-2(2)
C(2)	35(2)	44(3)	37(2)	-2(2)	15(2)	-4(2)
C(3)	29(2)	41(2)	30(2)	0(2)	6(2)	-5(2)
C(4)	59(3)	41(3)	71(4)	0(2)	38(3)	6(2)
C(5)	71(4)	101(6)	77(5)	16(4)	33(4)	45(4)
C(6)	76(4)	75(5)	93(5)	-32(4)	36(4)	2(4)
C(7)	52(3)	63(4)	66(4)	2(3)	37(3)	-9(3)
C(8)	60(3)	40(3)	45(3)	3(2)	18(2)	-5(2)

C(9)	38(2)	45(3)	42(2)	-8(2)	19(2)	2(2)
C(10)	39(3)	105(5)	56(3)	-16(3)	8(2)	16(3)
C(11)	56(3)	54(3)	45(3)	-15(2)	15(2)	5(2)
F(2)	131(4)	70(3)	241(7)	20(4)	136(5)	-9(3)
F(1)	96(3)	100(3)	43(2)	-27(2)	-12(2)	26(2)
C(12)	106(6)	94(6)	59(4)	-26(4)	-14(4)	28(5)

Table 25 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{24}H_{40}Br_3F_4I_3N_4$.

	x	y	z	U(eq)
H(4)	4523	10997	6847	64
H(5A)	1928	11117	6675	120
H(5B)	1852	10254	7261	120
H(5C)	2086	10236	6149	120
H(6A)	4204	11402	8424	118
H(6B)	5758	10868	8576	118
H(6C)	4252	10439	8769	118
H(7A)	2281	8765	7573	84
H(7B)	3434	9158	8560	84
H(7C)	3518	8177	8320	84
H(8A)	4895	7260	7767	71
H(8B)	6476	7196	7446	71
H(8C)	4860	7137	6607	71
H(9)	8025	8917	5861	48

H(10A)	9246	8551	7569	101
H(10B)	9841	7990	6782	101
H(10C)	8636	7607	7335	101
H(11A)	7802	7649	5017	77
H(11B)	6090	8006	4860	77
H(11C)	6710	7268	5656	77

7.3.6 Crystal data for $C_{11}H_{20}N_2Cl_3PO_2$ (**32**)

Table 26 : Crystal data and structure refinement for $C_{11}H_{20}N_2Cl_3PO_2$

Empirical formula	$C_{11}H_{20}N_2Cl_3PO_2$	
Formula weight	349.61	
Temperature	210(2) K	
Wavelength	71.073 pm	
Crystal system	orthorhombic	
Space group	P n m a	
Unit cell dimensions	a = 1293.44(6) pm	aqlpha= 90°.
	b = 954.96(5) pm	beta= 90°.
	c = 1334.05(9) pm	gamma = 90°.
Volume	1.64780(16) nm^3	
Z	4	
Density (calculated)	1.409 g/cm^3	
Absorption coefficient	0.652 mm^{-1}	
F(000)	728	

Crystal size	0.44 x 0.26 x 0.21 mm^3
Theta range for data collection	2.62 to 25.88°.
Index ranges	$-15 \le h \le 15, -11 \le k \le 11, -16 \le l \le 16$
Reflections collected	15020
Independent reflections	1699 [R(int) = 0.0466]
Completeness to theta = 25.88°	99.8 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1699 / 0 / 150
Goodness-of-fit on F^2	0.986
Final R indices [I>2sigma(I)]	R1 = 0.0487, wR2 = 0.1173
R indices (all data)	R1 = 0.0701, wR2 = 0.1261
Largest diff. peak and hole	0.680 and -0.327 e.Å^{-3}

Table 27 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (pm^{2}x 10^{-1}) for $C_{11}H_{20}N_2Cl_3PO_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	4677(1)	2500	5411(1)	53(1)
C(1)	3387(2)	2500	5251(2)	35(1)
N(1)	2696(2)	2500	5989(2)	29(1)
C(2)	1726(2)	2500	5553(2)	29(1)
C(3)	1853(2)	2500	4556(2)	30(1)
N(2)	2907(2)	2500	4376(2)	32(1)

C(4)	2915(3)	2500	7090(2)	39(1)
C(5)	3448(3)	1171(4)	7397(2)	49(1)
C(6)	771(3)	2500	6158(3)	48(1)
C(7)	1075(3)	2500	3746(3)	43(1)
C(8)	3407(3)	2500	3362(2)	42(1)
C(9)	4011(3)	1174(4)	3195(2)	56(1)
P(1)	2682(1)	-2500	5314(1)	49(1)
Cl(2)	1648(1)	-888(1)	5335(1)	70(1)
O(1)	3162(2)	-2500	4323(2)	76(1)
O(2)	3225(3)	-2500	6270(2)	81(1)

Table 28 : Bond lengths [pm] and angles [°] for $C_{11}H_{20}N_2Cl_3PO_2$.

Cl(1)-C(1)	168.2(3)	C(4)-C(5)	150.2(4)
C(1)-N(2)	132.3(4)	C(8)-C(9)#1	150.5(4)
C(1)-N(1)	132.9(4)	C(8)-C(9)	150.5(4)
N(1)-C(2)	138.3(4)	P(1)-O(2)	145.6(3)
N(1)-C(4)	149.6(4)	P(1)-O(1)	146.0(3)
C(2)-C(3)	134.0(4)	P(1)-Cl(2)#2	203.89(11)
C(2)-C(6)	147.5(5)	P(1)-Cl(2)	203.89(11)
C(3)-N(2)	138.5(4)		
C(3)-C(7)	147.7(5)	N(2)-C(1)-N(1)	109.7(3)
N(2)-C(8)	150.0(4)	N(2)-C(1)-Cl(1)	125.3(2)
C(4)-C(5)#1	150.2(4)	N(1)-C(1)-Cl(1)	125.0(2)

C(1)-N(1)-C(2)	107.4(2)	N(1)-C(4)-C(5)#1	110.7(2)
C(1)-N(1)-C(4)	126.9(3)	N(1)-C(4)-C(5)	110.7(2)
C(2)-N(1)-C(4)	125.7(3)	C(5)#1-C(4)-C(5)	115.4(4)
C(3)-C(2)-N(1)	107.9(3)	N(2)-C(8)-C(9)#1	111.0(2)
C(3)-C(2)-C(6)	130.1(3)	N(2)-C(8)-C(9)	111.0(2)
N(1)-C(2)-C(6)	122.0(3)	C(9)#1-C(8)-C(9)	114.6(4)
C(2)-C(3)-N(2)	107.0(3)	O(2)-P(1)-O(1)	126.0(2)
C(2)-C(3)-C(7)	130.1(3)	O(2)-P(1)-Cl(2)#2	107.71(9)
N(2)-C(3)-C(7)	122.9(3)	O(1)-P(1)-Cl(2)#2	106.94(8)
C(1)-N(2)-C(3)	108.0(3)	O(2)-P(1)-Cl(2)	107.71(9)
C(1)-N(2)-C(8)	126.4(3)	O(1)-P(1)-Cl(2)	106.94(8)
C(3)-N(2)-C(8)	125.6(3)	Cl(2)#2-P(1)-Cl(2)	98.05(7)

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+1/2,z #2 x,-y-1/2,z

Table 29 : Anisotropic displacement parameters (pm^{2}x 10^{-1}) for $C_{11}H_{20}N_2Cl_3PO_2$ (. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	23(1)	102(1)	33(1)	0	-2(1)	0
C(1)	24(2)	53(2)	28(2)	0	0(1)	0
N(1)	26(1)	41(2)	20(1)	0	-1(1)	0
C(2)	24(1)	34(2)	30(2)	0	1(1)	0
C(3)	25(2)	35(2)	31(2)	0	-1(1)	0

N(2)	24(1)	50(2)	21(1)	0	0(1)	0
C(4)	36(2)	60(3)	19(2)	0	1(1)	0
C(5)	62(2)	56(2)	30(2)	6(1)	-7(1)	-3(2)
C(6)	31(2)	77(4)	36(2)	0	4(2)	0
C(7)	30(2)	64(3)	35(2)	0	-4(2)	0
C(8)	34(2)	75(3)	18(2)	0	2(1)	0
C(9)	52(2)	74(3)	41(2)	-15(2)	8(1)	2(2)
P(1)	39(1)	79(1)	31(1)	0	-2(1)	0
Cl(2)	110(1)	44(1)	57(1)	-4(1)	-2(1)	9(1)
O(1)	43(2)	149(4)	36(2)	0	5(1)	0
O(2)	59(2)	143(4)	40(2)	0	-11(2)	0

Table 30 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (pm^2x 10^{-1}) for $C_{11}H_{20}N_2Cl_3PO_2$.

	x	y	z	U(eq)
H(4A)	2330(30)	2500	7340(30)	36(10)
H(5A)	4110(30)	1200(30)	7180(20)	48(9)
H(5B)	3450(20)	1150(30)	8010(30)	42(8)
H(5C)	3130(20)	460(40)	7160(20)	48(9)
H(6A)	760(30)	1730(40)	6580(30)	70(11)
H(6B)	240(40)	2500	5680(40)	65(14)
H(7A)	1100(30)	1680(40)	3380(30)	74(11)
H(7B)	460(40)	2500	3960(40)	70(16)

H(8A)	2920(30)	2500	2950(30)	36(10)
H(9A)	4640(20)	1210(30)	3530(20)	39(7)
H(9B)	4130(20)	1070(40)	2500(30)	62(10)
H(9C)	3610(30)	420(40)	3410(20)	54(9)

7.3.7 Cystal adta for $C_{11}H_{20}N_2I_4$ (**32**)

Table 31 : Crystal data and structure refinement for $C_{11}H_{20}N_2I_4$.

Empirical formula	$C_{11}H_{20}N_2I_4$	
Formula weight	687.89	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 8.062(5) Å	alpha= 90°.
	b = 21.017(10) Å	beta= 95.00(6)°.
	c = 11.637(7) Å	gamma = 90°.
Volume	1964.2(18) $Å^3$	
Z	4	
Density (calculated)	2.326 Mg/m^3	
Absorption coefficient	6.333 mm^{-1}	
F(000)	1248	
Theta range for data collection	2.54 to 22.50°.	
Index ranges	-8<=h<=1, -22<=k<=22, -12<=l<=12	

Reflections collected	4840
Independent reflections	2494 [R(int) = 0.0431]
Completeness to theta = 22.50°	97.0 %
Max. and min. transmission	0.8094 and 0.4733
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2494 / 0 / 161
Goodness-of-fit on F^2	1.089
Final R indices [I>2sigma(I)]	R1 = 0.0554, wR2 = 0.1623
R indices (all data)	R1 = 0.0784, wR2 = 0.1831
Extinction coefficient	0.0012(2)
Largest diff. peak and hole	2.940 and -1.223 e.Å^{-3}

Table 32 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{20}N_2I_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	-2916(1)	3752(1)	3770(1)	55(1)
I(2)	5059(1)	3753(1)	7573(1)	67(1)
I(3)	3434(1)	3866(1)	5214(1)	88(1)
I(4)	6696(1)	3634(1)	9874(1)	104(1)
N(2)	172(10)	3213(4)	2708(8)	42(2)
N(1)	77(9)	4265(4)	2605(8)	50(3)
C(1)	-731(12)	3741(5)	2940(11)	54(3)
C(4)	-351(14)	2555(5)	2944(11)	56(3)

C(9)	-518(15)	4925(5)	2742(12)	59(3)
C(2)	1572(13)	3413(5)	2197(10)	50(3)
C(3)	1513(14)	4052(5)	2138(10)	55(3)
C(7)	2921(13)	2979(5)	1860(12)	63(3)
C(8)	2783(14)	4483(6)	1686(13)	72(4)
C(5)	917(18)	2228(7)	3768(14)	89(5)
C(10)	732(18)	5314(6)	3474(15)	87(5)
C(6)	-770(20)	2178(7)	1838(14)	97(5)
C(11)	-1020(20)	5223(7)	1573(15)	104(6)

Table 33 : Bond lengths [Å] and angles [°] for $C_{11}H_{20}N_2I_4$.

I(1)-C(1)	2.082(11)	C(9)-C(11)	1.52(2)
I(2)-I(4)	2.891(2)	C(2)-C(3)	1.347(16)
I(2)-I(3)	2.947(2)	C(2)-C(7)	1.498(14)
N(2)-C(1)	1.367(13)	C(3)-C(8)	1.496(16)
N(2)-C(2)	1.386(14)	I(4)-I(2)-I(3)	179.15(4)
N(2)-C(4)	1.478(13)	C(1)-N(2)-C(2)	107.8(8)
N(1)-C(1)	1.355(13)	C(1)-N(2)-C(4)	123.9(8)
N(1)-C(3)	1.395(14)	C(2)-N(2)-C(4)	128.3(8)
N(1)-C(9)	1.481(13)	C(1)-N(1)-C(3)	106.7(8)
C(4)-C(5)	1.505(18)	C(1)-N(1)-C(9)	124.2(9)
C(4)-C(6)	1.524(17)	C(3)-N(1)-C(9)	129.1(9)
C(9)-C(10)	1.503(18)	N(1)-C(1)-N(2)	109.0(9)

N(1)-C(1)-I(1)	124.9(7)	C(10)-C(9)-C(11)	113.3(12)
N(2)-C(1)-I(1)	126.0(7)	C(3)-C(2)-N(2)	107.4(9)
N(2)-C(4)-C(5)	110.8(10)	C(3)-C(2)-C(7)	128.2(11)
N(2)-C(4)-C(6)	112.1(11)	N(2)-C(2)-C(7)	124.4(9)
C(5)-C(4)-C(6)	113.0(11)	C(2)-C(3)-N(1)	109.1(10)
N(1)-C(9)-C(10)	111.2(10)	C(2)-C(3)-C(8)	126.8(11)
N(1)-C(9)-C(11)	110.6(11)	N(1)-C(3)-C(8)	124.0(10)

Symmetry transformations used to generate equivalent atoms:

Table 34 : Anisotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{20}N_2I_4$. The anisotropic displacement factor exponent takes the form: -2□^{2}[h^2 a*^{2}U^{11} + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I(1)	44(1)	60(1)	63(1)	-4(1)	13(1)	-2(1)
I(2)	54(1)	62(1)	91(1)	-9(1)	29(1)	-12(1)
I(3)	61(1)	121(1)	85(1)	-3(1)	20(1)	1(1)
I(4)	88(1)	144(1)	84(1)	3(1)	22(1)	6(1)
N(2)	49(5)	31(5)	48(5)	-7(4)	11(4)	-6(4)
N(1)	38(5)	41(5)	74(7)	0(4)	16(5)	-1(4)
C(1)	30(5)	49(7)	85(9)	8(6)	16(5)	-9(5)
C(4)	56(7)	31(6)	84(10)	-4(6)	22(6)	3(5)
C(9)	69(8)	36(6)	75(8)	-3(6)	27(7)	5(5)
C(2)	52(7)	44(6)	55(7)	-7(5)	13(5)	2(5)
C(3)	68(8)	43(6)	53(8)	-2(6)	1(6)	11(6)

C(7)	46(7)	60(7)	87(10)	-23(7)	24(6)	-5(5)
C(8)	56(8)	61(7)	103(11)	18(8)	27(7)	-2(6)
C(5)	91(11)	62(8)	117(14)	20(8)	31(10)	10(7)
C(10)	95(11)	51(8)	118(14)	-22(8)	17(9)	8(7)
C(6)	134(14)	64(9)	91(12)	-23(8)	10(10)	-45(9)
C(11)	134(15)	70(9)	109(14)	35(10)	9(11)	26(9)

Table 35 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{11}H_{20}N_2I_4$.

	x	y	z	U(eq)
H(4)	-1378	2586	3333	67
H(9)	-1526	4901	3154	70
H(7A)	2432	2618	1454	95
H(7B)	3576	2836	2541	95
H(7C)	3621	3203	1371	95
H(8A)	3507	4239	1243	108
H(8B)	3426	4682	2320	108
H(8C)	2232	4804	1206	108
H(5A)	1106	2478	4458	133
H(5B)	1942	2183	3414	133
H(5C)	511	1816	3959	133
H(10A)	1476	5519	2990	131
H(10B)	1356	5041	4013	131

H(10C)	162	5630	3886	131
H(6A)	224	2107	1460	145
H(6B)	-1555	2414	1335	145
H(6C)	-1250	1776	2021	145
H(11A)	-1512	4905	1060	157
H(11B)	-46	5393	1260	157
H(11C)	-1802	5558	1660	157

7.3.8 Crystal data for $C_{22}H_{40}Cl_8N_4Te$ (**33**).

Table 36 : Crystal data and structure refinement

Empirical formula	$C_{22}H_{40}Cl_8N_4Te$	
Formula weight	385.89	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 9.490(2) Å	alpha= 90°.
	b = 9.829(2) Å	beta= 101.99(1)°.
	c = 18.485(3) Å	gamma = 90°.
Volume	1686.7(5) $Å^3$	
Z	2	
Density (calculated)	1.520 Mg/m^3	
Absorption coefficient	1.534 mm^{-1}	

F(000)	776
Crystal size	0.35 x 0.50 x 0.35 mm^3
Theta range for data collection	2.25 to 29.50°.
Index ranges	-13<=h<=10, -13<=k<=13, -25<=l<=25
Reflections collected	17369
Independent reflections	4702 [R(int) = 0.0472]
Completeness to theta = 29.50°	100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4702 / 0 / 241
Goodness-of-fit on F^2	2.110
Final R indices [I>2sigma(I)]	R1 = 0.0223, wR2 = 0.0531
R indices (all data)	R1 = 0.0269, wR2 = 0.0562
Extinction coefficient	0.0054(5)
Largest diff. peak and hole	0.727 and -0.383 e.$Å^{-3}$

Table 37 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{22}H_{40}Cl_8N_4Te$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Te(1)	0	5000	5000	23(1)
Cl(1)	2057(1)	5394(1)	6111(1)	44(1)

Cl(2)	-1576(1)	4121(1)	5849(1)	37(1)
Cl(3)	863(1)	2588(1)	4868(1)	41(1)
Cl(4)	124(1)	3618(1)	7509(1)	45(1)
Cl(1)	2057(1)	5394(1)	6111(1)	44(1)
N(1)	-227(2)	2687(1)	8829(1)	29(1)
N(2)	1545(2)	4144(2)	8895(1)	32(1)
C(1)	506(2)	3466(2)	8438(1)	33(1)
C(2)	390(2)	2865(2)	9575(1)	27(1)
C(3)	1491(2)	3775(2)	9618(1)	28(1)
C(4)	-1451(2)	1747(2)	8543(1)	33(1)
C(5)	-2770(2)	2506(3)	8127(1)	47(1)
C(6)	-983(3)	587(2)	8105(2)	50(1)
C(7)	-111(2)	2121(2)	10177(1)	37(1)
C(8)	2511(2)	4305(2)	10279(1)	36(1)
C(9)	2628(2)	5098(2)	8691(1)	43(1)
C(10)	1913(4)	6338(2)	8278(2)	56(1)
C(11)	3623(3)	4342(3)	8277(2)	56(1)

Table 38 : Bond lengths [Å] and angles [°] for $C_{22}H_{40}Cl_8N_4Te$.

Te(1)-Cl(2)	2.5335(5)	Te(1)-Cl(1)#1	2.5521(6)
Te(1)-Cl(2)#1	2.5335(5)	Te(1)-Cl(1)	2.5521(6)
Te(1)-Cl(3)	2.5368(6)	Cl(4)-C(1)	1.6862(17)
Te(1)-Cl(3)#1	2.5368(6)	N(1)-C(1)	1.341(2)

N(1)-C(2)	1.3929(19)	C(2)-C(7)	1.490(2)
N(1)-C(4)	1.493(2)	C(3)-C(8)	1.486(2)
N(2)-C(1)	1.335(2)	C(4)-C(6)	1.517(3)
N(2)-C(3)	1.3963(19)	C(4)-C(5)	1.522(3)
N(2)-C(9)	1.496(2)	C(9)-C(10)	1.521(3)
C(2)-C(3)	1.365(2)	C(9)-C(11)	1.527(4)

Cl(2)-Te(1)-Cl(2)#1	180.0	C(2)-N(1)-C(4)	124.34(13)
Cl(2)-Te(1)-Cl(3)	89.133(17)	C(1)-N(2)-C(3)	107.86(13)
Cl(2)#1-Te(1)-Cl(3)	90.867(17)	C(1)-N(2)-C(9)	127.60(14)
Cl(2)-Te(1)-Cl(3)#1	90.867(17)	C(3)-N(2)-C(9)	124.52(14)
Cl(2)#1-Te(1)-Cl(3)#1	89.133(17)	N(2)-C(1)-N(1)	110.00(14)
Cl(3)-Te(1)-Cl(3)#1	180.00(2)	N(2)-C(1)-Cl(4)	124.98(12)
Cl(2)-Te(1)-Cl(1)#1	89.72(2)	N(1)-C(1)-Cl(4)	124.97(13)
Cl(2)#1-Te(1)-Cl(1)#1	90.28(2)	C(3)-C(2)-N(1)	107.42(13)
Cl(3)-Te(1)-Cl(1)#1	89.358(18)	C(3)-C(2)-C(7)	129.60(15)
Cl(3)#1-Te(1)-Cl(1)#1	90.642(18)	N(1)-C(2)-C(7)	122.96(14)
Cl(2)-Te(1)-Cl(1)	90.28(2)	C(2)-C(3)-N(2)	107.07(13)
Cl(2)#1-Te(1)-Cl(1)	89.72(2)	C(2)-C(3)-C(8)	129.60(14)
Cl(3)-Te(1)-Cl(1)	90.642(18)	N(2)-C(3)-C(8)	123.31(14)
Cl(3)#1-Te(1)-Cl(1)	89.358(18)	N(1)-C(4)-C(6)	111.20(15)
Cl(1)#1-Te(1)-Cl(1)	180.0	N(1)-C(4)-C(5)	111.90(15)
C(1)-N(1)-C(2)	107.65(13)	C(6)-C(4)-C(5)	113.60(18)
C(1)-N(1)-C(4)	127.98(14)	N(2)-C(9)-C(10)	111.8(2)

N(2)-C(9)-C(11)	110.45(18)
C(10)-C(9)-C(11)	113.27(18)

Table 39 : Anisotropic displacement parameters ($Å^2$x 10^3) for $C_{22}H_{40}Cl_8N_4Te$. The anisotropicdisplacement factor exponent takes the form: $-2\Box^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Te(1)	21(1)	23(1)	25(1)	-1(1)	3(1)	4(1)
Cl(1)	38(1)	53(1)	36(1)	1(1)	-6(1)	-6(1)
Cl(2)	39(1)	39(1)	36(1)	-1(1)	14(1)	-2(1)
Cl(3)	39(1)	27(1)	59(1)	-1(1)	15(1)	9(1)
Cl(4)	52(1)	58(1)	26(1)	2(1)	9(1)	-13(1)
Cl(1)	38(1)	53(1)	36(1)	1(1)	-6(1)	-6(1)
N(1)	29(1)	30(1)	28(1)	1(1)	8(1)	-4(1)
N(2)	36(1)	34(1)	28(1)	2(1)	11(1)	-8(1)
C(1)	36(1)	36(1)	28(1)	1(1)	10(1)	-6(1)
C(2)	27(1)	27(1)	28(1)	1(1)	9(1)	0(1)
C(3)	34(1)	26(1)	26(1)	0(1)	9(1)	-1(1)
C(4)	27(1)	36(1)	36(1)	-2(1)	7(1)	-6(1)
C(5)	32(1)	56(1)	52(1)	5(1)	3(1)	-1(1)
C(6)	46(1)	44(1)	63(1)	-18(1)	19(1)	-10(1)
C(7)	34(1)	45(1)	33(1)	10(1)	9(1)	-5(1)
C(8)	45(1)	32(1)	31(1)	-2(1)	5(1)	-8(1)
C(9)	50(1)	46(1)	35(1)	3(1)	13(1)	-21(1)
C(10)	82(2)	35(1)	57(1)	5(1)	30(1)	-10(1)
C(11)	39(1)	72(2)	60(1)	20(1)	18(1)	-3(1)

Table 40 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{22}H_{40}Cl_8N_4Te$.

	x	y	z	U(eq)
H(4A)	-1700(30)	1350(30)	8983(17)	42(7)
H(5A)	-3000(40)	3320(40)	8410(20)	64(10)
H(5B)	-3570(40)	1900(40)	8070(19)	55(8)
H(5C)	-2660(40)	2880(40)	7660(20)	65(10)
H(6A)	-810(30)	870(30)	7687(18)	44(7)
H(6B)	-90(50)	160(40)	8350(30)	71(12)
H(6C)	-1710(50)	-90(40)	8030(20)	68(11)
H(7A)	90(40)	1150(40)	10170(20)	65(10)
H(7B)	500(40)	2360(30)	10661(19)	52(8)
H(7C)	-1060(40)	2300(40)	10139(19)	55(8)
H(8A)	3470(40)	4040(40)	10280(20)	64(10)
H(8B)	2480(40)	5280(30)	10280(20)	51(8)
H(8C)	2240(30)	3960(30)	10696(18)	50(8)
H(9A)	3110(40)	5410(40)	9160(20)	54(8)
H(10A)	2620(50)	6980(50)	8290(30)	83(12)
H(10B)	1510(40)	6170(40)	7760(20)	59(9)
H(10C)	1170(50)	6720(50)	8450(30)	86(13)
H(11A)	4440(60)	4950(40)	8260(30)	79(14)
H(11B)	3090(40)	4120(40)	7770(20)	59(9)
H(11C)	3970(50)	3590(50)	8560(20)	78(12)

7.3.9 Crystal data of the compound $C_{22}H_{40}Br_8N_4Te$ (**34**)

Table 41 : Crystal data and structure refinement for $C_{22}H_{40}Br_8N_4Te$.

Empirical formula	$C_{11.50}H_{21}Br_4Cl\ N_2Te_{0.50}$	
Formula weight	606.19	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.771(5) Å	alpha= 114.58(6)°.
	b = 10.068(5) Å	beta= 104.81(3)°.
	c = 11.236(5) Å	gamma = 91.29(3)°.
Volume	961.3(8) $Å^3$	
Z	2	
Density (calculated)	2.094 Mg/m^3	
Absorption coefficient	9.246 mm^{-1}	
F(000)	574	
Theta range for data collection	2.08 to 27.50°.	
Index ranges	-12<=h<=12, -11<=k<=11, -14<=l<=14	
Reflections collected	8387	
Independent reflections	4247 [R(int) = 0.1137]	
Completeness to theta = 27.50°	96.2 %	
Max. and min. transmission	0.5125 and 0.2157	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4247 / 2 / 180	

Goodness-of-fit on F^2	1.079
Final R indices [I>2sigma(I)]	R1 = 0.0630, wR2 = 0.1411
R indices (all data)	R1 = 0.0914, wR2 = 0.1626
Extinction coefficient	0.01(3)
Largest diff. peak and hole	2.050 and -2.440 e.Å^{-3}

Table 42 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) $C_{22}H_{40}Br_8N_4Te$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	6880(50)	1440(50)	130(30)	46(11)
N(1)	8300(300)	2000(300)	2900(300)	30(60)
N(2)	7000(300)	3700(300)	2700(300)	30(60)
C(1)	7400(400)	2400(400)	2000(300)	40(80)
C(4)	7600(400)	4100(400)	4100(300)	30(70)
C(5)	8500(300)	3000(400)	4200(300)	30(60)
C(6)	9100(400)	700(400)	2500(300)	40(90)
C(7)	8200(500)	-700(400)	1600(400)	50(110)
C(8)	10300(400)	900(500)	2000(400)	50(100)
C(9)	5900(400)	4500(400)	2200(300)	30(70)
C(10)	4500(400)	3600(400)	1400(400)	40(90)
C(11)	6600(400)	5200(500)	1500(400)	50(100)
C(12)	7200(400)	5300(400)	5200(300)	40(80)
C(13)	9400(400)	3000(400)	5400(300)	40(80)

Te(1)	5000	10000	5000	25(8)
Br(2)	2500(40)	10930(40)	5360(40)	40(10)
Br(3)	5220(40)	8900(40)	6850(40)	38(10)
Br(4)	3550(40)	7420(40)	2980(40)	46(11)
C(14)	1000(2000)	6000(2000)	800(1400)	200(1000)
Cl(1)	-300(200)	5900(200)	-700(200)	120(70)

Table 43 : Bond lengths [Å] and angles [°] for $C_{22}H_{40}Br_8N_4Te$.

Br(1)-C(1)	1.8(3)	C(9)-C(11)	1.5(4)
N(1)-C(1)	1.3(4)	C(9)-C(10)	1.5(5)
N(1)-C(5)	1.4(4)	Te(1)-Br(4)	2.68(4)
N(1)-C(6)	1.5(4)	Te(1)-Br(4)#1	2.68(4)
N(2)-C(1)	1.3(4)	Te(1)-Br(2)#1	2.70(3)
N(2)-C(4)	1.4(4)	Te(1)-Br(2)	2.70(3)
N(2)-C(9)	1.5(4)	Te(1)-Br(3)#1	2.70(3)
C(4)-C(5)	1.4(4)	Te(1)-Br(3)	2.70(3)
C(4)-C(12)	1.5(4)	C(14)-Cl(1)#2	2(2)
C(5)-C(13)	1.5(4)	C(14)-Cl(1)	1.9(4)
C(6)-C(7)	1.5(5)	Cl(1)-C(14)#2	2(2)
C(6)-C(8)	1.5(5)		
C(1)-N(1)-C(5)	109(10)	C(5)-N(1)-C(6)	123(10)
C(1)-N(1)-C(6)	128(10)	C(1)-N(2)-C(4)	108(10)

C(1)-N(2)-C(9)	129(10)
C(4)-N(2)-C(9)	123(10)
N(2)-C(1)-N(1)	109(10)
N(2)-C(1)-Br(1)	125(10)
N(1)-C(1)-Br(1)	126(10)
C(5)-C(4)-N(2)	106(10)
C(5)-C(4)-C(12)	131(10)
N(2)-C(4)-C(12)	123(10)
C(4)-C(5)-N(1)	108(10)
C(4)-C(5)-C(13)	128(10)
N(1)-C(5)-C(13)	124(10)
N(1)-C(6)-C(7)	113(10)

N(1)-C(6)-C(8)	112(10)	Br(4)-Te(1)-Br(3)#1	89.4(11)
C(7)-C(6)-C(8)	112(10)	Br(4)#1-Te(1)-Br(3)#1	90.6(11)
N(2)-C(9)-C(11)	111(10)	Br(2)#1-Te(1)-Br(3)#1	91.0(10)
N(2)-C(9)-C(10)	111(10)	Br(2)-Te(1)-Br(3)#1	89.0(10)
C(11)-C(9)-C(10)	115(10)	Br(4)-Te(1)-Br(3)	90.6(11)
Br(4)-Te(1)-Br(4)#1	180.000(6)	Br(4)#1-Te(1)-Br(3)	89.4(11)
Br(4)-Te(1)-Br(2)#1	90.3(13)	Br(2)#1-Te(1)-Br(3)	89.0(10)
Br(4)#1-Te(1)-Br(2)#1	89.7(13)	Br(2)-Te(1)-Br(3)	91.0(10)
Br(4)-Te(1)-Br(2)	89.7(13)	Br(3)#1-Te(1)-Br(3)	180.000(8)
Br(4)#1-Te(1)-Br(2)	90.3(13)	Cl(1)#2-C(14)-Cl(1)	110(10)
Br(2)#1-Te(1)-Br(2)	180.000(7)	C(14)#2-Cl(1)-C(14)	70(10)

Table 44 : Anisotropic displacement parameters ($Å^2$x 10^3) for $C_{22}H_{40}Br_8N_4Te$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	50(30)	50(20)	22(15)	8(15)	6(16)	20(20)
N(1)	20(140)	40(160)	30(130)	20(120)	10(120)	0(130)
N(2)	30(160)	40(160)	30(130)	20(130)	0(130)	10(140)
C(1)	0(200)	0(200)	30(140)	20(150)	10(150)	0(170)
C(4)	30(180)	30(170)	30(140)	10(130)	10(140)	10(150)
C(5)	30(170)	30(170)	30(140)	10(130)	10(140)	10(140)
C(6)	0(200)	0(200)	30(160)	10(160)	20(170)	20(190)
C(7)	100(400)	20(180)	40(180)	0(150)	0(200)	0(200)

C(8)	0(200)	100(300)	40(190)	0(180)	10(180)	0(200)
C(9)	30(190)	40(190)	30(160)	20(150)	10(160)	10(160)
C(10)	0(200)	100(200)	40(190)	30(190)	0(170)	10(180)
C(11)	0(200)	100(200)	40(190)	30(190)	0(200)	0(200)
C(12)	0(200)	30(180)	40(170)	10(140)	10(170)	10(170)
C(13)	0(200)	40(190)	30(150)	10(140)	0(160)	10(170)
Te(1)	26(16)	24(15)	26(13)	12(11)	7(12)	9(12)
Br(2)	30(20)	40(20)	50(20)	24(18)	18(18)	19(17)
Br(3)	40(20)	40(20)	38(17)	26(16)	11(17)	9(17)
Br(4)	40(20)	30(20)	44(19)	4(16)	11(19)	0(18)
Cl(1)	110(160)	80(120)	140(160)	10(110)	30(140)	20(120)

Table 45 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{22}H_{40}Br_8N_4Te$.

	x	y	z	U(eq)
H(6)	9578	692	3408	49
H(7A)	7312	-718	1923	80
H(7B)	8645	-1511	1679	80
H(7C)	7876	-836	701	80
H(8A)	9936	947	1103	77
H(8B)	10865	108	1842	77
H(8C)	10957	1828	2594	77
H(9)	5812	5341	3052	40

H(10A)	4508	2898	546	63
H(10B)	3787	4253	1295	63
H(10C)	4188	3126	1911	63
H(11A)	6551	4507	618	69
H(11B)	7554	5670	2031	69
H(11C)	6040	5998	1424	69
H(12A)	6238	5156	5118	56
H(12B)	7485	6220	5190	56
H(12C)	7770	5374	6107	56
H(13A)	9367	3810	6232	55
H(13B)	10370	2936	5385	55
H(13C)	9060	2082	5465	55
H(14A)	1791	5913	885	242
H(14B)	635	6534	1641	242

7.3.10 Crystal data for $C_{11}H_{20}Br_6N_2Te$ (**35**)

Table 46 : Crystal data and structure refinement for $C_{11}H_{20}Br_6N_2Te$.

Empirical formula	$C_{11}H_{20}Br_6N_2Te$
Formula weight	787.35
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1

Unit cell dimensions	a = 9.084(4) Å	alpha= 106.78(5)°.
	b = 10.323(4) Å	beta= 90.64(4)°.
	c = 12.969(10) Å	gamma = 114.35(3)°.
Volume	1049.0(10) Å^3	
Z	2	
Density (calculated)	2.493 Mg/m^3	
Absorption coefficient	12.839 mm^{-1}	
F(000)	724	
Theta range for data collection	4.49 to 29.50°.	
Index ranges	-12<=h<=-1, -12<=k<=13, -17<=l<=17	
Reflections collected	4746	
Independent reflections	4746 [R(int) = 0.0000]	
Completeness to theta = 29.50°	81.1 %	
Max. and min. transmission	0.7391 and 0.3549	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4746 / 0 / 188	
Goodness-of-fit on F^2	1.064	
Final R indices [I>2sigma(I)]	R1 = 0.0494, wR2 = 0.1164	
R indices (all data)	R1 = 0.0771, wR2 = 0.1289	
Extinction coefficient	0.0033(5)	
Largest diff. peak and hole	1.390 and -1.182 e.Å^{-3}	

Table 47 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{11}H_{20}Br_6N_2Te$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Te(1)	6319(1)	9684(1)	1224(1)	19(1)
Br(1)	5697(1)	9119(1)	3034(1)	39(1)
Br(2)	4926(1)	6765(1)	194(1)	36(1)
Br(3)	9093(1)	9638(1)	1359(1)	34(1)
Br(4)	7717(1)	12623(1)	2404(1)	33(1)
Br(5)	3084(1)	9725(1)	815(1)	30(1)
Br(6)	3979(1)	6749(1)	4974(1)	36(1)
N(1)	2636(7)	5157(8)	2755(5)	22(1)
N(2)	1712(7)	6792(8)	3492(5)	22(1)
C(1)	2727(8)	6198(9)	3660(6)	22(1)
C(2)	1494(8)	5041(9)	1957(6)	21(1)
C(3)	915(9)	6064(10)	2427(7)	25(2)
C(4)	3525(9)	4177(10)	2544(7)	27(2)
C(5)	2934(12)	3079(11)	3160(8)	37(2)
C(6)	5393(10)	5149(12)	2752(9)	39(2)
C(7)	987(10)	3892(11)	842(7)	31(2)
C(8)	-369(10)	6360(11)	1922(8)	32(2)
C(9)	1509(11)	7978(11)	4331(7)	32(2)
C(10)	-39(13)	7332(14)	4837(9)	47(3)
C(11)	1630(13)	9287(12)	3959(10)	47(3)

Table 48 : Bond lengths [Å] and angles [°] for $C_{11}H_{20}Br_6N_2Te$.

Te(1)-Br(3)	2.5447(13)	N(2)-C(1)	1.346(9)
Te(1)-Br(1)	2.593(2)	N(2)-C(3)	1.388(10)
Te(1)-Br(2)	2.6377(19)	N(2)-C(9)	1.462(10)
Te(1)-Br(4)	2.694(2)	C(2)-C(3)	1.363(11)
Te(1)-Br(5)#1	2.889(2)	C(2)-C(7)	1.501(12)
Te(1)-Br(5)	2.9998(15)	C(3)-C(8)	1.509(10)
Br(5)-Te(1)#1	2.889(2)	C(4)-C(5)	1.499(13)
Br(6)-C(1)	1.840(8)	C(4)-C(6)	1.545(12)
N(1)-C(1)	1.317(10)	C(9)-C(10)	1.534(13)
N(1)-C(2)	1.402(9)	C(9)-C(11)	1.525(14)
N(1)-C(4)	1.507(9)		

Br(3)-Te(1)-Br(1)	93.43(5)	Br(1)-Te(1)-Br(5)	92.56(5)
Br(3)-Te(1)-Br(2)	90.00(5)	Br(2)-Te(1)-Br(5)	88.74(5)
Br(1)-Te(1)-Br(2)	88.54(6)	Br(4)-Te(1)-Br(5)	91.84(5)
Br(3)-Te(1)-Br(4)	89.84(5)	Br(5)#1-Te(1)-Br(5)	86.01(4)
Br(1)-Te(1)-Br(4)	87.54(6)	Te(1)#1-Br(5)-Te(1)	93.99(4)
Br(2)-Te(1)-Br(4)	176.06(4)	C(1)-N(1)-C(2)	108.6(6)
Br(3)-Te(1)-Br(5)#1	87.98(5)	C(1)-N(1)-C(4)	129.0(7)
Br(1)-Te(1)-Br(5)#1	178.41(3)	C(2)-N(1)-C(4)	122.4(7)
Br(2)-Te(1)-Br(5)#1	90.73(6)	C(1)-N(2)-C(3)	108.2(6)
Br(4)-Te(1)-Br(5)#1	93.20(6)	C(1)-N(2)-C(9)	123.7(7)
Br(3)-Te(1)-Br(5)	173.85(3)	C(3)-N(2)-C(9)	128.1(7)

N(1)-C(1)-N(2)	109.4(7)
N(1)-C(1)-Br(6)	126.6(6)
N(2)-C(1)-Br(6)	124.1(6)
C(3)-C(2)-N(1)	106.7(7)
C(3)-C(2)-C(7)	129.6(7)
N(1)-C(2)-C(7)	123.5(7)
C(2)-C(3)-N(2)	107.0(6)
C(2)-C(3)-C(8)	127.8(8)
N(2)-C(3)-C(8)	125.2(7)
C(5)-C(4)-N(1)	110.3(6)
C(5)-C(4)-C(6)	114.9(7)
N(1)-C(4)-C(6)	110.5(8)
N(2)-C(9)-C(10)	111.3(8)
N(2)-C(9)-C(11)	113.8(8)
C(10)-C(9)-C(11)	113.0(8)

Table 49 : Anisotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{20}Br_6N_2Te$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Te(1)	20(1)	18(1)	19(1)	5(1)	4(1)	10(1)
Br(1)	54(1)	31(1)	25(1)	11(1)	11(1)	11(1)
Br(2)	45(1)	20(1)	34(1)	2(1)	9(1)	10(1)
Br(3)	28(1)	38(1)	41(1)	9(1)	2(1)	22(1)
Br(4)	39(1)	20(1)	34(1)	4(1)	11(1)	11(1)
Br(5)	24(1)	41(1)	29(1)	13(1)	9(1)	18(1)
Br(6)	45(1)	44(1)	19(1)	1(1)	-6(1)	27(1)
N(1)	24(3)	26(3)	18(3)	4(3)	5(2)	15(3)
N(2)	24(3)	21(3)	22(3)	8(3)	5(2)	12(2)
C(1)	18(3)	25(4)	22(4)	3(3)	0(2)	10(3)
C(2)	19(3)	24(4)	22(4)	11(3)	2(2)	7(3)
C(3)	21(3)	31(4)	20(4)	6(3)	-1(3)	9(3)
C(4)	26(3)	29(4)	30(4)	6(3)	3(3)	19(3)
C(5)	48(5)	35(5)	29(5)	7(4)	1(4)	22(4)
C(6)	32(4)	48(6)	47(6)	17(5)	15(4)	25(4)
C(7)	39(4)	37(5)	20(4)	6(4)	6(3)	20(4)
C(8)	30(4)	43(5)	34(5)	22(4)	2(3)	20(4)
C(9)	45(5)	37(5)	21(4)	1(4)	9(3)	30(4)
C(10)	62(6)	57(7)	38(6)	18(5)	29(5)	38(6)
C(11)	53(6)	33(6)	56(7)	6(5)	8(5)	24(5)

Table 50 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{11}H_{20}Br_6N_2Te$.

	x	y	z	U(eq)
H(4)	3231	3590	1767	32
H(5A)	3284	3605	3926	55
H(5B)	3376	2360	2931	55
H(5C)	1762	2567	3019	55
H(6A)	5938	4510	2548	59
H(6B)	5735	5726	3511	59
H(6C)	5666	5816	2326	59
H(7A)	28	3857	492	47
H(7B)	753	2924	904	47
H(7C)	1856	4161	417	47
H(8A)	153	7196	1659	47
H(8B)	-1022	6588	2459	47
H(8C)	-1054	5486	1327	47
H(9)	2429	8393	4916	38
H(10A)	-986	6958	4307	71
H(10B)	-59	8106	5454	71
H(10C)	-41	6528	5068	71
H(11A)	2519	9542	3549	71
H(11B)	1817	10139	4585	71
H(11C)	629	9005	3511	71

7.3.11 Crystal data of the compound $C_{11}H_{20}I_2N_2Te$ (**36**)

Table 51 : Crystal data and structure refinement for $C_{11}H_{20}I_2N_2Te$.

Empirical formula	$C_{11}H_{20}I_2N_2Te$	
Formula weight	561.69	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 14.574(4) Å	alpha= 90°.
	b = 11.9536(19) Å	beta= 91.21(3)°.
	c = 9.691(5) Å	gamma = 90°.
Volume	1687.8(10) $Å^3$	
Z	4	
Density (calculated)	2.210 Mg/m^3	
Absorption coefficient	5.403 mm^{-1}	
F(000)	1032	
Crystal size	0.25 x 0.5 x 0.25 mm^3	
Theta range for data collection	2.20 to 27.51°.	
Index ranges	-18<=h<=18, -15<=k<=15, -12<=l<=10	
Reflections collected	14574	
Independent reflections	3886 [R(int) = 0.0396]	
Completeness to theta = 27.51°	100.0 %	
Max. and min. transmission	0.3018 and 0.2459	
Refinement method	Full-matrix least-squares on F^2	

Data / restraints / parameters	3886 / 0 / 223
Goodness-of-fit on F^2	1.120
Final R indices [I>2sigma(I)]	R1 = 0.0283, wR2 = 0.0625
R indices (all data)	R1 = 0.0359, wR2 = 0.0687
Extinction coefficient	0.00072(7)
Largest diff. peak and hole	1.486 and -1.529 e.Å^{-3}

Table 52 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{20}I_2N_2Te$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Te(1)	2356(1)	4071(1)	-496(1)	25(1)
I(1)	1913(1)	1976(1)	943(1)	48(1)
I(2)	2762(1)	6349(1)	-1427(1)	36(1)
N(1)	1721(2)	5180(3)	2201(3)	21(1)
N(2)	3188(2)	4859(3)	2320(3)	24(1)
C(1)	2432(3)	4767(3)	1499(4)	22(1)
C(2)	2957(3)	5329(4)	3556(4)	28(1)
C(3)	2035(3)	5531(3)	3486(4)	25(1)
C(4)	4117(3)	4488(4)	1907(5)	31(1)
C(5)	4779(4)	5459(5)	1903(6)	41(1)
C(6)	4439(4)	3487(5)	2755(8)	48(1)
C(7)	3598(4)	5554(6)	4739(5)	42(1)

C(8)	1463(4)	6007(5)	4572(5)	34(1)
C(9)	767(3)	5253(4)	1635(5)	27(1)
C(10)	468(4)	6461(5)	1480(7)	44(1)
C(11)	119(4)	4542(6)	2435(8)	50(2)

Table 53 : Bond lengths [Å] and angles [°] for $C_{11}H_{20}I_2N_2Te$.

Te(1)-C(1)	2.105(4)	N(2)-C(4)	1.487(5)
Te(1)-I(2)	2.9333(6)	C(2)-C(3)	1.366(6)
Te(1)-I(1)	2.9446(7)	C(2)-C(7)	1.487(6)
N(1)-C(1)	1.346(5)	C(3)-C(8)	1.471(6)
N(1)-C(3)	1.382(5)	C(4)-C(5)	1.510(7)
N(1)-C(9)	1.486(5)	C(4)-C(6)	1.520(7)
N(2)-C(1)	1.350(5)	C(9)-C(11)	1.500(7)
N(2)-C(2)	1.372(5)	C(9)-C(10)	1.515(7)
C(1)-Te(1)-I(2)	84.74(11)	C(1)-N(2)-C(2)	109.6(3)
C(1)-Te(1)-I(1)	84.79(11)	C(1)-N(2)-C(4)	123.6(3)
I(2)-Te(1)-I(1)	169.412(14)	C(2)-N(2)-C(4)	126.8(3)
C(1)-N(1)-C(3)	108.8(3)	N(2)-C(1)-N(1)	107.5(3)
C(1)-N(1)-C(9)	124.0(3)	N(2)-C(1)-Te(1)	126.9(3)
C(3)-N(1)-C(9)	127.2(3)	N(1)-C(1)-Te(1)	125.7(3)
C(3)-C(2)-N(2)	106.7(4)		
C(3)-C(2)-C(7)	127.4(4)		

N(2)-C(2)-C(7)	125.8(4)
C(2)-C(3)-N(1)	107.4(4)
C(2)-C(3)-C(8)	127.2(4)
N(1)-C(3)-C(8)	125.5(4)
N(2)-C(4)-C(5)	110.9(4)
N(2)-C(4)-C(6)	111.2(4)
C(5)-C(4)-C(6)	114.6(4)
N(1)-C(9)-C(11)	111.8(4)

Table 54 : Anisotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{20}I_2N_2Te$.The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Te(1)	24(1)	29(1)	20(1)	-4(1)	0(1)	2(1)
I(1)	76(1)	30(1)	39(1)	1(1)	-1(1)	-9(1)
I(2)	42(1)	34(1)	33(1)	4(1)	7(1)	1(1)
N(1)	16(2)	25(2)	23(2)	-2(1)	6(1)	2(1)
N(2)	20(2)	29(2)	24(2)	-2(1)	0(1)	2(1)
C(1)	21(2)	22(2)	22(2)	-1(2)	2(2)	0(1)
C(2)	30(2)	31(2)	22(2)	-4(2)	-1(2)	0(2)
C(3)	29(2)	23(2)	24(2)	-1(2)	2(2)	0(2)
C(4)	18(2)	40(2)	33(2)	-3(2)	1(2)	4(2)
C(5)	23(2)	51(3)	50(3)	-1(3)	10(2)	-2(2)
C(6)	30(3)	41(3)	73(4)	6(3)	0(3)	9(2)
C(7)	37(3)	57(3)	30(2)	-10(2)	-9(2)	2(2)

C(8)	34(2)	41(3)	27(2)	-6(2)	9(2)	5(2)
C(9)	19(2)	33(2)	28(2)	-1(2)	2(2)	0(2)
C(10)	31(3)	38(3)	61(4)	11(3)	-9(2)	5(2)
C(11)	25(3)	57(4)	68(4)	21(3)	0(3)	-9(2)

Table 55 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{20}I_2N_2Te$.

	x	y	z	U(eq)
H(4A)	4030(40)	4260(40)	1010(60)	38(14)
H(5A)	4930(50)	5750(60)	2820(70)	62(19)
H(5B)	4520(50)	6100(60)	1490(70)	60(19)
H(5C)	5290(50)	5240(60)	1530(70)	60(20)
H(6A)	4960(40)	3240(50)	2410(60)	43(15)
H(6B)	4560(50)	3700(60)	3790(80)	70(20)
H(6C)	4040(40)	2910(40)	2800(50)	33(13)
H(7A)	3300(40)	5900(50)	5520(60)	49
H(7B)	3930(40)	6130(50)	4530(60)	49
H(7C)	3930(40)	5010(50)	4930(60)	49
H(8A)	1070(40)	6560(50)	4280(60)	48(16)
H(8B)	1070(50)	5510(70)	4960(80)	80(20)
H(8C)	1820(40)	6230(50)	5340(60)	46(16)
H(9A)	770(30)	4950(40)	820(50)	30(13)
H(10A)	420(60)	6770(80)	2410(100)	110(30)

H(10B)	870(60)	6930(70)	1140(80)	80(30)
H(10C)	-110(40)	6530(50)	930(60)	52(17)
H(11A)	-460(50)	4500(50)	1930(70)	58(18)
H(11B)	320(60)	3840(70)	2650(90)	90(30)
H(11C)	10(60)	4810(70)	3280(90)	100(30)

7.3.12 crystal adta for $C_{11}H_{20}ClN_2$ (**38**)

Table 56 : Crystal data and structure refinement for $C_{11}H_{20}ClN_2$.

Empirical formula	$C_{11}H_{20}ClN_2$	
Formula weight	216.75	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 11.572(5) Å	alpha= 90°.
	b = 7.602(6) Å	beta= 105.509(15)°.
	c = 15.071(6) Å	gamma = 90°.
Volume	1277.4(13) $Å^3$	
Z	4	
Density (calculated)	1.127 Mg/m^3	
Absorption coefficient	0.268 mm^{-1}	
F(000)	472	
Crystal size	0.45 x 0.25 x 0.05 mm^3	

Theta range for data collection	2.58 to 27.53°.
Index ranges	-15<=h<=10, -9<=k<=9, -19<=l<=19
Reflections collected	10432
Independent reflections	2931 [R(int) = 0.0982]
Completeness to theta = 27.53°	100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2931 / 0 / 201
Goodness-of-fit on F^2	2.093
Final R indices [I>2sigma(I)]	R1 = 0.0515, wR2 = 0.1327
R indices (all data)	R1 = 0.0739, wR2 = 0.1580
Extinction coefficient	0.001(4)
Largest diff. peak and hole	0.419 and -0.337 e.Å^{-3}

Table 57 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3)for $C_{11}H_{20}ClN_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	207(1)	488(1)	7929(1)	41(1)
N(1)	2869(1)	606(2)	1189(1)	28(1)
N(2)	4659(2)	-462(3)	1541(1)	35(1)
C(1)	4010(2)	913(3)	1649(1)	32(1)
C(2)	3927(2)	-1691(3)	982(1)	35(1)
C(3)	2796(2)	-1018(3)	771(1)	31(1)

C(4)	1859(2)	1853(3)	1077(2)	33(1)
C(5)	1694(2)	2836(4)	174(2)	46(1)
C(6)	2062(2)	3058(5)	1892(2)	54(1)
C(7)	1629(2)	-1774(3)	230(2)	41(1)
C(8)	4390(3)	-3354(4)	707(2)	48(1)
C(9)	5985(2)	-619(4)	1941(2)	45(1)
C(10)	6593(3)	679(13)	1476(4)	161(4)
C(11)	6305(3)	-314(7)	2956(2)	66(1)

Table 58 : Bond lengths [Å] and angles [°] for $C_{11}H_{20}ClN_2$.

N(1)-C(1)	1.337(2)	C(2)-C(8)	1.475(4)
N(1)-C(3)	1.379(3)	C(3)-C(7)	1.494(3)
N(1)-C(4)	1.479(3)	C(4)-C(6)	1.500(3)
N(2)-C(1)	1.323(3)	C(4)-C(5)	1.518(4)
N(2)-C(2)	1.385(3)	C(9)-C(11)	1.493(4)
N(2)-C(9)	1.496(3)	C(9)-C(10)	1.491(6)
C(2)-C(3)	1.361(3)		

C(1)-N(1)-C(3)	108.94(17)	N(2)-C(1)-N(1)	108.25(19)
C(1)-N(1)-C(4)	126.03(18)	C(3)-C(2)-N(2)	106.4(2)
C(3)-N(1)-C(4)	124.85(16)	C(3)-C(2)-C(8)	131.0(2)
C(1)-N(2)-C(2)	109.42(17)	N(2)-C(2)-C(8)	122.7(2)
C(1)-N(2)-C(9)	124.7(2)	C(2)-C(3)-N(1)	107.00(18)
C(2)-N(2)-C(9)	125.9(2)	C(2)-C(3)-C(7)	130.9(2)

N(1)-C(3)-C(7)	122.03(19)	C(11)-C(9)-N(2)	110.3(2)
N(1)-C(4)-C(6)	110.56(18)	C(11)-C(9)-C(10)	111.1(4)
N(1)-C(4)-C(5)	108.91(19)	N(2)-C(9)-C(10)	108.7(2)
C(6)-C(4)-C(5)	112.9(2)		

Symmetry transformations used to generate equivalent atoms:

Table 59 : Anisotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{20}ClN_2$. The anisotropic displacement factor exponent takes the form: $-2\square^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	32(1)	45(1)	42(1)	5(1)	4(1)	3(1)
N(1)	25(1)	33(1)	26(1)	-3(1)	7(1)	-1(1)
N(2)	31(1)	46(1)	28(1)	-3(1)	5(1)	7(1)
C(1)	25(1)	43(1)	29(1)	-6(1)	5(1)	1(1)
C(2)	41(1)	38(1)	25(1)	0(1)	8(1)	4(1)
C(3)	35(1)	32(1)	26(1)	-1(1)	8(1)	-2(1)
C(4)	23(1)	36(1)	40(1)	-7(1)	8(1)	-2(1)
C(5)	47(1)	35(1)	51(1)	2(1)	5(1)	3(1)
C(6)	35(1)	66(2)	59(2)	-27(1)	12(1)	6(1)
C(7)	41(1)	36(1)	41(1)	-6(1)	3(1)	-10(1)
C(8)	63(2)	40(1)	41(1)	-2(1)	12(1)	15(1)
C(9)	31(1)	67(2)	36(1)	0(1)	4(1)	16(1)
C(10)	26(1)	321(11)	142(4)	156(6)	34(2)	30(3)
C(11)	42(1)	103(3)	43(1)	-1(2)	-6(1)	8(2)

Table 60 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{11}H_{20}ClN_2$.

	x	y	z	U(eq)
H(10A)	6294	1837	1538	241
H(10B)	6435	387	835	241
H(10C)	7442	647	1756	241
H(1A)	4270(20)	1960(40)	1990(20)	34(6)
H(4A)	1190(30)	1110(50)	1090(20)	40(7)
H(5A)	1060(30)	3730(60)	60(30)	56(9)
H(6A)	2730(30)	3830(60)	1990(30)	55(9)
H(7A)	1750(30)	-2980(50)	70(30)	57(9)
H(8A)	5020(30)	-3210(60)	390(30)	65(11)
H(9A)	6160(40)	-1930(60)	1880(30)	75(12)
H(11A)	7140(40)	-460(50)	3180(30)	59(10)
H(5B)	2430(30)	3570(50)	180(30)	57(9)
H(6B)	2150(30)	2280(50)	2440(30)	52(9)
H(7B)	1040(30)	-1710(50)	640(20)	43(8)
H(8B)	3760(30)	-3880(50)	270(30)	51(9)
H(11B)	5940(50)	-1220(90)	3200(40)	107(19)
H(5C)	1500(30)	2070(50)	-360(30)	55(9)
H(6C)	1400(40)	3810(60)	1830(30)	73(12)
H(7C)	1300(30)	-1150(50)	-330(30)	51(9)
H(8C)	4720(40)	-4020(60)	1210(30)	68(11)
H(11C)	6060(60)	1100(110)	3030(50)	130(20)

7.3.13 Crystal data for $C_{22}H_{42}F_6N_4Sb_2O$ (**40**).

Table 61 : Crystal data and structure refinement for $C_{22}H_{42}F_6N_4Sb_2O$.

Empirical formula	$C_{22}H_{42}F_6N_4Sb_2O$	
Formula weight	736.10	
Temperature	220(2) K	
Wavelength	71.073 pm	
Crystal system	monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 963.78(9) pm	alpha= 90°.
	b = 1610.1(2) pm	beta= 87.46(1)°.
	c = 1897.6(2) pm	gamma = 90°.
Volume	2.9419(6) nm^3	
Z	4	
Density (calculated)	1.662 g/cm^3	
Absorption coefficient	1.895 mm^{-1}	
F(000)	1464	
Crystal size	0.44 x 0.35 x 0.4 mm^3	
Theta range for data collection	2.33 to 25.03°.	
Index ranges	$-11 \leq h \leq 11, -19 \leq k \leq 19, -22 \leq l \leq 22$	
Reflections collected	25253	
Independent reflections	5084 [R(int) = 0.0486]	
Completeness to theta = 25.03°	97.7 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5084 / 0 / 406	

Goodness-of-fit on F^2	1.023
Final R indices [I> 2σ(I)]	R1 = 0.0459, wR2 = 0.1209
R indices (all data)	R1 = 0.0538, wR2 = 0.1255
Largest diff. peak and hole	3.432 and -0.935 e.Å^{-3}

Table 62 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (pm^{2}x 10^{-1})for $C_{22}H_{42}F_6N_4Sb_2O$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
F(1)	307(6)	-1657(2)	-461(2)	90(1)
F(2)	2495(4)	-2389(3)	-1084(3)	82(1)
F(3)	1232(4)	-3845(2)	-1444(2)	65(1)
Sb(1)	682(1)	-2828(1)	-815(1)	44(1)
O(1)	-90(3)	-2421(2)	-1673(2)	41(1)
Sb(2)	-1146(1)	-3212(1)	-2189(1)	48(1)
F(4)	-565(5)	-2539(3)	-3055(2)	77(1)
F(5)	-2800(4)	-2555(3)	-2203(2)	76(1)
F(6)	-1836(4)	-3534(2)	-1160(2)	68(1)
C(1)	-195(5)	402(3)	1658(3)	38(1)
N(1)	703(4)	-135(2)	1375(2)	39(1)
C(2)	2039(5)	60(3)	992(3)	44(1)
C(3)	1870(7)	43(4)	205(3)	52(1)
C(4)	2591(6)	891(4)	1235(4)	51(1)

C(5)	176(5)	-934(3)	1476(3)	42(1)
C(6)	916(7)	-1704(4)	1228(3)	57(1)
C(7)	-1058(5)	-855(3)	1836(3)	44(1)
C(8)	-2055(7)	-1512(4)	2071(4)	65(2)
N(2)	-1276(4)	-15(2)	1945(2)	40(1)
C(9)	-2536(5)	353(4)	2302(3)	51(1)
C(10)	-3622(7)	480(7)	1771(5)	76(2)
C(11)	-2178(7)	1152(4)	2675(4)	59(2)
C(12)	1063(5)	1600(3)	4348(3)	41(1)
N(3)	1733(4)	889(2)	4445(2)	41(1)
C(13)	2612(6)	450(4)	3903(3)	52(1)
C(14)	2098(9)	570(5)	3187(4)	84(2)
C(15)	4117(3)	706(2)	3970(2)	90(3)
C(16)	1526(3)	656(2)	5141(2)	40(1)
C(17)	2144(3)	-112(2)	5441(2)	54(1)
C(18)	715(5)	1242(3)	5460(3)	42(1)
C(19	198(7)	1303(5)	6204(4)	59(2)
N(4)	439(4)	1825(2)	4949(2)	41(1)
C(20)	-423(5)	2589(3)	5041(3)	45(1)
C(21)	-1754(6)	2484(4)	4680(4)	67(2)
C(22)	400(7)	3332(4)	4797(5)	58(2)

Table 63 : Bond lengths [pm] and angles [°] for $C_{22}H_{42}F_6N_4Sb_2O$.

F(1)-Sb(1)	202.8(4)	F(2)-Sb(1)	193.2(4)

F(3)-Sb(1)	208.1(3)	N(2)-C(9)	148.8(6)
Sb(1)-O(1)	193.5(3)	C(9)-C(10)	150(1)
Sb(1)-Sb(2)	327.02(6)	C(9)-C(11)	151.4(9)
O(1)-Sb(2)	192.4(3)	C(12)-N(4)	131.6(7)
Sb(2)-F(5)	191.4(4)	C(12)-N(3)	133.1(6)
Sb(2)-F(4)	202.7(4)	N(3)-C(16)	137.9(6)
Sb(2)-F(6)	210.0(4)	N(3)-C(13)	148.3(7)
C(1)-N(1)	132.0(6)	C(13)-C(14)	148(1)
C(1)-N(2)	133.5(6)	C(13)-C(15)	151.9(7)
N(1)-C(5)	139.4(6)	C(16)-C(18)	135.0(6)
N(1)-C(2)	148.3(6)	C(16)-C(17)	149.6(6)
C(2)-C(3)	151.0(9)	C(18)-N(4)	138.4(7)
C(2)-C(4)	151.8(8)	C(18)-C(19)	148.0(8)
C(5)-C(7)	135.1(7)	N(4)-C(20)	149.0(6)
C(5)-C(6)	149.5(7)	C(20)-C(21)	149.0(9)
C(7)-N(2)	138.3(7)	C(20)-C(22)	149.8(8)
C(7)-C(8)	148.4(7)		

F(2)-Sb(1)-O(1)	91.77(16)	F(1)-Sb(1)-F(3)	163.38(17)
F(2)-Sb(1)-F(1)	83.9(2)	F(2)-Sb(1)-Sb(2)	111.80(15)
O(1)-Sb(1)-F(1)	83.92(16)	O(1)-Sb(1)-Sb(2)	31.97(10)
F(2)-Sb(1)-F(3)	86.10(19)	F(1)-Sb(1)-Sb(2)	110.17(13)
O(1)-Sb(1)-F(3)	83.17(15)	F(3)-Sb(1)-Sb(2)	61.73(11)

Sb(2)-O(1)-Sb(1)	115.85(16)	C(1)-N(2)-C(7)	108.7(4)
F(5)-Sb(2)-O(1)	95.84(16)	C(1)-N(2)-C(9)	126.3(4)
F(5)-Sb(2)-F(4)	83.88(19)	C(7)-N(2)-C(9)	125.0(4)
O(1)-Sb(2)-F(4)	85.72(16)	N(2)-C(9)-C(10)	109.2(5)
F(5)-Sb(2)-F(6)	85.42(18)	N(2)-C(9)-C(11)	110.7(4)
O(1)-Sb(2)-F(6)	80.99(14)	C(10)-C(9)-C(11)	112.3(6)
F(4)-Sb(2)-F(6)	161.98(16)	N(4)-C(12)-N(3)	108.8(5)
F(5)-Sb(2)-Sb(1)	112.69(14)	C(12)-N(3)-C(16)	108.4(4)
O(1)-Sb(2)-Sb(1)	32.17(10)	C(12)-N(3)-C(13)	125.5(5)
F(4)-Sb(2)-Sb(1)	113.99(13)	C(16)-N(3)-C(13)	126.0(4)
F(6)-Sb(2)-Sb(1)	57.64(11)	C(14)-C(13)-N(3)	111.7(5)
N(1)-C(1)-N(2)	108.9(4)	C(14)-C(13)-C(15)	113.6(6)
C(1)-N(1)-C(5)	108.6(4)	N(3)-C(13)-C(15)	109.4(4)
C(1)-N(1)-C(2)	126.9(4)	C(18)-C(16)-N(3)	107.2(4)
C(5)-N(1)-C(2)	124.4(4)	C(18)-C(16)-C(17)	129.7(3)
N(1)-C(2)-C(3)	110.6(5)	N(3)-C(16)-C(17)	123.1(2)
N(1)-C(2)-C(4)	110.2(4)	C(16)-C(18)-N(4)	106.6(4)
C(3)-C(2)-C(4)	111.7(5)	C(16)-C(18)-C(19)	129.6(5)
C(7)-C(5)-N(1)	106.9(4)	N(4)-C(18)-C(19)	123.8(5)
C(7)-C(5)-C(6)	129.3(5)	C(12)-N(4)-C(18)	109.0(4)
N(1)-C(5)-C(6)	123.8(4)	C(12)-N(4)-C(20)	124.1(4)
C(5)-C(7)-N(2)	106.9(4)	C(18)-N(4)-C(20)	127.0(4)
C(5)-C(7)-C(8)	129.0(5)	N(4)-C(20)-C(21)	109.8(5)
N(2)-C(7)-C(8)	124.1(5)	N(4)-C(20)-C(22)	109.8(4)

C(21)-C(20)-C(22) 113.7(6)

Symmetry transformations used to generate equivalent atoms:

Table 64 : Anisotropic displacement parameters ($pm^2 x\ 10^{-1}$) for $C_{22}H_{42}F_6N_4Sb_2O$.The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
F(1)	152(4)	53(2)	67(3)	-20(2)	-40(3)	35(2)
F(2)	56(2)	79(3)	111(4)	26(2)	-27(2)	-16(2)
F(3)	84(2)	48(2)	63(2)	-2(2)	1(2)	19(2)
Sb(1)	60(1)	35(1)	39(1)	5(1)	-9(1)	8(1)
O(1)	47(2)	33(2)	43(2)	10(1)	-9(2)	-5(1)
Sb(2)	65(1)	35(1)	45(1)	2(1)	-9(1)	-6(1)
F(4)	109(3)	73(2)	47(2)	17(2)	-5(2)	0(2)
F(5)	57(2)	80(3)	94(3)	0(2)	-25(2)	13(2)
F(6)	71(2)	53(2)	80(3)	21(2)	9(2)	-13(2)
C(1)	42(2)	33(3)	41(3)	4(2)	-2(2)	3(2)
N(1)	43(2)	38(2)	37(2)	5(2)	0(2)	2(2)
C(2)	39(2)	45(3)	48(3)	8(2)	4(2)	4(2)
C(3)	55(3)	52(3)	49(3)	-4(3)	10(3)	-4(3)
C(4)	45(3)	62(4)	44(4)	-1(3)	1(3)	-9(3)
C(5)	53(3)	37(2)	35(3)	3(2)	-6(2)	-1(2)
C(6)	77(4)	46(3)	48(3)	3(2)	-7(3)	8(3)

C(7)	58(3)	40(3)	35(3)	0(2)	-3(2)	-7(2)
C(8)	80(4)	54(3)	59(4)	2(3)	5(3)	-24(3)
N(2)	42(2)	38(2)	41(2)	4(2)	5(2)	-4(2)
C(9)	48(3)	52(3)	51(3)	6(3)	13(2)	-3(2)
C(10)	49(4)	99(6)	82(6)	2(5)	-4(4)	8(4)
C(11)	58(3)	49(3)	67(4)	-1(3)	22(3)	8(3)
C(12)	52(3)	32(2)	37(3)	5(2)	0(2)	5(2)
N(3)	50(2)	30(2)	44(3)	1(2)	-7(2)	3(2)
C(13)	66(3)	30(3)	58(4)	-6(2)	4(3)	10(2)
C(14)	131(6)	67(4)	54(4)	-5(3)	6(4)	47(4)
C(15)	67(4)	81(5)	120(7)	-40(5)	18(4)	9(4)
C(16)	39(2)	35(2)	46(3)	5(2)	-10(2)	-4(2)
C(17)	64(3)	38(3)	63(4)	11(3)	-18(3)	2(2)
C(18)	36(2)	46(3)	43(3)	8(2)	-4(2)	-5(2)
C(19)	50(3)	78(5)	50(4)	11(3)	5(3)	0(3)
N(4)	44(2)	36(2)	42(2)	3(2)	1(2)	2(2)
C(20)	54(3)	41(3)	39(3)	-1(2)	7(2)	11(2)
C(21)	52(3)	52(3)	96(5)	1(3)	-2(3)	19(3)
C(22)	57(4)	41(3)	75(5)	-9(3)	7(4)	6(3)

Table 65 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (pm^{2}x 10 $^{-1}$) for $C_{22}H_{42}F_6N_4Sb_2O$.

	x	y	z	U(eq)
H(1A)	-170(40)	920(30)	1660(20)	21(11)
H(2A)	2630(60)	-340(40)	1120(30)	48(15)
H(3A)	2720(70)	160(40)	10(30)	58(17)
H(3B)	1560(60)	-480(50)	10(40)	60(18)
H(3C)	1240(60)	410(40)	60(30)	52(17)
H(4A)	3530(70)	940(40)	1030(40)	62(18)
H(4B)	2710(50)	920(40)	1680(40)	42(15)
H(4C)	2020(60)	1380(40)	1020(30)	51(15)
H(6A)	1806	-1740	1446	68
H(6B)	1063	-1684	719	68
H(6C)	359	-2186	1358	68
H(8A)	-2934	-1425	1852	78
H(8B)	-2198	-1488	2580	78
H(8C)	-1685	-2052	1936	78
H(9A)	-2850(60)	-40(40)	2680(30)	51(15)
H(10A)	-4260(90)	770(50)	2000(40)	80(20)
H(10B)	-3200(80)	910(50)	1440(40)	80(20)
H(10C)	-3920(80)	40(50)	1520(40)	70(20)
H(11A)	-2920(60)	1340(30)	2970(30)	41(14)
H(11B)	-1430(70)	1110(40)	2960(40)	60(20)
H(11C)	-1960(50)	1620(40)	2340(30)	40(13)
H(12A)	990(50)	1850(40)	3990(30)	39(15)
H(13A)	2510(50)	0(40)	4040(30)	28(13)

H(14A)	2115	1157	3071	101
H(14B)	1154	364	3173	101
H(14C)	2689	269	2848	101
H(15C)	4695	417	3618	108
H(15B)	4413	564	4437	108
H(15A)	4204	1301	3898	108
H(17A)	3148	-84	5387	65
H(17B)	1813	-595	5193	65
H(17C)	1870	-155	5938	65
H(19A)	-560(130)	1290(90)	6220(70)	150(50)
H(19B)	460(60)	1880(40)	6340(30)	46(15)
H(19C)	490(70)	780(50)	6490(40)	66(18)
H(20A)	-600(50)	2630(30)	5470(30)	33(13)
H(21A)	-2253	2009	4878	80
H(21B)	-1559	2395	4180	80
H(21C)	-2316	2980	4748	80
H(22A)	-240(90)	3850(60)	4850(50)	100(30)
H(22B)	1200(70)	3290(40)	4960(30)	46(17)
H(22C)	650(60)	3290(40)	4360(40)	50(17)

7.3.14 Crystal data for $C_{22}H_{42}F_7N_4Sb_3O_2$ (**41**)

Table 66 : Crystal data and structure refinement for $C_{22}H_{42}F_7N_4Sb_3O_2$.

Empirical formula	$C_{22}H_{42}F_7N_4Sb_3O_2$

Formula weight	892.85	
Temperature	220(2) K	
Wavelength	71.073 pm	
Crystal system	monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 2137.7(3) pm	alpha= 90°.
	b = 974.43(9) pm	beta= 116.48(2)°.
	c = 1709.0(2) pm	gamma = 90°.
Volume	3.1863(7) nm^3	
Z	4	
Density (calculated)	1.861 Mg/m^3	
Absorption coefficient	2.591 mm^{-1}	
F(000)	1736	
Crystal size	0.4 x 0.4 x 0.4 mm^3	
Theta range for data collection	2.92 to 28.03°.	
Index ranges	$-28 \le h \le 28$, $-12 \le k \le 12$, $-21 \le l \le 21$	
Reflections collected	24261	
Independent reflections	3585 [R(int) = 0.0462]	
Completeness to theta = 28.03°	92.8 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3585 / 0 / 257	
Goodness-of-fit on F^2	0.882	
Final R indices [I>2sigma(I)]	R1 = 0.0316, wR2 = 0.0745	

R indices (all data)	R1 = 0.0530, wR2 = 0.0788
Largest diff. peak and hole	2.242 and -0.489 e.Å^{-3}

Table 67 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (pm^{2}x 10^{-1})for $C_{22}H_{42}F_7N_4Sb_3O_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Sb(1)	1070(1)	1353(1)	3258(1)	35(1)
Sb(2)	0	-1359(1)	2500	40(1)
F(1)	0	2313(3)	2500	50(1)
F(2)	1996(1)	474(3)	3612(2)	71(1)
F(3)	1311(1)	2432(3)	2488(2)	56(1)
F(4)	-574(2)	-1000(3)	1171(2)	69(1)
O(1)	657(1)	-82(3)	2407(2)	37(1)
C(1)	1458(2)	5988(4)	603(3)	31(1)
N(1)	1063(2)	5202(3)	-65(2)	28(1)
C(2)	1266(2)	3882(4)	-309(3)	35(1)
C(3)	1768(3)	3098(5)	490(4)	49(2)
C(4)	1558(3)	4154(7)	-950(5)	58(2)
C(5)	428(2)	5858(4)	-541(3)	26(1)
C(6)	-145(2)	5261(5)	-1340(3)	34(1)
C(7)	459(2)	7068(4)	-141(3)	27(1)
C(8)	-59(2)	8208(5)	-381(4)	36(1)

N(2)	1109(2)	7114(3)	583(2)	28(1)
C(9)	1376(3)	8235(5)	1250(3)	39(1)
C(10)	1824(3)	7661(7)	2139(4)	55(1)
C(11)	1717(4)	9336(7)	964(5)	73(2)

Table 68 : Bond lengths [pm] and angles [°] for $C_{22}H_{42}F_7N_4Sb_3O_2$.

Sb(1)-O(1)	192.4(3)	N(1)-C(5)	138.9(5)
Sb(1)-F(3)	192.5(3)	N(1)-C(2)	147.5(5)
Sb(1)-F(2)	198.7(2)	C(2)-C(4)	150.7(7)
Sb(1)-F(1)	226.8(2)	C(2)-C(3)	151.5(7)
Sb(2)-O(1)	193.5(3)	C(5)-C(7)	135.0(5)
Sb(2)-O(1)#1	193.5(3)	C(5)-C(6)	148.7(6)
Sb(2)-F(4)#1	207.4(3)	C(7)-N(2)	139.0(5)
Sb(2)-F(4)	207.4(3)	C(7)-C(8)	149.0(5)
F(1)-Sb(1)#1	226.8(2)	N(2)-C(9)	149.6(5)
C(1)-N(2)	131.7(5)	C(9)-C(11)	149.5(8)
C(1)-N(1)	132.1(5)	C(9)-C(10)	149.6(8)

O(1)-Sb(1)-F(3)	93.1(1)	F(3)-Sb(1)-F(1)	83.88(9)
O(1)-Sb(1)-F(2)	88.7(1)	F(2)-Sb(1)-F(1)	165.0(1)
F(3)-Sb(1)-F(2)	84.8(1)	O(1)-Sb(2)-O(1)#1	99.96(16)
O(1)-Sb(1)-F(1)	82.1(1)	O(1)-Sb(2)-F(4)#1	83.41(13)

O(1)#1-Sb(2)-F(4)#1	84.14(12)	C(7)-C(5)-N(1)	106.9(3)
O(1)-Sb(2)-F(4)	84.14(12)	C(7)-C(5)-C(6)	129.9(4)
O(1)#1-Sb(2)-F(4)	83.41(13)	N(1)-C(5)-C(6)	123.2(4)
F(4)#1-Sb(2)-F(4)	160.58(18)	C(5)-C(7)-N(2)	106.4(3)
Sb(1)#1-F(1)-Sb(1)	131.26(15)	C(5)-C(7)-C(8)	130.4(4)
Sb(1)-O(1)-Sb(2)	120.71(15)	N(2)-C(7)-C(8)	123.1(4)
N(2)-C(1)-N(1)	109.0(3)	C(1)-N(2)-C(7)	109.0(3)
C(1)-N(1)-C(5)	108.6(3)	C(1)-N(2)-C(9)	124.7(3)
C(1)-N(1)-C(2)	126.6(3)	C(7)-N(2)-C(9)	126.3(3)
C(5)-N(1)-C(2)	124.7(3)	C(11)-C(9)-N(2)	110.5(4)
N(1)-C(2)-C(4)	108.7(4)	C(11)-C(9)-C(10)	114.5(5)
N(1)-C(2)-C(3)	111.4(4)	N(2)-C(9)-C(10)	110.7(4)
C(4)-C(2)-C(3)	112.8(4)		

Table 69: Anisotropic displacement parameters (pm^{2}x 10^{-1}) for $C_{22}H_{42}F_7N_4Sb_3O_2$. The anisotropic displacement factor exponent takes the form: -2□^{2}[h^2 a*^{2}U^{11} + ... + 2 h k a* b* U^{12}].

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Sb(1)	28(1)	37(1)	36(1)	-1(1)	10(1)	3(1)
Sb(2)	52(1)	30(1)	44(1)	0	28(1)	0
F(1)	44(2)	32(2)	71(3)	0	21(2)	0
F(2)	28(1)	67(2)	92(3)	5(2)	4(1)	13(1)
F(3)	40(1)	59(2)	68(2)	12(1)	23(1)	-7(1)

F(4)	79(2)	87(2)	39(2)	-22(2)	24(2)	-35(2)
O(1)	35(1)	42(2)	40(2)	-7(1)	22(1)	-2(1)
C(1)	25(2)	35(2)	31(3)	-3(2)	10(2)	-2(2)
N(1)	26(1)	27(2)	32(2)	0(1)	14(1)	-2(1)
C(2)	33(2)	29(2)	41(3)	-8(2)	13(2)	0(2)
C(3)	39(3)	31(2)	63(4)	0(2)	9(3)	1(2)
C(4)	60(3)	61(3)	65(5)	-9(3)	40(3)	12(3)
C(5)	26(2)	27(2)	27(2)	3(2)	13(2)	-3(1)
C(6)	36(2)	29(2)	30(3)	2(2)	9(2)	-3(2)
C(7)	29(2)	29(2)	27(2)	2(2)	17(2)	-2(1)
C(8)	41(2)	33(2)	36(3)	0(2)	19(2)	4(2)
N(2)	30(2)	29(2)	27(2)	-3(1)	14(1)	-5(1)
C(9)	40(2)	41(2)	37(3)	-14(2)	17(2)	-2(2)
C(10)	65(4)	57(4)	36(3)	-16(3)	16(3)	-4(3)
C(11)	94(5)	45(3)	76(5)	-17(3)	33(4)	-32(3)

Table 70 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (pm^2x 10^{-1}) for $C_{22}H_{42}F_7N_4Sb_3O_2$.

	x	y	z	U(eq)
H(1A)	1810(20)	5810(40)	970(30)	29(11)
H(2A)	870(20)	3350(40)	-560(30)	37(12)
H(3A)	2050(30)	3400(50)	640(40)	37(16)
H(3B)	1820(20)	2230(50)	220(30)	42(12)
H(3C)	1550(20)	2970(50)	930(30)	44(13)

H(4A)	1670(20)	3420(50)	-1080(40)	48(15)
H(4B)	1160(20)	4350(40)	-1580(30)	26(10)
H(4C)	1940(20)	4520(40)	-680(30)	33(13)
H(6A)	-210(20)	4530(50)	-1270(30)	36(13)
H(6B)	-510(30)	5780(50)	-1530(30)	46(13)
H(6C)	-40(20)	5160(50)	-1830(40)	54(15)
H(8A)	-440(20)	8000(40)	-880(30)	34(11)
H(8B)	120(20)	9010(50)	-440(30)	31(11)
H(8C)	-180(20)	8390(40)	50(30)	25(10)
H(9A)	1080(20)	8550(40)	1220(30)	22(12)
H(10A)	2190(30)	7360(60)	2170(40)	70(20)
H(10B)	1880(30)	8190(50)	2480(40)	45(15)
H(10C)	1610(30)	6850(60)	2260(30)	55(15)
H(11A)	2080(20)	8960(40)	1070(30)	15(11)
H(11B)	1890(30)	70(60)	1340(40)	55(14)
H(11C)	1300(40)	9760(90)	180(70)	160(30)

7.3.15 Crystal data for $C_{18}H_{26}N_2O_2$ (**42**)

Table71 : Crystal data and structure refinement for $C_{18}H_{26}N_2O_2$

Empirical formula	$C_{18}H_{26}N_2O_2$
Formula weight	302.41
Temperature	223(2) K
Wavelength	1.54184 A
Crystal system, space group	Monoclinic, P21/c

Unit cell dimensions	a = 12.1059(12) A alpha = 90 deg.
	b = 10.8221(4) A beta = 109.057(8) deg.
	c = 14.0833(11) A gamma = 90 deg.
Volume	1743.9(2) A^3
Z, Calculated density	4, 1.152 Mg/m^3
Absorption coefficient	0.594 mm^-1
F(000)	656
Crystal size	0.60 x 0.40 x 0.25 mm
Theta range for data collection	5.27 to 64.97 deg.
Index ranges	-1<=h<=14, -1<=k<=12, -16<=l<=15
Reflections collected / unique	3806 / 2954 [R(int) = 0.0469]
Completeness to 2theta = 64.97	94.2%
Absorption correction	None
Max. and min. transmission	and
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2954 / 0 / 304
Goodness-of-fit on F^2	1.061
Final R indices [I>2sigma(I)]	R1 = 0.0550, wR2 = 0.1495
R indices (all data)	R1 = 0.0617, wR2 = 0.1566
Extinction coefficient	0.038(2)
Largest diff. peak and hole	0.295 and -0.168 e.A^-3

Table 72 : Atomic coordinates (x 10^4) and equivalent isotropicdisplacement parameters (A^2 x 10^3) for $C_{18}H_{26}N_2O_2$. U(eq) is defined as one third of the trace of the orthogonalized :Uij tensor.

	x	y	z	U(eq)
C(1)	7992(2)	3419(2)	1921(1)	54(1)
C(3)	8505(2)	2220(2)	3279(1)	51(1)
C(4)	7379(2)	1953(2)	2735(1)	50(1)
C(11)	7282(1)	7553(2)	188(1)	47(1)
C(12)	7446(2)	7749(2)	-727(1)	57(1)
C(13)	7165(2)	8876(2)	-1218(2)	71(1)
C(14)	6738(2)	9822(3)	-782(2)	76(1)
C(15)	6573(2)	9645(2)	129(2)	69(1)
C(16)	6841(2)	8518(2)	614(1)	55(1)
C(21)	10036(2)	3733(2)	3047(2)	60(1)
C(22)	10481(2)	3776(3)	2167(2)	79(1)
C(23)	9963(3)	4994(4)	3468(3)	100(1)
C(31)	9263(2)	1725(3)	4263(2)	66(1)
C(41)	6569(2)	1014(2)	2903(2)	63(1)
C(51)	5885(2)	2883(2)	1158(1)	52(1)
C(52)	5952(2)	3159(2)	125(2)	64(1)
C(53)	5277(2)	3899(2)	1539(2)	68(1)
C(111)	7568(2)	6322(2)	727(1)	56(1)
N(2)	8872(1)	3133(2)	2748(1)	53(1)
N(5)	7077(1)	2714(2)	1890(1)	49(1)

O(111)	7940(1)	5476(2)	305(1)	71(1)
O(112)	7386(2)	6228(2)	1535(1)	107(1)

Table 73 : Bond lengths [A] and angles [deg] for $C_{18}H_{26}N_2O_2$.

C(1)-N(2)	1.333(2)	C(51)-C(53)	1.515(3)
C(1)-N(5)	1.334(2)	C(51)-C(52)	1.514(3)
C(3)-C(4)	1.359(3)	C(111)-O(112)	1.233(3)
C(3)-N(2)	1.396(2)	C(111)-O(111)	1.252(2)
C(3)-C(31)	1.491(3)		
C(4)-N(5)	1.394(2)	N(2)-C(1)-N(5)	108.28(17)
C(4)-C(41)	1.484(3)	C(4)-C(3)-N(2)	106.57(15)
C(11)-C(12)	1.384(3)	C(4)-C(3)-C(31)	130.33(19)
C(11)-C(16)	1.395(3)	N(2)-C(3)-C(31)	123.08(18)
C(11)-C(111)	1.516(3)	C(3)-C(4)-N(5)	106.86(16)
C(12)-C(13)	1.388(3)	C(3)-C(4)-C(41)	130.56(18)
C(13)-C(14)	1.378(4)	N(5)-C(4)-C(41)	122.51(17)
C(14)-C(15)	1.375(3)	C(12)-C(11)-C(16)	118.53(18)
C(15)-C(16)	1.383(3)	C(12)-C(11)-C(111)	121.46(18)
C(21)-N(2)	1.482(2)	C(16)-C(11)-C(111)	120.01(16)
C(21)-C(23)	1.502(4)	C(11)-C(12)-C(13)	120.8(2)
C(21)-C(22)	1.504(3)	C(14)-C(13)-C(12)	119.8(2)
C(51)-N(5)	1.486(2)	C(13)-C(14)-C(15)	120.1(2)

C(14)-C(15)-C(16)	120.1(2)	O(112)-C(111)-C(11)	117.02(19)
C(15)-C(16)-C(11)	120.54(18)	O(111)-C(111)-C(11)	117.71(17)
N(2)-C(21)-C(23)	109.71(19)	C(1)-N(2)-C(3)	109.20(15)
N(2)-C(21)-C(22)	110.03(18)	C(1)-N(2)-C(21)	124.12(16)
C(23)-C(21)-C(22)	112.4(3)	C(3)-N(2)-C(21)	126.66(15)
N(5)-C(51)-C(53)	108.53(15)	C(1)-N(5)-C(4)	109.09(15)
N(5)-C(51)-C(52)	110.27(16)	C(1)-N(5)-C(51)	124.05(16)
C(53)-C(51)-C(52)	112.44(19)	C(4)-N(5)-C(51)	126.12(15)
O(112)-C(111)-O(111)	125.2(2)		

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Table 74 : Anisotropic displacement parameters (A^2 x 10^3) for $C_{18}H_{26}N_2O_2$ The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	40(1)	65(1)	49(1)	11(1)	4(1)	1(1)
C(3)	50(1)	60(1)	42(1)	9(1)	12(1)	7(1)
C(4)	51(1)	58(1)	42(1)	1(1)	15(1)	1(1)
C(11)	38(1)	62(1)	36(1)	-1(1)	3(1)	-5(1)
C(12)	52(1)	74(1)	44(1)	-2(1)	13(1)	-3(1)
C(13)	79(2)	88(2)	43(1)	9(1)	17(1)	-6(1)
C(14)	88(2)	70(2)	58(1)	13(1)	9(1)	4(1)
C(15)	73(1)	69(1)	59(1)	-1(1)	12(1)	9(1)
C(16)	54(1)	66(1)	42(1)	-4(1)	9(1)	-2(1)
C(21)	41(1)	77(1)	54(1)	10(1)	1(1)	-1(1)

C(22)	53(1)	105(2)	77(2)	4(2)	21(1)	-12(1)
C(23)	64(2)	118(3)	107(2)	-48(2)	15(2)	-22(2)
C(31)	59(1)	83(2)	48(1)	18(1)	7(1)	6(1)
C(41)	64(1)	72(1)	51(1)	4(1)	17(1)	-10(1)
C(51)	41(1)	60(1)	45(1)	1(1)	1(1)	-2(1)
C(52)	58(1)	76(2)	46(1)	7(1)	2(1)	-7(1)
C(53)	44(1)	78(2)	68(1)	-15(1)	-1(1)	7(1)
C(111)	52(1)	64(1)	44(1)	-2(1)	4(1)	-7(1)
N(2)	40(1)	65(1)	47(1)	13(1)	5(1)	4(1)
N(5)	41(1)	59(1)	42(1)	7(1)	5(1)	1(1)
O(111)	72(1)	64(1)	79(1)	3(1)	29(1)	6(1)
O(112)	194(2)	76(1)	59(1)	14(1)	54(1)	17(1)

Table 75 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for $C_{18}H_{26}N_2O_2$.

	x	y	z	U(eq)
H(1)	7974(19)	4070(2)	1402(17)	69(6)
H(12)	7780(2)	7060(2)	-1027(18)	75(7)
H(13)	7280(2)	8950(2)	-1820(2)	84(8)
H(14)	6600(2)	10640(3)	-1110(2)	98(9)
H(15)	6270(3)	10330(3)	440(2)	100(9)
H(16)	6700(2)	8370(2)	1254(18)	72(6)
H(21)	10530(2)	3180(2)	3566(18)	71(6)

H(22A)	11260(2)	4120(2)	2401(19)	85(7)
H(22B)	9960(3)	4350(3)	1600(2)	101(9)
H(22C)	10550(3)	2900(4)	1900(3)	132(13)
H(23A)	10740(3)	5370(3)	3690(2)	111(10)
H(23B)	9670(3)	4930(3)	4060(2)	111(10)
H(23C)	9420(4)	5520(4)	2860(3)	160(16)
H(31A)	10010(2)	1400(2)	4231(18)	80(7)
H(31B)	9500(2)	2420(3)	4800(2)	96(9)
H(31C)	8830(2)	1070(3)	4480(2)	86(8)
H(41A)	5850(3)	1410(2)	2959(19)	86(8)
H(41B)	6970(2)	610(2)	3539(19)	74(7)

7.3.16 Crystal data for $C_{13}H_{22}N_2O_4$ (**43**)

Table 76 : Crysata data and structure refinment for $C_{13}H_{22}N_2O_4$

Empirical formula	$C_{13}H_{22}N_2O_4$	
Formula weight	270.33	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 11.780(5) Å	alpha= 90°.
	b = 11.460(3) Å	beta= 105.69(3)°.
	c = 10.772(2) Å	gamma= 90°.
Volume	1399.9(10) Å^3	

Z	4
Density (calculated)	1.283 Mg/m^3
Absorption coefficient	0.095 mm^{-1}
F(000)	584
Theta range for data collection	3.49 to 31.11°.
Index ranges	-17<=h<=16, -1<=k<=16, -1<=l<=15
Reflections collected	2769
Independent reflections	1148 [R(int) = 0.0476]
Completeness to theta = 31.11°	49.6 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2548 / 2 / 260
Goodness-of-fit on F^2	1.025
Final R indices [I>2sigma(I)]	R1 = 0.0531, wR2 = 0.1362
R indices (all data)	R1 = 0.0811, wR2 = 0.1555
Extinction coefficient	0.000(3)
Largest diff. peak and hole	0.362 and -0.334 e.Å^{-3}

Table 77 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3)for $C_{13}H_{22}N_2O_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	841(2)	9855(2)	8219(2)	50(1)
O(1)	4826(2)	25(2)	1147(2)	63(1)

O(2)	5609(4)	1778(3)	1541(3)	109(1)
C(1)	0	10526(4)	7500	52(1)
C(2)	525(3)	8696(3)	7956(3)	49(1)
C(3)	1242(3)	7705(3)	8637(4)	60(1)
C(4)	1923(3)	10272(3)	9178(3)	56(1)
C(5)	1635(4)	10530(7)	10430(4)	98(2)
C(6)	2449(4)	11290(4)	8667(4)	73(1)
C(7)	5146(2)	921(3)	1849(3)	51(1)

Table 78 :   Bond lengths [Å] and angles [°] for $C_{13}H_{22}N_2O_4$

N(1)-C(1)	1.323(4)	C(1)-N(1)-C(4)	125.7(3)
N(1)-C(2)	1.388(4)	C(2)-N(1)-C(4)	125.6(3)
N(1)-C(4)	1.482(4)	N(1)#1-C(1)-N(1)	109.0(4)
O(1)-C(7)	1.269(4)	C(2)#1-C(2)-N(1)	106.80(16)
O(2)-C(7)	1.211(4)	C(2)#1-C(2)-C(3)	129.99(19)
C(1)-N(1)#1	1.323(4)	N(1)-C(2)-C(3)	123.2(3)
C(2)-C(2)#1	1.351(6)	N(1)-C(4)-C(6)	110.9(3)
C(2)-C(3)	1.484(4)	N(1)-C(4)-C(5)	109.2(3)
C(4)-C(6)	1.493(5)	C(6)-C(4)-C(5)	113.1(4)
C(4)-C(5)	1.505(6)	O(2)-C(7)-O(1)	125.1(3)
C(7)-C(7)#2	1.531(6)	O(2)-C(7)-C(7)#2	118.6(2)
		O(1)-C(7)-C(7)#2	116.2(2)
C(1)-N(1)-C(2)	108.7(3)		

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,-z+3/2   #2 -x+1,y,-z+1/2

Table 79 : Anisotropic displacement parameters (Å^{2}x 10^3) for $C_{13}H_{22}N_2O_4$. The anisotropic displacement factor exponent takes the form: $-2\square^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	44(1)	49(1)	55(2)	1(1)	10(1)	0(1)
O(1)	81(2)	54(1)	62(2)	-7(1)	31(1)	-13(1)
O(2)	168(3)	89(2)	87(2)	-23(2)	64(2)	-65(2)
C(1)	49(2)	46(2)	56(3)	0	7(2)	0
C(2)	45(1)	51(2)	51(2)	2(1)	15(1)	1(1)
C(3)	57(2)	53(2)	69(2)	9(2)	18(2)	8(1)
C(4)	43(2)	65(2)	58(2)	1(2)	8(1)	-5(1)
C(5)	66(3)	174(5)	54(2)	-19(3)	14(2)	-39(3)
C(6)	63(2)	71(2)	77(3)	6(2)	6(2)	-17(2)
C(7)	48(2)	50(2)	54(2)	-1(1)	11(1)	-6(1)

Table 80 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{13}H_{22}N_2O_4$.

	x	y	z	U(eq)
H(1)	0	11310(40)	7500	44(11)
H(3A)	880(40)	6980(40)	8310(40)	77(12)

H(4A)	2460(30)	9650(30)	9320(30)	54(9)
H(5A)	1170(60)	9850(50)	10750(60)	130(20)
H(6A)	1920(50)	11950(50)	8410(50)	97(14)
H(1B)	4910(140)	530(130)	390(150)	320(70)
H(3B)	2030(50)	7750(40)	8510(50)	99(14)
H(5B)	2380(40)	10740(40)	11070(50)	92(13)
H(6B)	3170(40)	11570(40)	9310(40)	82(12)
H(3C)	1290(40)	7680(40)	9610(40)	80(12)
H(5C)	940(70)	11160(60)	10180(60)	140(20)
H(6C)	2570(50)	11110(50)	7780(60)	116(18)

7.3.17 Crystal data for $C_{22}H_{40}N_4AuCl$ (**44**)

Table 81 : Crystal data and structure refinement for $C_{22}H_{40}N_4AuCl$.

Empirical formula	$C_{23}H_{41.50}AuClN_{4.50}$
Formula weight	613.52
Temperature	223(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, P212121
Unit cell dimensions	a = 9.240(2) A alpha = 90 deg.
	b = 14.243(4) A beta = 90 deg.
	c = 23.759(4) A gamma = 90 deg.
Volume	3126.8(12) A^3
Z, Calculated density	4, 1.303 Mg/m^3

Absorption coefficient	4.804 mm^-1
F(000)	1228
Crystal size	0.50 x 0.30 x 0.10 mm
Theta range for data collection	3.14 to 26.03 deg.
Index ranges	-1<=h<=11, -1<=k<=17, -29<=l<=29
Reflections collected / unique	8186 / 6129 [R(int) = 0.0384]
Completeness to 2theta = 26.03	99.3%
Absorption correction	DIFABS
Max. and min. transmission	0.714 and 0.259
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6129 / 0 / 265
Goodness-of-fit on F^2	1.022
Final R indices [I>2sigma(I)]	R1 = 0.0483, wR2 = 0.1223
R indices (all data)	R1 = 0.0884, wR2 = 0.1364
Absolute structure parameter	0.046(16)
Largest diff. peak and hole	1.540 and -0.882 e.A^-3

Table 82 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for $C_{22}H_{40}N_4AuCl$.U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
C(11)	5511(12)	9730(7)	3419(4)	36(2)
C(13)	4696(12)	9940(8)	4314(4)	39(3)
C(14)	5808(14)	10536(6)	4242(4)	40(2)

C(21)	5864(14)	8745(7)	1830(4)	37(2)
C(23)	5385(11)	8499(8)	914(4)	35(3)
C(24)	6475(12)	7939(8)	1056(4)	35(2)
C(121)	3404(14)	8724(11)	3691(6)	62(4)
C(122)	2048(16)	9167(14)	3505(7)	86(5)
C(123)	3570(3)	7962(19)	4054(11)	62(9)
C(124)	3750(6)	7630(3)	3570(2)	130(2)
C(131)	3831(16)	9767(11)	4837(6)	67(4)
C(141)	6519(16)	11200(10)	4633(5)	64(4)
C(151)	7470(13)	11004(8)	3449(5)	48(3)
C(152)	6923(18)	11623(12)	2974(8)	97(6)
C(153)	8751(18)	10453(13)	3350(11)	123(9)
C(221)	3815(13)	9688(9)	1393(5)	47(3)
C(222)	2767(14)	9503(10)	1866(6)	63(4)
C(223)	4451(18)	10709(10)	1397(7)	76(4)
C(231)	4671(13)	8677(8)	350(4)	45(3)
C(241)	7311(12)	7223(7)	724(4)	39(2)
C(251)	7923(13)	7609(8)	1936(4)	42(3)
C(252)	7242(15)	6852(10)	2301(7)	80(5)
C(253)	8900(13)	8239(10)	2246(5)	59(3)
N(12)	4531(10)	9445(6)	3807(4)	42(2)
N(15)	6293(10)	10420(6)	3692(4)	37(2)
N(22)	4996(10)	8984(6)	1394(3)	38(2)
N(25)	6768(10)	8118(6)	1625(3)	34(2)

Cl(2)	952(3)	8110(2)	846(1)	51(1)
Au(1)	5729(1)	9263(1)	2627(1)	40(1)
C(1)	3620(3)	4863(15)	8919(8)	38(5)
C(2)	4310(3)	4086(17)	8708(10)	55(6)
N(3)	4840(4)	3520(3)	8455(14)	109(11)

Table 83 : Bond lengths [A] and angles [deg] for $C_{22}H_{40}N_4AuCl$.

C(11)-N(12)	1.354(14)	C(121)-C(122)	1.47(2)
C(11)-N(15)	1.382(13)	C(121)-N(12)	1.488(16)
C(11)-Au(1)	2.007(10)	C(121)-C(124)	1.61(5)
C(13)-C(14)	1.343(15)	C(151)-C(153)	1.44(2)
C(13)-N(12)	1.405(13)	C(151)-N(15)	1.487(14)
C(13)-C(131)	1.499(17)	C(151)-C(152)	1.519(19)
C(14)-N(15)	1.390(13)	C(221)-N(22)	1.483(14)
C(14)-C(141)	1.481(15)	C(221)-C(222)	1.506(18)
C(21)-N(25)	1.316(13)	C(221)-C(223)	1.568(19)
C(21)-N(22)	1.354(13)	C(251)-C(253)	1.469(16)
C(21)-Au(1)	2.036(10)	C(251)-N(25)	1.487(13)
C(23)-C(24)	1.328(15)	C(251)-C(252)	1.520(16)
C(23)-N(22)	1.380(12)	C(1)-C(2)	1.37(3)
C(23)-C(231)	1.515(15)	C(2)-N(3)	1.12(4)
C(24)-N(25)	1.401(13)		
C(24)-C(241)	1.505(14)	N(12)-C(11)-N(15)	104.1(8)
C(121)-C(123)	1.40(3)	N(12)-C(11)-Au(1)	127.3(7)

N(15)-C(11)-Au(1)	128.6(8)
C(14)-C(13)-N(12)	106.9(9)
C(14)-C(13)-C(131)	128.1(10)
N(12)-C(13)-C(131)	124.8(10)
C(13)-C(14)-N(15)	106.9(9)
C(13)-C(14)-C(141)	131.6(10)
N(15)-C(14)-C(141)	121.5(10)
N(25)-C(21)-N(22)	105.3(8)
N(25)-C(21)-Au(1)	129.0(8)
N(22)-C(21)-Au(1)	125.8(8)
C(24)-C(23)-N(22)	106.7(9)
C(24)-C(23)-C(231)	130.9(9)
N(22)-C(23)-C(231)	122.2(10)
C(23)-C(24)-N(25)	106.4(9)
C(23)-C(24)-C(241)	131.6(10)
N(25)-C(24)-C(241)	122.0(9)
C(123)-C(121)-C(122)	127.7(18)
C(123)-C(121)-N(12)	110.3(16)
C(122)-C(121)-N(12)	110.8(12)
C(123)-C(121)-C(124)	48.4(19)
C(122)-C(121)-C(124)	122(2)
N(12)-C(121)-C(124)	124(2)
C(153)-C(151)-N(15)	111.1(11)
C(153)-C(151)-C(152)	118.0(14)
N(15)-C(151)-C(152)	111.8(10)
N(22)-C(221)-C(222)	110.7(10)
N(22)-C(221)-C(223)	110.6(10)
C(222)-C(221)-C(223)	113.5(11)
C(253)-C(251)-N(25)	113.1(9)
C(253)-C(251)-C(252)	113.7(10)
N(25)-C(251)-C(252)	109.4(10)
C(11)-N(12)-C(13)	111.1(9)
C(11)-N(12)-C(121)	123.3(9)
C(13)-N(12)-C(121)	125.6(10)
C(11)-N(15)-C(14)	110.9(9)
C(11)-N(15)-C(151)	126.7(9)
C(14)-N(15)-C(151)	122.4(9)
C(21)-N(22)-C(23)	110.6(9)
C(21)-N(22)-C(221)	127.4(9)
C(23)-N(22)-C(221)	122.0(9)
C(21)-N(25)-C(24)	110.9(9)
C(21)-N(25)-C(251)	127.0(8)
C(24)-N(25)-C(251)	122.0(8)
C(11)-Au(1)-C(21)	177.1(5)
N(3)-C(2)-C(1)	169(3)

Table 84 : Anisotropic displacement parameters (A^2 x 10^3) for $C_{22}H_{40}N_4AuCl$. The anisotropic displacement factor exponent takes the form:-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(11)	32(7)	39(6)	37(5)	-3(4)	-5(5)	-7(5)
C(13)	38(7)	36(6)	42(5)	-12(5)	7(5)	0(5)
C(14)	46(6)	32(6)	40(5)	-5(4)	0(5)	1(6)
C(21)	44(7)	26(5)	41(5)	3(4)	-6(6)	-2(6)
C(23)	19(6)	47(6)	39(5)	-13(5)	1(4)	-6(5)
C(24)	32(6)	35(6)	39(5)	5(5)	2(5)	-4(5)
C(121)	37(8)	65(9)	83(10)	-14(8)	-2(7)	2(7)
C(122)	45(8)	118(14)	95(10)	-13(11)	-10(8)	-25(11)
C(131)	54(9)	81(10)	68(8)	0(7)	11(7)	1(8)
C(141)	80(10)	74(9)	38(6)	-25(6)	13(6)	-23(8)
C(151)	35(7)	49(8)	60(6)	7(5)	7(5)	-8(6)
C(152)	62(11)	71(10)	158(17)	51(11)	5(11)	-15(9)
C(153)	41(9)	90(14)	240(3)	50(15)	27(13)	-11(9)
C(221)	41(7)	52(7)	49(6)	-11(5)	-4(5)	13(6)
C(222)	40(7)	74(10)	76(8)	-8(7)	-8(7)	17(7)
C(223)	80(11)	52(8)	95(10)	19(8)	9(9)	31(10)
C(231)	52(8)	40(6)	43(5)	-6(5)	1(5)	-2(6)
C(241)	35(6)	34(6)	49(5)	-10(5)	0(5)	2(5)
C(251)	38(7)	39(6)	49(6)	-2(5)	-7(5)	8(5)

C(252)	54(8)	69(9)	118(12)	45(9)	8(9)	9(8)
C(253)	44(8)	68(8)	63(8)	-8(6)	-12(6)	7(6)
N(12)	40(6)	39(5)	46(4)	-9(4)	-2(4)	-3(5)
N(15)	31(5)	35(5)	44(4)	-7(4)	4(4)	-1(4)
N(22)	35(5)	40(5)	38(4)	-8(4)	-3(4)	4(4)
N(25)	37(5)	29(5)	38(4)	-10(4)	0(4)	5(4)
Cl(2)	44(2)	61(2)	49(1)	-1(1)	-5(1)	-3(2)
Au(1)	40(1)	41(1)	39(1)	-9(1)	-3(1)	4(1)

Table 85 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for $C_{22}H_{40}N_4AuCl$.

	x	y	z	U(eq)
H(121)	3762	8455	3333	74
H(12A)	1333	8686	3428	129
H(12B)	2224	9528	3165	129
H(12C)	1692	9581	3798	129
H(12D)	4587	7811	4091	92
H(12E)	3055	7423	3903	92
H(12F)	3175	8120	4421	92
H(12G)	4705	7480	3717	190
H(12H)	3724	7514	3170	190
H(12I)	3033	7244	3759	190
H(13A)	3108	9289	4764	101

H(13B)	3356	10344	4951	101
H(13C)	4469	9557	5136	101
H(14A)	7290	11528	4438	96
H(14B)	6919	10857	4950	96
H(14C)	5814	11652	4768	96
H(151)	7741	11446	3753	57
H(15A)	6065	11957	3097	145
H(15B)	6688	11237	2650	145
H(15C)	7668	12072	2871	145
H(15D)	8977	10091	3685	185
H(15E)	9555	10866	3263	185
H(15F)	8585	10030	3037	185
H(221)	3275	9611	1036	57
H(22A)	2409	8865	1839	95
H(22B)	1962	9938	1840	95
H(22C)	3255	9588	2224	95
H(22D)	5108	10785	1082	113
H(22E)	4970	10814	1747	113
H(22F)	3668	11160	1366	113
H(23A)	3895	9128	396	67
H(23B)	4281	8093	205	67
H(23C)	5382	8923	88	67
H(24A)	8049	6944	961	59
H(24B)	7767	7526	404	59

H(24C)	6658	6738	592	59
H(251)	8520	7283	1651	50
H(25A)	6627	6456	2071	120
H(25B)	6667	7144	2595	120
H(25C)	7998	6474	2471	120
H(25D)	9313	8694	1988	88
H(25E)	9671	7874	2416	88
H(25F)	8362	8564	2537	88
H(1A)	4327	5317	9046	57
H(1B)	3007	4679	9232	57
H(1C)	3024	5142	8626	57

7.3.18 Crystal data for $C_{22}H_{40}N_4S_2Cl_2$ (**45**)

Table 86 : Crystal data and structure refinement for $C_{22}H_{40}N_4S_2Cl_2$.

Empirical formula	$C_{10}H_{20}Cl_{0.36}N_2O_{0.36}S$
Formula weight	219.05
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 11.509(8) A alpha = 90 deg.
	b = 19.946(5) A beta = 99.66(3) deg.
	c = 12.394(2) A gamma = 90 deg.
Volume	2805(2) A^3

Z, Calculated density	11, 1.427 Mg/m^3
Absorption coefficient	0.375 mm^-1
F(000)	1310
Crystal size	0.05 x 0.4 x 0.15 mm
Theta range for data collection	2.04 to 27.67 deg.
Limiting indices	-15<=h<=1, -25<=k<=1, -16<=l<=16
Reflections collected / unique	7829 / 6459 [R(int) = 0.0603]
Completeness to theta = 27.67	98.7 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6459 / 0 / 314
Goodness-of-fit on F^2	1.021
Final R indices [I>2sigma(I)]	R1 = 0.0721, wR2 = 0.1513
R indices (all data)	R1 = 0.1417, wR2 = 0.1837
Extinction coefficient	0.0005(5)
Largest diff. peak and hole	0.766 and -0.843 e.A^-3

Table 87 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for $C_{22}H_{40}N_4S_2Cl_2$. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S(3)	8328(1)	2502(1)	7378(1)	39(1)
S(4)	7140(1)	2800(1)	8404(1)	41(1)
N(1)	8198(3)	2108(2)	10228(3)	29(1)

N(3)	10358(3)	3156(2)	8224(3)	29(1)
N(4)	9208(3)	3766(2)	7045(3)	33(1)
C(19)	10370(4)	4860(2)	7114(4)	41(1)
N(2)	8173(3)	3192(2)	10470(4)	38(1)
C(3)	8706(4)	2911(2)	11447(4)	37(1)
C(1)	7880(4)	2692(2)	9731(4)	32(1)
C(4)	7919(4)	1443(2)	9704(4)	33(1)
C(2)	8715(4)	2234(2)	11306(4)	30(1)
C(6)	6915(4)	1116(2)	10142(4)	45(1)
C(5)	9000(4)	996(2)	9758(4)	43(1)
C(15)	11162(4)	2671(2)	10035(4)	42(1)
C(12)	9319(4)	3167(2)	7546(4)	33(1)
C(7)	9210(4)	1709(2)	12099(4)	41(1)
C(13)	10806(4)	2542(2)	8841(4)	34(1)
C(18)	10199(4)	4152(2)	7424(4)	30(1)
C(16)	10931(3)	3764(2)	8158(3)	27(1)
C(14)	11742(4)	2215(2)	8286(4)	47(1)
C(17)	12074(4)	3946(2)	8825(4)	36(1)
C(21)	7311(5)	4337(3)	6535(5)	60(2)
C(11)	8956(4)	4283(2)	9957(5)	48(1)
C(22)	8590(5)	4094(3)	5116(4)	49(1)
C(10)	6763(4)	4146(3)	9895(6)	60(2)
C(9)	7959(5)	3936(2)	10335(6)	60(2)
C(8)	9198(4)	3312(2)	12438(4)	46(1)

C(20)	8213(4)	3919(3)	6146(4)	51(1)
Cl(1)	9770(1)	2262(1)	5411(1)	47(1)
Cl(2)	7047(1)	917(1)	6925(1)	42(1)
Cl(3)	11269(1)	321(1)	7734(1)	59(1)
O(1)	9343(3)	906(2)	6274(3)	45(1)

Table 88 : Bond lengths [A] and angles [deg] for $C_{22}H_{40}N_4S_2Cl_2$.

S(3)-C(12)	1.738(5)	C(3)-C(2)	1.362(6)
S(3)-S(4)	2.103(2)	C(3)-C(8)	1.495(6)
S(4)-C(1)	1.733(5)	C(4)-C(6)	1.506(6)
N(1)-C(1)	1.339(5)	C(4)-C(5)	1.522(6)
N(1)-C(2)	1.391(5)	C(2)-C(7)	1.483(6)
N(1)-C(4)	1.489(5)	C(15)-C(13)	1.490(6)
N(3)-C(12)	1.342(5)	C(13)-C(14)	1.519(7)
N(3)-C(16)	1.388(5)	C(18)-C(16)	1.370(6)
N(3)-C(13)	1.489(5)	C(16)-C(17)	1.477(6)
N(4)-C(12)	1.342(5)	C(21)-C(20)	1.473(7)
N(4)-C(18)	1.391(5)	C(11)-C(9)	1.482(7)
N(4)-C(20)	1.490(6)	C(22)-C(20)	1.458(7)
C(19)-C(18)	1.486(6)	C(10)-C(9)	1.454(7)
N(2)-C(1)	1.359(6)		
N(2)-C(3)	1.381(6)	C(12)-S(3)-S(4)	01.11(17)
N(2)-C(9)	1.508(6)	C(1)-S(4)-S(3)	105.84(16)

C(1)-N(1)-C(2)	108.9(3)	C(3)-C(2)-N(1)	106.9(4)
C(1)-N(1)-C(4)	123.5(3)	C(3)-C(2)-C(7)	128.8(4)
C(2)-N(1)-C(4)	127.3(3)	N(1)-C(2)-C(7)	124.3(4)
C(12)-N(3)-C(16)	109.4(3)	N(3)-C(12)-N(4)	108.0(4)
C(12)-N(3)-C(13)	122.0(4)	N(3)-C(12)-S(3)	124.2(3)
C(16)-N(3)-C(13)	128.4(3)	N(4)-C(12)-S(3)	127.9(3)
C(12)-N(4)-C(18)	109.1(3)	N(3)-C(13)-C(15)	112.4(4)
C(12)-N(4)-C(20)	122.0(4)	N(3)-C(13)-C(14)	109.4(4)
C(18)-N(4)-C(20)	128.5(4)	C(15)-C(13)-C(14)	115.2(4)
C(1)-N(2)-C(3)	108.4(4)	C(16)-C(18)-N(4)	106.8(4)
C(1)-N(2)-C(9)	129.1(4)	C(16)-C(18)-C(19)	128.0(4)
C(3)-N(2)-C(9)	122.5(4)	N(4)-C(18)-C(19)	125.1(4)
C(2)-C(3)-N(2)	107.6(4)	C(18)-C(16)-N(3)	106.7(4)
C(2)-C(3)-C(8)	128.7(5)	C(18)-C(16)-C(17)	128.5(4)
N(2)-C(3)-C(8)	123.7(4)	N(3)-C(16)-C(17)	124.7(4)
N(1)-C(1)-N(2)	108.1(4)	C(10)-C(9)-C(11)	118.8(5)
N(1)-C(1)-S(4)	126.7(3)	C(10)-C(9)-N(2)	116.9(4)
N(2)-C(1)-S(4)	125.1(3)	C(11)-C(9)-N(2)	111.9(4)
N(1)-C(4)-C(6)	110.5(4)	C(22)-C(20)-C(21)	118.7(5)
N(1)-C(4)-C(5)	112.8(3)	C(22)-C(20)-N(4)	113.6(4)
C(6)-C(4)-C(5)	113.7(4)	C(21)-C(20)-N(4)	111.5(4)

Table 90 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for$C_{22}H_{42}N_4S_2Cl_2$.

	x	y	z	U(eq)
H(19A)	11162	5008	7443	61
H(19B)	9782	5144	7379	61
H(19C)	10279	4896	6316	61
H(4)	7630	1532	8911	39
H(6A)	7199	941	10878	68
H(6B)	6599	747	9659	68
H(6C)	6293	1447	10175	68
H(5A)	9662	1262	9589	64
H(5B)	8827	632	9225	64
H(5C)	9205	807	10496	64
H(15A)	11942	2883	10168	62
H(15B)	11193	2246	10435	62
H(15C)	10587	2970	10287	62
H(7A)	9948	1540	11905	61
H(7B)	8645	1340	12077	61
H(7C)	9366	1900	12837	61
H(13)	10130	2221	8766	41
H(14A)	11495	2231	7490	70
H(14B)	11845	1747	8523	70
H(14C)	12489	2456	8487	70
H(17A)	11956	4061	9568	54

H(17B)	12406	4333	8494	54
H(17C)	12617	3566	8855	54
H(21A)	7675	4751	6857	90
H(21B)	6969	4090	7089	90
H(21C)	6691	4448	5918	90
H(11A)	9015	4133	9215	72
H(11B)	9690	4177	10452	72
H(11C)	8823	4768	9955	72
H(22A)	7896	4158	4548	74
H(22B)	9078	3732	4898	74
H(22C)	9049	4510	5209	74
H(10A)	6634	4113	9095	89
H(10B)	6648	4611	10109	89
H(10C)	6201	3856	10185	89
H(9)	8043	4089	11113	72
H(8A)	9455	3009	13054	70
H(8B)	8589	3615	12621	70
H(8C)	9871	3576	12288	70
H(20)	7812	3477	5989	62
H(41)	8340(40)	950(20)	6690(40)	55(15)
H(40)	9990(70)	560(40)	6840(70)	130(30)
H(42)	9510(60)	1370(40)	6120(60)	100(30)

7.3.19 Crysta data for $C_8H_{14}Cl_2N_2$ (**46**)

Table 91 : Crystal data and structure refinement for $C_8H_{14}Cl_2N_2$.

Empirical formula	$C_8H_{14}Cl_2N_2$	
Formula weight	209.11	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 8.5733(17) Å	alpha= 90°.
	b = 9.2192(9) Å	beta= 104.686(12)°.
	c = 13.542(3) Å	gamma = 90°.
Volume	1035.4(3) $Å^3$	
Z	4	
Density (calculated)	1.342 Mg/m^3	
Absorption coefficient	0.578 mm^{-1}	
F(000)	440	
Theta range for data collection	2.46 to 27.53°.	
Index ranges	-11<=h<=11, -11<=k<=11, -17<=l<=17	
Reflections collected	9156	
Independent reflections	2380 [R(int) = 0.0411]	
Completeness to theta = 27.53°	99.9 %	
Absorption correctionEmpirical		
Max. and min. transmission	0.3319 and 0.3018	
Refinement method	Full-matrix least-squares on F^2	

Data / restraints / parameters	2380 / 0 / 166
Goodness-of-fit on F^2	1.867
Final R indices [I>2sigma(I)]	R1 = 0.0369, wR2 = 0.0942
R indices (all data)	R1 = 0.0470, wR2 = 0.1045
Extinction coefficient	0.005(3)
Largest diff. peak and hole	0.437 and -0.431 e.Å^{-3}

Table 92 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3)for $C_8H_{14}Cl_2N_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	8154(1)	4214(1)	3702(1)	50(1)
Cl(2)	2674(1)	677(1)	8272(1)	33(1)
N(1)	6330(2)	944(2)	4182(1)	27(1)
N(2)	8231(2)	1535(2)	5512(1)	26(1)
C(1)	6830(2)	3628(2)	4468(1)	33(1)
C(2)	7111(2)	2068(2)	4716(1)	26(1)
C(3)	6960(2)	-346(2)	4654(1)	26(1)
C(4)	8161(2)	29(2)	5493(1)	25(1)
C(5)	5043(2)	1043(2)	3237(1)	36(1)
C(6)	6376(2)	-1795(2)	4244(2)	35(1)
C(7)	9241(2)	-894(2)	6280(1)	32(1)
C(8)	9383(2)	2412(2)	6262(2)	37(1)

Table 93 : Bond lengths [Å] and angles [°] for $C_8H_{14}Cl_2N_2$.

Cl(1)-C(1)	1.803(2)	C(3)-N(1)-C(5)	124.97(15)
N(1)-C(2)	1.342(2)	C(2)-N(2)-C(4)	109.45(13)
N(1)-C(3)	1.392(2)	C(2)-N(2)-C(8)	124.98(15)
N(1)-C(5)	1.465(2)	C(4)-N(2)-C(8)	125.53(14)
N(2)-C(2)	1.341(2)	C(2)-C(1)-Cl(1)	109.37(13)
N(2)-C(4)	1.389(2)	N(2)-C(2)-N(1)	107.90(14)
N(2)-C(8)	1.468(2)	N(2)-C(2)-C(1)	125.41(15)
C(1)-C(2)	1.482(2)	N(1)-C(2)-C(1)	126.67(15)
C(3)-C(4)	1.370(2)	C(4)-C(3)-N(1)	106.74(14)
C(3)-C(6)	1.485(2)	C(4)-C(3)-C(6)	130.45(16)
C(4)-C(7)	1.489(2)	N(1)-C(3)-C(6)	122.79(15)
		C(3)-C(4)-N(2)	106.67(14)
C(2)-N(1)-C(3)	109.23(13)	C(3)-C(4)-C(7)	130.50(15)
C(2)-N(1)-C(5)	125.79(15)	N(2)-C(4)-C(7)	122.83(14)

Symmetry transformations used to generate equivalent atoms:

Table 94 : Anisotropic displacement parameters ($Å^2 x 10^3$) for $C_8H_{14}Cl_2N_2$. The anisotropic displacement factor exponent takes the form: -2□^{2}[h^2 $a^{*2}U^{11}$ + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	58(1)	37(1)	60(1)	16(1)	22(1)	2(1)
Cl(2)	31(1)	30(1)	36(1)	0(1)	5(1)	-4(1)
N(1)	26(1)	29(1)	23(1)	-1(1)	3(1)	3(1)

N(2)	24(1)	24(1)	27(1)	1(1)	2(1)	1(1)
C(1)	34(1)	27(1)	35(1)	3(1)	4(1)	7(1)
C(2)	25(1)	26(1)	26(1)	0(1)	4(1)	3(1)
C(3)	28(1)	25(1)	26(1)	1(1)	7(1)	0(1)
C(4)	27(1)	22(1)	27(1)	1(1)	7(1)	1(1)
C(5)	32(1)	43(1)	27(1)	-5(1)	-3(1)	9(1)
C(6)	39(1)	31(1)	35(1)	-7(1)	7(1)	-6(1)
C(7)	33(1)	27(1)	31(1)	6(1)	2(1)	3(1)
C(8)	33(1)	27(1)	42(1)	-4(1)	-8(1)	-1(1)

Table 95 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for $C_8H_{14}Cl_2N_2$.

	x	y	z	U(eq)
H(8C)	9980(50)	2950(40)	5950(30)	68(10)
H(5C)	4290(50)	300(40)	3290(30)	65(10)
H(1A)	6970(30)	4310(20)	5078(18)	26(5)
H(8A)	9950(40)	1840(40)	6710(30)	57(8)
H(7C)	9100(40)	-650(40)	6920(30)	56(9)
H(1B)	5700(30)	3920(30)	4120(20)	34(6)
H(7A)	10330(50)	-710(40)	6280(30)	63(10)
H(6A)	5250(40)	-1880(30)	4120(20)	51(8)
H(5B)	5480(40)	850(30)	2730(30)	47(8)
H(7B)	8960(40)	-1840(40)	6140(30)	51(8)

H(6B)	6650(40)	-1970(30)	3640(20)	42(7)
H(8B)	8860(50)	3200(40)	6500(30)	64(10)
H(6C)	6850(40)	-2580(40)	4700(30)	60(9)
H(5A)	4570(50)	1870(40)	3220(30)	65(10)

7.3.20 Crystal data for $C_8H_{14}Br_2N_2$ (**47**)

Table 96 : Crystal data and structure refinement for $C_8H_{14}Br_2N_2$

Empirical formula	$C_8H_{14}Br_2N_2$	
Formula weight	298.03	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Rhombohedral	
Space group	R-3	
Unit cell dimensions	a = 20.494(2) Å	alpha= 90°.
	b = 20.494(2) Å	beta= 90°.
	c = 15.291(4) Å	gamma = 120°.
Volume	5561.9(15) $Å^3$	
Z	18	
Density (calculated)	2.135 Mg/m^3	
Absorption coefficient	8.691 mm^{-1}	
F(000)	3504	
Theta range for data collection	2.65 to 20.00°.	
Index ranges	-19<=h<=19, -19<=k<=19, -14<=l<=14	
Reflections collected	6123	

Independent reflections	1157 [R(int) = 0.1855]
Completeness to theta = 20.00°	99.8 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1157 / 0 / 128
Goodness-of-fit on F^2	1.106
Final R indices [I>2sigma(I)]	R1 = 0.0692, wR2 = 0.1801
R indices (all data)	R1 = 0.1088, wR2 = 0.2153
Extinction coefficient	0.00000(14)
Largest diff. peak and hole	1.388 and -0.768 e.Å^{-3}

. Table 97 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for $C_8H_{14}Br_2N_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	998(1)	3259(1)	1346(1)	43(1)
Br(2)	2420(1)	5503(1)	2631(1)	61(1)
N(1)	986(7)	4328(8)	4165(7)	32(4)
N(2)	1122(8)	5465(8)	4186(7)	37(4)
C(1)	1336(10)	5028(11)	2725(9)	38(5)
C(2)	1128(9)	4928(9)	3666(9)	30(4)
C(3)	882(9)	4490(11)	5021(9)	35(5)
C(4)	967(9)	5188(11)	5049(9)	35(4)

C(5)	941(11)	3626(10)	3896(9)	39(5)
C(6)	692(11)	3929(10)	5757(9)	39(5)
C(7)	934(12)	5644(11)	5783(10)	48(5)
C(8)	1268(12)	6216(10)	3889(10)	45(5)

Table 98 : Bond lengths [Å] and angles [°] for $C_8H_{14}Br_2N_2$.

Br(2)-C(1)	1.933(17)	C(2)-N(2)-C(4)	108.2(14)
N(1)-C(2)	1.348(19)	C(2)-N(2)-C(8)	125.5(12)
N(1)-C(3)	1.393(19)	C(4)-N(2)-C(8)	126.3(14)
N(1)-C(5)	1.46(2)	C(2)-C(1)-Br(2)	108.6(11)
N(2)-C(2)	1.363(19)	N(1)-C(2)-N(2)	108.7(12)
N(2)-C(4)	1.408(18)	N(1)-C(2)-C(1)	126.2(14)
N(2)-C(8)	1.49(2)	N(2)-C(2)-C(1)	124.8(15)
C(1)-C(2)	1.49(2)	C(4)-C(3)-N(1)	109.0(13)
C(3)-C(4)	1.35(2)	C(4)-C(3)-C(6)	129.0(14)
C(3)-C(6)	1.51(2)	N(1)-C(3)-C(6)	122.0(17)
C(4)-C(7)	1.48(2)	C(3)-C(4)-N(2)	106.3(14)
C(2)-N(1)-C(3)	107.7(13)	C(3)-C(4)-C(7)	131.8(14)
C(2)-N(1)-C(5)	128.3(12)	N(2)-C(4)-C(7)	121.9(17)
C(3)-N(1)-C(5)	124.0(13)		

Symmetry transformations used to generate equivalent atom.

Table 99 : Anisotropic displacement parameters ($Å^2 x 10^3$) for $C_8H_{14}Br_2N_2$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	47(2)	62(2)	21(1)	1(1)	0(1)	28(1)
Br(2)	55(2)	84(2)	45(1)	13(1)	12(1)	34(1)
N(1)	45(10)	39(10)	12(7)	6(7)	1(6)	22(8)
N(2)	45(10)	38(10)	23(8)	1(7)	3(6)	17(8)
C(1)	44(12)	63(14)	16(8)	12(9)	6(8)	34(11)
C(2)	41(12)	27(11)	30(10)	-6(9)	-9(7)	23(9)
C(3)	17(10)	45(14)	31(11)	5(9)	-1(7)	6(9)
C(4)	33(11)	53(14)	20(9)	5(8)	-1(7)	21(10)
C(5)	64(15)	40(13)	12(8)	-9(7)	-4(8)	26(13)
C(6)	64(15)	45(13)	23(9)	-1(8)	-10(9)	39(13)
C(7)	65(16)	62(14)	28(10)	4(10)	9(9)	41(14)
C(8)	69(16)	28(12)	28(10)	2(8)	-2(9)	17(11)

Table 100 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2 x 10^3$) for $C_8H_{14}Br_2N_2$.

	x	y	z	U(eq)
H(1A)	1164	5341	2448	0(30)
H(1B)	1099	4543	2433	160(130)
H(5A)	437	3217	3983	80(70)

H(5B)	1285	3543	4240	70(60)
H(5C)	1072	3656	3289	60(50)
H(6A)	636	4141	6292	0(30)
H(6B)	1089	3814	5819	20(40)
H(6C)	229	3476	5625	100(90)
H(7A)	736	5332	6293	60(50)
H(7B)	613	5842	5625	30(40)
H(7C)	1431	6052	5905	300(200)
H(8A)	1801	6549	3832	220(180)
H(8B)	1068	6417	4310	80(70)
H(8C)	1029	6167	3334	20(40)

7.3.21 Crysatal data for $C_9H_{14}Br_4N_2$ (**48**)

Table101 : Crystal data and structure refinement for $C_9H_{14}Br_4N_2$.

Empirical formula	$C_{17}H_{28}Br_5C_{l0.50}N_4O_{0.50}$	
Formula weight	713.71	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 10.333(3) Å	alpha= 90°.
	b = 9.873(4) Å	beta= 100.47(3)°.
	c = 27.656(11) Å	gamma□ = 90°.

Volume	2774.4(18) Å^3
Z	4
Density (calculated)	1.709 Mg/m^3
Absorption coefficient	7.302 mm^{-1}
F(000) 1382	
Crystal size	0.35 x 0.35 x 0.45 mm^3
Theta range for data collection	2.01 to 27.53°.
Index ranges	-13<=h<=9, -12<=k<=12, -35<=l<=35
Reflections collected	23042
Independent reflections	6381 [R(int) = 0.0798]
Completeness to theta = 27.53°	99.8 %
Max. and min. transmission	0.5453 and 0.1530
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6381 / 0 / 262
Goodness-of-fit on F^2	1.702
Final R indices [I>2sigma(I)]	R1 = 0.0757, wR2 = 0.1889
R indices (all data)	R1 = 0.1003, wR2 = 0.2195
Extinction coefficient	0.0030(7)
Largest diff. peak and hole	9.090 and -0.918 e.Å^{-3}

Table 102 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for $C_9H_{14}Br_4N_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

atom	x	y	z	U(eq)
Br(1)	35(1)	11021(1)	953(1)	29(1)
Br(2)	935(1)	9737(1)	2012(1)	28(1)
Br(3)	2683(1)	12007(1)	1643(1)	30(1)
Br(4)	9615(1)	8866(1)	3019(1)	31(1)
Br(5)	7559(1)	1675(1)	91(1)	30(1)
Cl(1)	1017(4)	3903(4)	8476(1)	28(1)
O(1)	318(14)	4956(16)	8332(5)	35(3)
N(1)	4060(7)	8189(7)	1801(3)	25(1)
N(2)	4760(8)	9698(7)	1349(3)	26(1)
N(3)	2695(7)	4501(7)	487(2)	20(1)
N(4)	1861(7)	2997(7)	-48(3)	26(1)
C(1)	1562(8)	10450(8)	1436(3)	23(1)
C(2)	2271(8)	9348(9)	1188(3)	23(2)
C(3)	3672(8)	9073(8)	1437(3)	23(1)
C(4)	5411(9)	8299(9)	1959(3)	29(2)
C(5)	5846(9)	9257(9)	1678(4)	29(2)
C(6)	3250(9)	7211(9)	2007(3)	30(2)
C(7)	6121(11)	7468(10)	2369(4)	41(2)
C(8)	7201(10)	9821(13)	1683(5)	46(3)
C(9)	4806(11)	10728(11)	966(4)	39(2)
C(10)	207(8)	4553(9)	229(3)	26(2)
C(11)	1551(8)	3986(9)	242(3)	24(2)
C(12)	3735(7)	3815(8)	349(3)	21(1)

C(13)	3216(8)	2881(8)	14(3)	23(1)
C(14)	2839(9)	5577(10)	854(3)	30(2)
C(15)	5133(8)	4153(9)	567(3)	26(2)
C(16)	3863(9)	1903(9)	-277(4)	29(2)
C(17)	924(9)	2048(10)	-334(4)	33(2)

Table 103 : Bond lengths [Å] and angles [°] for $C_9H_{14}Br_4N_2$.

Br(1)-C(1)	1.956(8)	N(4)-C(11)	1.338(11)
Br(2)-C(1)	1.955(8)	N(4)-C(13)	1.384(10)
Br(3)-C(1)	1.947(8)	N(4)-C(17)	1.471(11)
Cl(1)-O(1)	1.286(15)	C(1)-C(2)	1.541(11)
N(1)-C(3)	1.340(10)	C(2)-C(3)	1.509(11)
N(1)-C(4)	1.389(11)	C(4)-C(5)	1.352(14)
N(1)-C(6)	1.458(10)	C(4)-C(7)	1.481(12)
N(2)-C(3)	1.343(11)	C(5)-C(8)	1.505(13)
N(2)-C(5)	1.381(11)	C(10)-C(11)	1.491(11)
N(2)-C(9)	1.475(12)	C(10)-C(10)#1	1.539(18)
N(3)-C(11)	1.351(11)	C(12)-C(13)	1.347(12)
N(3)-C(12)	1.382(10)	C(12)-C(15)	1.498(10)
N(3)-C(14)	1.458(10)	C(13)-C(16)	1.491(11)
C(3)-N(1)-C(4)	109.4(7)	C(3)-N(2)-C(9)	125.7(8)
C(3)-N(1)-C(6)	127.6(8)	C(5)-N(2)-C(9)	124.2(8)
C(4)-N(1)-C(6)	123.0(8)	C(11)-N(3)-C(12)	109.3(7)
C(3)-N(2)-C(5)	110.1(7)	C(11)-N(3)-C(14)	126.4(7)

C(12)-N(3)-C(14)	124.3(7)
C(11)-N(4)-C(13)	109.5(7)
C(11)-N(4)-C(17)	125.5(7)
C(13)-N(4)-C(17)	124.5(7)
C(2)-C(1)-Br(3)	112.5(6)
C(2)-C(1)-Br(2)	111.3(5)
Br(3)-C(1)-Br(2)	108.2(4)
C(2)-C(1)-Br(1)	106.8(5)
Br(3)-C(1)-Br(1)	109.6(4)
Br(2)-C(1)-Br(1)	108.4(4)
C(3)-C(2)-C(1)	114.8(7)
N(2)-C(3)-N(1)	106.8(7)
N(2)-C(3)-C(2)	126.9(7)
N(1)-C(3)-C(2)	126.2(8)
C(5)-C(4)-N(1)	107.1(7)
C(5)-C(4)-C(7)	131.1(9)
N(1)-C(4)-C(7)	121.8(9)
C(4)-C(5)-N(2)	106.5(7)
C(4)-C(5)-C(8)	131.3(9)
N(2)-C(5)-C(8)	122.2(9)
C(11)-C(10)-C(10)#1	110.7(9)
N(4)-C(11)-N(3)	107.0(7)
N(4)-C(11)-C(10)	125.7(7)
N(3)-C(11)-C(10)	126.6(7)
C(13)-C(12)-N(3)	107.1(7)
C(13)-C(12)-C(15)	131.5(7)
N(3)-C(12)-C(15)	121.4(7)
C(12)-C(13)-N(4)	107.2(7)
C(12)-C(13)-C(16)	130.8(7)
N(4)-C(13)-C(16)	122.0(7)

Table 104: Anisotropic displacement parameters (Å^{2}x 10^3) for $C_9H_{14}Br_4N_2$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	20(1)	31(1)	33(1)	-1(1)	0(1)	4(1)
Br(2)	25(1)	32(1)	28(1)	-3(1)	9(1)	-1(1)
Br(3)	26(1)	29(1)	35(1)	-7(1)	7(1)	-5(1)
Br(4)	27(1)	34(1)	32(1)	1(1)	4(1)	2(1)

Br(5)	17(1)	38(1)	36(1)	2(1)	4(1)	3(1)
Cl(1)	22(2)	37(2)	29(2)	14(2)	17(1)	16(2)
O(1)	21(6)	48(8)	37(6)	-5(6)	11(5)	12(6)
N(1)	20(3)	27(3)	25(3)	2(3)	3(3)	-3(3)
N(2)	22(3)	27(3)	32(3)	6(3)	8(3)	-1(3)
N(3)	14(3)	26(3)	21(3)	-3(2)	6(2)	2(2)
N(4)	13(3)	26(3)	37(4)	-7(3)	4(3)	-1(2)
C(1)	21(4)	23(3)	22(3)	-1(3)	-1(3)	1(3)
C(2)	19(4)	30(4)	21(3)	0(3)	5(3)	7(3)
C(3)	20(4)	23(3)	24(3)	0(3)	0(3)	2(3)
C(4)	20(4)	29(4)	34(4)	-3(3)	-5(3)	3(3)
C(5)	16(4)	31(4)	39(4)	-4(3)	1(3)	-5(3)
C(6)	25(4)	28(4)	38(4)	3(3)	9(3)	-9(3)
C(7)	30(5)	30(4)	54(6)	3(4)	-15(4)	1(4)
C(8)	19(5)	50(6)	69(7)	8(5)	9(4)	-7(4)
C(9)	37(5)	42(5)	41(5)	10(4)	16(4)	-2(4)
C(10)	17(4)	29(4)	35(4)	-5(3)	9(3)	-2(3)
C(11)	16(4)	29(4)	27(3)	0(3)	7(3)	1(3)
C(12)	10(3)	28(3)	25(3)	3(3)	5(3)	4(3)
C(13)	12(3)	29(4)	29(3)	1(3)	7(3)	-2(3)
C(14)	24(4)	33(4)	33(4)	-10(3)	4(3)	3(3)
C(15)	11(3)	33(4)	31(4)	-1(3)	0(3)	-1(3)
C(16)	17(4)	34(4)	39(4)	-6(3)	7(3)	2(3)
C(17)	20(4)	38(4)	42(5)	-9(4)	4(3)	-4(3)

Table 105 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for $C_9H_{14}Br_4N_2$.

	x	y	z	U(eq)
H(2A)	1774	8512	1180	28
H(2B)	2272	9617	851	28
H(6A)	2364	7249	1827	45
H(6B)	3256	7425	2346	45
H(6C)	3598	6317	1983	45
H(7A)	5767	7641	2661	61
H(7B)	7039	7698	2427	61
H(7C)	6017	6526	2285	61
H(8A)	7499	9557	1387	69
H(8B)	7796	9474	1963	69
H(8C)	7174	10791	1701	69
H(9A)	3946	10829	768	59
H(9B)	5415	10447	761	59
H(9C)	5086	11578	1118	59
H(10A)	199	5087	523	32
H(10B)	-417	3818	225	32
H(14A)	3070	6406	709	45
H(14B)	3519	5339	1125	45
H(14C)	2023	5696	968	45
H(15A)	5715	3594	419	39
H(15B)	5282	3993	915	39

H(15C)	5298	5089	506	39
H(16A)	4796	1905	-158	44
H(16B)	3689	2163	-617	44
H(16C)	3521	1011	-244	44
H(17A)	821	2265	-677	50
H(17B)	88	2120	-231	50
H(17C)	1251	1140	-280	50

7.3.22 Crystal Data for $C_{15}H_{20}O_2N_2$ (**49**)

Table 106 : Crystal data and structure refinement for $C_{15}H_{20}O_2N_2$.

Empirical formula	$C_{6.32}H_{8.42}N_{0.84}O_{0.84}$
Formula weight	109.61
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Tetragonal, P4(1)2(1)2
Unit cell dimensions	a = 9.9434(11) A alpha = 90 deg.
	b = 9.9434(11) A beta = 90 deg.
	c = 28.835(4) A gamma = 90 deg.
Volume	2851.0(6) A^3
Z, Calculated density	19, 1.213 Mg/m^3
Absorption coefficient	0.081 mm^-1
F(000)	1120
Crystal size	0.5 x 1.2 x 0.5 mm
Theta range for data collection	2.17 to 27.50 deg.

Limiting indices	-1<=h<=12, -12<=k<=1, -37<=l<=1
Reflections collected / unique	4690 / 3274 [R(int) = 0.0259]
Completeness to theta = 27.50	100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3274 / 0 / 178
Goodness-of-fit on F^2	1.028
Final R indices [I>2sigma(I)]	R1 = 0.0907, wR2 = 0.2625
R indices (all data)	R1 = 0.1254, wR2 = 0.2999
Absolute structure parameter	4(5)
Extinction coefficient	0.014(5)
Largest diff. peak and hole	0.522 and -0.262 e.A^-3

Table 107 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for $C_{15}H_{20}O_2N_2$.U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(2)	9319(4)	7986(4)	606(1)	48(1)
N(1)	10380(3)	8148(3)	316(1)	57(1)
N(2)	9285(4)	6349(3)	101(1)	64(1)
C(9)	1152(4)	4290(5)	864(1)	58(1)
C(1)	10362(4)	7150(3)	3(1)	52(1)
C(3)	8641(4)	6888(4)	473(1)	54(1)

C(10)	1293(6)	5417(5)	1120(2)	88(2)
C(5)	11391(4)	9224(4)	360(2)	71(1)
C(15)	-57(5)	3384(5)	920(2)	73(1)
C(7)	7426(5)	6292(6)	692(2)	90(2)
O(1)	-54(6)	2335(6)	678(2)	145(2)
O(2)	-911(4)	3710(5)	1200(2)	124(2)
C(6)	9038(6)	8879(6)	997(2)	95(2)
C(4)	11361(6)	6874(6)	-374(2)	93(2)
C(8)	8965(6)	5100(5)	-143(2)	92(2)
C(14)	2193(7)	3985(13)	583(3)	147(3)
C(12)	3333(10)	5879(15)	826(4)	190(7)
C(11)	2359(10)	6246(9)	1098(4)	146(4)
C(13)	3327(11)	4770(20)	530(4)	249(11)

Table 108 : Bond lengths [A] and angles [deg] for $C_{15}H_{20}O_2N_2$

C(2)-C(3)	1.339(5)	N(2)-C(8)	1.463(6)
C(2)-N(1)	1.354(5)	C(9)-C(14)	1.349(7)
C(2)-C(6)	1.463(6)	C(9)-C(10)	1.349(7)
N(1)-C(1)	1.341(5)	C(9)-C(15)	1.510(6)
N(1)-C(5)	1.474(6)	C(1)-C(4)	1.498(6)
N(2)-C(3)	1.359(5)	C(3)-C(7)	1.486(6)
N(2)-C(1)	1.364(5)	C(10)-C(11)	1.344(9)

C(15)-O(2)	1.216(6)	C(14)-C(9)-C(15)	122.7(6)
C(15)-O(1)	1.254(7)	C(10)-C(9)-C(15)	121.3(4)
C(14)-C(13)	1.382(17)	N(1)-C(1)-N(2)	107.6(3)
C(12)-C(11)	1.298(17)	N(1)-C(1)-C(4)	128.0(4)
C(12)-C(13)	1.39(2)	N(2)-C(1)-C(4)	124.3(4)
		C(2)-C(3)-N(2)	108.0(3)
C(3)-C(2)-N(1)	108.2(3)	C(2)-C(3)-C(7)	127.8(4)
C(3)-C(2)-C(6)	128.2(4)	N(2)-C(3)-C(7)	124.1(4)
N(1)-C(2)-C(6)	123.5(4)	C(11)-C(10)-C(9)	124.4(8)
C(1)-N(1)-C(2)	108.5(3)	O(2)-C(15)-O(1)	126.3(5)
C(1)-N(1)-C(5)	127.1(3)	O(2)-C(15)-C(9)	117.9(5)
C(2)-N(1)-C(5)	124.4(3)	O(1)-C(15)-C(9)	115.8(5)
C(3)-N(2)-C(1)	107.6(3)	C(9)-C(14)-C(13)	124.5(11)
C(3)-N(2)-C(8)	127.7(4)	C(11)-C(12)-C(13)	126.1(9)
C(1)-N(2)-C(8)	124.6(4)	C(12)-C(11)-C(10)	116.4(10)
C(14)-C(9)-C(10)	115.8(6)	C(14)-C(13)-C(12)	112.5(10)

Table 109 : Anisotropic displacement parameters (A^2 x 10^3) for $C_{15}H_{20}O_2N_2$. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(2)	44(2)	52(2)	50(2)	3(2)	7(1)	5(2)
N(1)	55(2)	57(2)	60(2)	0(1)	4(1)	5(1)

N(2)	71(2)	56(2)	63(2)	8(2)	-3(2)	-7(2)
C(9)	48(2)	76(2)	49(2)	5(2)	-2(2)	-4(2)
C(1)	61(2)	47(2)	50(2)	3(2)	9(2)	-6(2)
C(3)	47(2)	55(2)	61(2)	15(2)	4(2)	5(2)
C(10)	106(4)	65(3)	94(3)	15(2)	-39(3)	-9(3)
C(5)	67(2)	63(2)	82(3)	-6(2)	20(2)	-13(2)
C(15)	72(3)	85(3)	63(2)	-6(2)	-13(2)	-10(2)
C(7)	66(3)	88(3)	116(4)	19(3)	23(3)	-15(2)
O(1)	153(5)	132(4)	150(4)	-56(4)	-48(4)	-28(3)
O(2)	87(3)	133(4)	153(4)	11(3)	59(3)	-15(3)
C(6)	87(4)	104(4)	95(3)	-22(3)	38(3)	-4(3)
C(4)	111(4)	83(3)	84(3)	-10(3)	43(3)	-8(3)
C(8)	113(4)	59(3)	103(3)	-5(3)	-9(3)	-18(3)
C(14)	93(4)	235(10)	112(5)	-24(6)	47(4)	5(6)
C(12)	79(5)	302(17)	189(10)	152(11)	-38(6)	-80(8)
C(11)	124(6)	117(5)	199(9)	81(6)	-75(6)	-51(5)
C(13)	94(6)	500(30)	153(9)	46(14)	53(7)	-17(12)

Table 110 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for $C_{15}H_{20}O_2N_2$.

	x	y	z	U(eq)
H(10)	592	5640	1330	106
H(5A)	12077	8955	586	106
H(5B)	10953	10052	465	106

H(5C)	11816	9379	58	106
H(7A)	7697	5579	907	135
H(7B)	6843	5913	451	135
H(7C)	6936	6991	861	135
H(6A)	8109	8746	1101	143
H(6B)	9162	9816	901	143
H(6C)	9655	8673	1253	143
H(4A)	11783	7719	-471	139
H(4B)	10900	6465	-639	139
H(4C)	12052	6256	-258	139
H(8A)	8725	5305	-465	138
H(8B)	8205	4656	10	138
H(8C)	9749	4503	-138	138
H(14)	2143	3173	410	176
H(12)	4124	6415	830	228
H(11)	2398	7056	1271	176
H(13)	4026	4581	315	299

7.3.23 Crystal data for $C_{22}H_{22}N_2O_2$ (**53**)

Table 111 : Crystal data and structure refinement for $C_{22}H_{22}N_2O_2$

Empirical formula	$C_{9.60}H_{9.60}Cl_{2.40}N_{0.80}O_{0.80}$
Formula weight	234.06
Temperature	173(2) K
Wavelength	0.71073 Å

Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 15.589(2) Å	alpha= 90°.
	b = 16.086(2) Å	beta= 122.388(10)°.
	c = 12.9363(16) Å	gama = 90°.
Volume	2739.2(6) Å^3	
Z	10	
Density (calculated)	1.419 Mg/m^3	
Absorption coefficient	0.652 mm^{-1}	
F(000)	1200	
Crystal size	0.3 x 0.8 x 0.3 mm^3	
Theta range for data collection	2.09 to 27.50°.	
Index ranges	-20<=h<=12, -20<=k<=20, -16<=l<=14	
Reflections collected	10987	
Independent reflections	3134 [R(int) = 0.0361]	
Completeness to theta = 27.50°	99.3 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3134 / 0 / 158	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0445, wR2 = 0.1050	
R indices (all data)	R1 = 0.0579, wR2 = 0.1127	
Extinction coefficient	0.0026(3)	

Largest diff. peak and hole	0.649 and -0.634 e.Å^{-3}

Table 112 : Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for $C_{22}H_{22}N_2O_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	963(1)	1720(1)	9637(1)	38(1)
N(1)	785(1)	3074(1)	7774(2)	30(1)
C(1)	0	2574(2)	7500	27(1)
C(2)	493(2)	3902(1)	7674(2)	34(1)
C(3)	1789(2)	2785(2)	8109(2)	45(1)
C(4)	1211(2)	4601(1)	7950(2)	51(1)
C(5)	0	1662(2)	7500	27(1)
C(6)	627(1)	1295(1)	8686(2)	27(1)
C(7)	977(1)	407(1)	8858(2)	28(1)
C(8)	1263(2)	27(1)	8124(2)	33(1)
C(9)	1623(2)	-783(1)	8357(2)	46(1)
C(10)	1694(2)	-1221(1)	9321(2)	53(1)
C(11)	1420(2)	-849(1)	10063(2)	48(1)
C(12)	1075(2)	-33(1)	9846(2)	36(1)
C(13)	610(2)	3240(1)	765(2)	43(1)
Cl(1)	-628(1)	3637(1)	127(1)	70(1)
Cl(2)	1089(1)	2917(1)	2276(1)	73(1)
Cl(3)	1420(1)	3993(1)	758(1)	57(1)

Table 113 : Bond lengths [Å] and angles [°] for $C_{22}H_{22}N_2O_2$.

O(1)-C(6)	1.251(2)	C(2)-N(1)-C(3)	125.40(17)
N(1)-C(1)	1.344(2)	N(1)-C(1)-N(1)#1	106.5(2)
N(1)-C(2)	1.391(2)	N(1)-C(1)-C(5)	126.76(11)
N(1)-C(3)	1.458(3)	N(1)#1-C(1)-C(5)	126.76(11)
C(1)-N(1)#1	1.344(2)	C(2)#1-C(2)-N(1)	106.81(11)
C(1)-C(5)	1.467(3)	C(2)#1-C(2)-C(4)	130.92(14)
C(2)-C(2)#1	1.350(4)	N(1)-C(2)-C(4)	122.3(2)
C(2)-C(4)	1.490(3)	C(6)#1-C(5)-C(6)	131.2(2)
C(5)-C(6)#1	1.432(2)	C(6)#1-C(5)-C(1)	114.38(11)
C(5)-C(6)	1.432(2)	C(6)-C(5)-C(1)	114.38(11)
C(6)-C(7)	1.502(2)	O(1)-C(6)-C(5)	120.92(17)
C(7)-C(8)	1.389(3)	O(1)-C(6)-C(7)	116.47(16)
C(7)-C(12)	1.395(3)	C(5)-C(6)-C(7)	122.48(17)
C(8)-C(9)	1.387(3)	C(8)-C(7)-C(12)	119.07(18)
C(9)-C(10)	1.384(4)	C(8)-C(7)-C(6)	122.67(17)
C(10)-C(11)	1.378(4)	C(12)-C(7)-C(6)	118.13(17)
C(11)-C(12)	1.390(3)	C(9)-C(8)-C(7)	120.4(2)
C(13)-Cl(3)	1.754(3)	C(10)-C(9)-C(8)	120.1(2)
C(13)-Cl(2)	1.756(2)	C(11)-C(10)-C(9)	120.1(2)
C(13)-Cl(1)	1.763(2)	C(10)-C(11)-C(12)	120.0(2)
		C(11)-C(12)-C(7)	120.3(2)
C(1)-N(1)-C(2)	109.96(17)	Cl(3)-C(13)-Cl(2)	109.38(14)
C(1)-N(1)-C(3)	124.64(17)	Cl(3)-C(13)-Cl(1)	110.96(13)

Cl(2)-C(13)-Cl(1)   109.56(13)

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,-z+3/2

Table 114 : Anisotropic displacement parameters ($Å^2$x 10^3) for $C_{22}H_{22}N_2O_2$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	46(1)	28(1)	32(1)	-5(1)	16(1)	3(1)
N(1)	28(1)	24(1)	34(1)	1(1)	15(1)	-3(1)
C(1)	28(1)	23(1)	28(1)	0	13(1)	0
C(2)	45(1)	22(1)	31(1)	0(1)	18(1)	-4(1)
C(3)	28(1)	44(1)	59(1)	7(1)	20(1)	-2(1)
C(4)	69(2)	32(1)	55(1)	-3(1)	34(1)	-17(1)
C(5)	25(1)	20(1)	33(1)	0	14(1)	0
C(6)	26(1)	23(1)	32(1)	-2(1)	15(1)	-1(1)
C(7)	24(1)	23(1)	30(1)	0(1)	11(1)	0(1)
C(8)	33(1)	30(1)	37(1)	0(1)	19(1)	3(1)
C(9)	51(1)	35(1)	53(1)	-2(1)	28(1)	12(1)
C(10)	63(2)	26(1)	59(2)	5(1)	25(1)	13(1)
C(11)	62(2)	31(1)	44(1)	11(1)	23(1)	3(1)
C(12)	41(1)	33(1)	32(1)	1(1)	18(1)	0(1)
C(13)	52(1)	37(1)	47(1)	-2(1)	30(1)	8(1)
Cl(1)	55(1)	92(1)	61(1)	6(1)	30(1)	25(1)

Cl(2)	84(1)	82(1)	68(1)	36(1)	51(1)	34(1)
Cl(3)	74(1)	47(1)	60(1)	-3(1)	44(1)	-4(1)

Table 115 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for $C_{22}H_{22}N_2O_2$.

	x	y	z	U(eq)
H(3A)	1790	2177	8070	68
H(3B)	2294	2967	8943	68
H(3C)	1959	3017	7539	68
H(4A)	872	5130	7884	77
H(4B)	1428	4596	7366	77
H(4C)	1806	4539	8783	77
H(8)	1212	324	7459	39
H(9)	1820	-1039	7853	55
H(10)	1932	-1779	9472	64
H(11)	1468	-1151	10723	58
H(12)	905	227	10373	44
H(13)	582	2751	271	52

7.3.24 Crysta data for $C_{16}H_{19}N_2O_3SCl$ (**55**)

Table 116 : Crystal data and structure refinement for $C_{16}H_{19}N_2O_3SCl$

Empirical formula	$C_{16}H_{19}N_2O_3SCl$
Formula weight	371.88

Temperature	293(2) K
Wavelength / Radiation	0.71073 A / MoK\a
Crystal system / Spacegroup	Orthorhombic/ P212121
Unit cell dimensions	a = 8.3009(8)A alpha = 90 deg.
	b = 13.9260(7)A beta = 90 deg.
	c = 15.978(3)A gamma = 90 deg.
Volume	1847.0(4) A^3
Z	4
Density (calculated)	1.337 Mg/m^3
Absorption coefficient	0.339 mm^-1
F(000)	784
Crystal description	colourless block
Crystal size	0.5 x 0.3 x 0.2 mm
No. of reflns. (lattice)	25
Theta range (lattice)	8.87 to 13.79 deg.
Theta range for data collection	3.13 to 25.99 deg.
Index ranges	-1<=h<=10, -1<=k<=17, -19<=l<=19
Reflections collected	4917
Independent reflections	3611 [R(int) = 0.0206]
Reflections observed	2974
Criterion for observation	>2sigma(I)
Absorption correction	Psi-Scan
Max. and min. transmission	0.9707 and 0.9310

Measurement method	omega scans
No. of standard reflections	3
Interval count/time	300/3600
Decay	10.0 %
Structure solution	SHELXS-97 (Sheldrick, 1990)
Structure refinement	SHELXL-97 (Sheldrick, 1997)
Molecular graphics	SCHAKAL, ZORTEP
Publication material	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full-matrix least-squares on F^2
Solution primary	direct
Hydrogen treatment	mixed
Data / restraints / parameters	3611 / 0 / 305
Final R indices [I>2sigma(I)]	R1 = 0.0377, wR2 = 0.0952
Goodness-of-fit on F^2	1.048
Final R indices (all) R1 / wR2	0.0578 / 0.1036
Absolute structure parameter	-0.01(8)
Largest diff. peak and hole	0.211 and -0.118 e.A^-3

Table 117 : Atomic coordinates and equivalent isotropic displacement parameters (A^2 x 10^3) for $C_{16}H_{19}N_2O_3SCl$. (U(eq) is defined as one third of the trace of the orthogonalized Uij tensor).

	x	y	z	U(eq)
S(1)	0.0593(1)	0.6961(1)	0.3550(1)	49(1)
C(1)	0.2619(3)	0.6968(2)	0.3193(2)	44(1)

C(2)	0.3882(4)	0.7044(2)	0.3752(2)	56(1)
C(3)	0.5446(4)	0.6994(2)	0.3450(3)	72(1)
C(4)	0.5771(4)	0.6893(2)	0.2621(3)	73(1)
C(5)	0.4511(5)	0.6827(2)	0.2069(3)	69(1)
C(6)	0.2935(4)	0.6851(2)	0.2341(2)	54(1)
O(7)	0.0213(3)	0.5956(2)	0.3673(1)	68(1)
O(8)	0.0581(3)	0.7496(2)	0.4322(1)	82(1)
O(9)	-0.0335(3)	0.7400(2)	0.2901(2)	89(1)
Cl(1)	0.2191(1)	0.5239(1)	0.0450(1)	76(1)
C(10)	0.4444(3)	0.4332(2)	0.1382(2)	44(1)
N(11)	0.4279(3)	0.4347(1)	0.2222(1)	46(1)
C(12)	0.5759(3)	0.4506(2)	0.2587(2)	45(1)
C(13)	0.6829(3)	0.4601(2)	0.1955(2)	43(1)
N(14)	0.6001(3)	0.4485(2)	0.1218(1)	43(1)
C(15)	0.6004(5)	0.4544(3)	0.3502(2)	66(1)
C(16)	0.2772(5)	0.4226(3)	0.2676(3)	70(1)
C(17)	0.3161(4)	0.4140(3)	0.0782(2)	61(1)
C(18)	0.8589(4)	0.4795(3)	0.1972(3)	63(1)
C(19)	0.6756(5)	0.4502(3)	0.0388(2)	62(1)
C(31)	0.3962(6)	0.7404(3)	-0.0692(2)	87(1)
C(32)	0.5072(5)	0.6688(4)	-0.0950(3)	83(1)
N(33)	0.3058(8)	0.7961(4)	-0.0498(3)	148(2)

Table 118 : Full bond lengths [A] and angles [deg] for $C_{16}H_{19}N_2O_3SCl$.

S(1)-O(9)	1.429(2)
S(1)-O(8)	1.442(2)
S(1)-O(7)	1.448(2)
S(1)-C(1)	1.776(3)
C(1)-C(2)	1.380(4)
C(1)-C(6)	1.396(4)
C(2)-C(3)	1.387(5)
C(3)-C(4)	1.359(6)
C(4)-C(5)	1.370(6)
C(5)-C(6)	1.379(5)
Cl(1)-C(17)	1.808(3)
C(10)-N(14)	1.336(4)
C(10)-N(11)	1.349(3)
C(10)-C(17)	1.458(4)
N(11)-C(12)	1.378(3)
N(11)-C(16)	1.456(4)
C(12)-C(13)	1.352(4)
C(12)-C(15)	1.477(4)
C(13)-N(14)	1.372(3)
C(13)-C(18)	1.486(4)
N(14)-C(19)	1.468(4)
C(31)-N(33)	1.123(6)
C(31)-C(32)	1.419(6)
O(9)-S(1)-O(8)	113.33(18)
O(9)-S(1)-O(7)	113.24(17)
O(8)-S(1)-O(7)	112.43(14)
O(9)-S(1)-C(1)	106.02(13)
O(8)-S(1)-C(1)	106.16(14)
O(7)-S(1)-C(1)	104.78(13)
C(2)-C(1)-C(6)	119.8(3)
C(2)-C(1)-S(1)	120.8(2)
C(6)-C(1)-S(1)	119.3(2)
C(1)-C(2)-C(3)	118.9(3)
C(4)-C(3)-C(2)	122.0(3)
C(3)-C(4)-C(5)	118.8(3)
C(4)-C(5)-C(6)	121.3(4)
C(5)-C(6)-C(1)	119.2(3)
N(14)-C(10)-N(11)	106.9(2)
N(14)-C(10)-C(17)	127.4(3)
N(11)-C(10)-C(17)	125.7(3)
C(10)-N(11)-C(12)	109.5(2)
C(10)-N(11)-C(16)	125.5(3)
C(12)-N(11)-C(16)	125.0(3)
C(13)-C(12)-N(11)	106.6(2)

C(13)-C(12)-C(15)	130.2(3)
N(11)-C(12)-C(15)	123.2(3)
C(12)-C(13)-N(14)	107.5(2)
C(12)-C(13)-C(18)	130.5(3)
N(14)-C(13)-C(18)	122.0(3)
C(10)-N(14)-C(13)	109.6(2)
C(10)-N(14)-C(19)	126.4(2)
C(13)-N(14)-C(19)	124.0(2)
C(10)-C(17)-Cl(1)	111.3(2)
N(33)-C(31)-C(32)	178.5(6)

Table 119 : Anisotropic displacement parameters (A^2 x 10^3) for $C_{16}H_{19}N_2O_3SCl$. The anisotropic displacement factor exponent takes the form:-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12].

	U11	U22	U33	U23	U13	U12
S(1)	45(1)	56(1)	47(1)	3(1)	3(1)	-1(1)
C(1)	43(1)	36(1)	54(1)	3(1)	0(1)	-2(1)
C(2)	51(2)	48(2)	70(2)	-1(1)	-10(1)	-4(1)
C(3)	46(2)	52(2)	119(3)	-3(2)	-14(2)	-4(2)
C(4)	49(2)	48(2)	123(3)	-1(2)	17(2)	-4(2)
C(5)	82(2)	48(2)	76(2)	0(2)	30(2)	-1(2)
C(6)	60(2)	49(2)	54(2)	-1(1)	5(1)	1(1)
O(7)	68(1)	67(1)	68(1)	3(1)	11(1)	-19(1)
O(8)	79(2)	90(2)	77(1)	-26(1)	22(1)	-6(1)
O(9)	49(1)	124(2)	93(2)	43(2)	-3(1)	15(1)
Cl(1)	68(1)	92(1)	69(1)	5(1)	-19(1)	14(1)
C(10)	44(1)	41(1)	48(1)	-3(1)	-5(1)	1(1)
N(11)	47(1)	39(1)	51(1)	-3(1)	4(1)	1(1)
C(12)	49(1)	38(1)	47(1)	-3(1)	-5(1)	6(1)
C(13)	40(1)	40(1)	50(1)	-4(1)	-6(1)	4(1)
N(14)	42(1)	45(1)	42(1)	-1(1)	-2(1)	2(1)
C(15)	87(3)	67(2)	45(1)	-1(2)	-1(2)	10(2)
C(16)	55(2)	62(2)	94(3)	-8(2)	26(2)	-3(2)
C(17)	49(2)	67(2)	68(2)	-10(2)	-15(2)	3(2)
C(18)	64(2)	80(2)	43(2)	-2(2)	1(2)	-2(2)

C(31)	99(3)	91(3)	72(2)	0(2)	13(2)	11(2)
C(32)	68(2)	99(3)	83(3)	-2(2)	-7(2)	2(2)
N(33)	167(5)	142(4)	135(4)	-12(3)	37(4)	38(4)

Table 120 : Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for $C_{16}H_{19}N_2O_3SCl$.

	x	y	z	U(eq)
H(2)	3590(4)	7180(2)	4350(2)	62(9)
H(3)	6290(7)	7050(3)	3880(3)	119(16)
H(4)	6750(5)	6900(3)	2410(2)	77(11)
H(5)	4790(5)	6690(3)	1630(3)	77(13)
H(6)	2070(4)	6820(2)	2015(17)	48(8)
H(151)	5300(5)	4990(3)	3780(2)	88(12)
H(152)	5980(5)	3950(3)	3720(2)	80(11)
H(153)	7010(6)	4850(3)	3610(3)	95(13)
H(161)	2310(7)	3740(4)	2560(3)	122(19)
H(162)	3135(14)	4098(8)	3235(6)	125(9)
H(163)	2380(7)	4730(4)	2720(3)	130(2)
H(171)	2330(4)	3760(2)	1044(19)	57(8)
H(172)	3660(4)	3930(2)	290(2)	66(9)
H(181)	9040(5)	4300(3)	1710(2)	79(11)
H(182)	8850(4)	4840(2)	2540(2)	64(9)
H(183)	8820(4)	5430(3)	1630(2)	69(9)

H(19)	6070(4)	4490(2)	30(2)	62(10)
H(191)	7470(6)	5110(3)	310(3)	97(12)
H(192)	7560(5)	4010(3)	400(2)	94(12)
H(321)	5230(6)	6650(4)	-1690(3)	127(16)
H(322)	5910(6)	6920(3)	-710(3)	105(15)
H(323)	4740(6)	6010(4)	-830(3)	121(17)

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