Derivate des Imidazols und der Barbitursäure - Heterozyklische Carben-Fragmente mit π-Donor und Akzeptor Funktion

****

Imidazol and Barbituric Acid Derivatives - Heterocyclic Carbene Fragments with π-Donor and Acceptor Properties

DISSERTATION

der Fakultät für Chemie und Pharmazie

der Eberhard-Karls-Universität Tübingen

zur Erlangung des Grades eines Doktors

der Naturwissenschaften

2006

vorgelegt von

Kamal Sweidan

Tag der mündlichen Prüfung:	06.04.2006
Dekan:	Prof. Dr. S. Laufer
1. Berichterstatter:	Prof. Dr. N. Kuhn
2. Berichterstatter:	Prof. Dr. H. A. Mayer

To my

parents, wife, sisters, brothers,

children Nagam and Hla

Die vorliegende Arbeit wurde am

Institut für Anorganische Chemie der

Eberhard-Karls-Universität Tübingen

unter der Leitung von Prof. Dr. Norbert Kuhn

angefertigt.

Meinem Doktorvater,

Herrn Prof. Dr. Norbert Kuhn,

danke ich herzlich für die Themenstellung,

für die Bereitstellung ausgezeichneter Arbeitsbedingungen,

für wertvolle Anregungen und Diskussionen

sowie sein stetes Interesse an dieser Arbeit.

Ich möchte mich herzlich bedanken bei

Herrn M. Steimann, Frau Dr. C. Maichle-Mößmer, Herrn Dr. S. Schwartz, Herrn Dr. M. Ströbele, Herrn Dr. M. Richter, Herrn H. Kolb und Herrn Dr. K. Eichele für die Durchführung der Kristallstrukturanalysen,

Frau Heike Dorn, Frau Angleika Ehmann und den Messberechtigten am 250 und 400 MHz DRX-Gerät für Hochauflösungs-NMR-Spektren,

Herrn Prof. Dr. H. A. Mayer für die Hilfe bei NMR-Problemen,

Herrn Bartholomä und Herrn Dr. Müller für die geduldige Durchführung zahlreicher Massenspektren,

Herrn W. Bock für die Durchführung vieler schwieriger Elementaranalysen,

Frau Barbara Saller für die Durchführung zahlreicher IR-Messungen und viele Hilfestellungen,

Frau Roswitha Conrad und Herrn Dr. H. Ebert für die Hilfe bei bürokratischen Angleegenheiten,

Herrn Dr. Mahmoud Sunjuk, Herrn Dr. Adnan Al-Labadi, Herrn Dipl. Chem. Adeeb Al-Dahschan, Herrn Dr. Ismail Warad, Herrn Dr. Ahmed Al-Sheikh, Herrn Dr. Ahmed Abu-Rayyan und Herrn Dr. Murat Ciftci danke ich für viele entspannende Stunden in der Freizeit.

Frau Dr. Isabel Walker, Herrn Dr. Ahmed Al-Sheikh, Herrn Dr. Ahmed Abu-Rayyan, für die Zusammenarbeit im Arbeitskreis, die oft hilfreichen Diskussionen, Anregungen und Auseinandersetzungen.

Nicht zuletzt möchte ich mich ganz herzlich bei meiner Familie für ihre Unterstützung und bei meiner Frau für ihre Geduld und ihren Beistand bedanken.

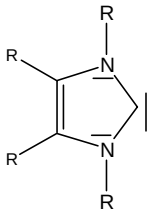
1. Introduction

1.1 Carbenes

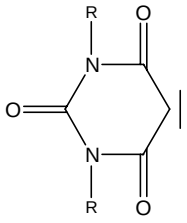
1.1.1 Definition

Carbenes are two–coordinate carbon compounds that have two nonbonding electrons and no formal charge on the carbon. These types of two-coordinate carbene compounds essentially represent carbon in an oxidation state of +II. This oxidation state is well represented in carbon chemistry by stable monocoordinated carbon centers such as those in carbon monoxide and isonitriles. The stability of these carbenes is derived from a combination of steric and electronic factors [1].

In general, carbenes are electronically stabilized by +M donor substituents and destabilized by –M acceptor substituents, e.g. **21** and **24** (see 1.3).



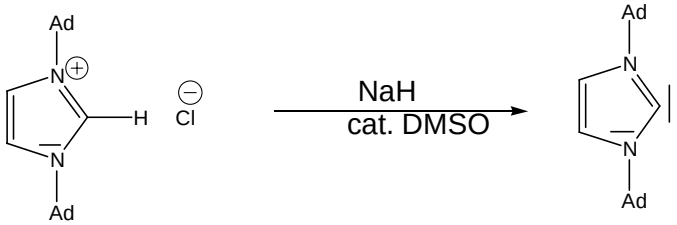
21



24

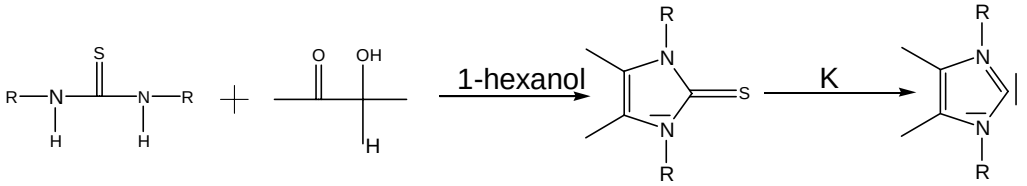
1.1.2 Preparation of diaminocarbenes

Arduengo et al. prepared the first crystalline carbene. It was 1,3-diadamantyl-imidazol-2-ylidene **2**, forms colorless crystal with sufficient kinetic and thermodynamic stability to be easily isolated and characterized. This carbene was obtained by deprotonation of the 1,3-diadamantylimidazolium chloride **1** with sodium hydride in the presence of catalytic amount of dimethylsulfoxide [2].



1 **2**

In 1993, Kuhn and co-workers developed a new effective route for alkyl-substituted N-heterocyclic carbenes **4** synthesis. This method depends on the reduction of imidazole-2-thiones **3** with potassium in boiling THF. The synthesis of the imidazol-2-thiones is best achieved by a condensation reaction starting from the thioureas and 3-hydroxy-2-butanone in boiling 1-hexanol [3].



3 a-c **4 a-c**

a: R= Me

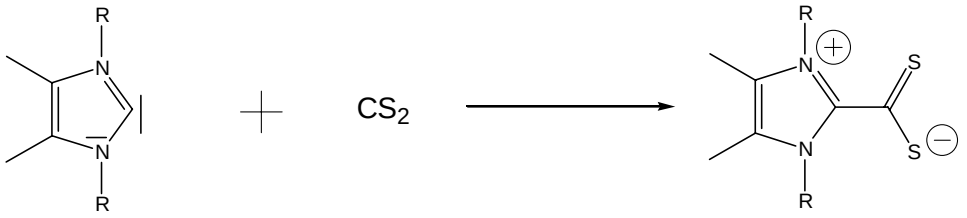
b: R= Et

c: R= i-Pr

1.1.3 Chemistry of carbenes

Owing to their highly nucleophilic character heterocyclic carbenes act as ligands in complexes of metal and metalloid centres in a manner similar to tertiary phosphanes [4].

For example, the carbenes react readily with carbon disulfide to give carbon disulfide adducts **5** [5].



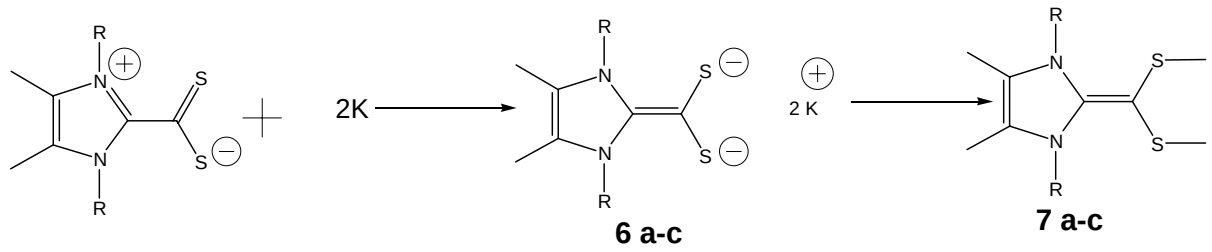
5 a-c

a: R= Me

b: R= Et

c: R= i-Pr

Reduction with potassium in tetrahydrofuran gives the salt **6** [6], which react with methyl iodide to give the 2-methyleneimidazolines **7** [7].



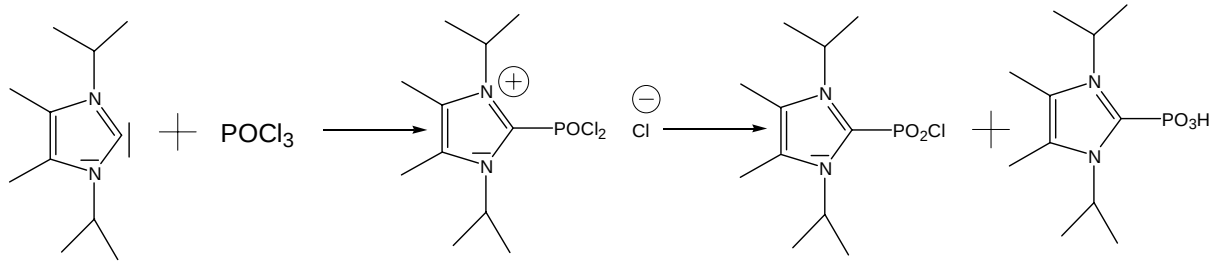
6 a-c **7 a-c**

a: R= Me

b: R= Et

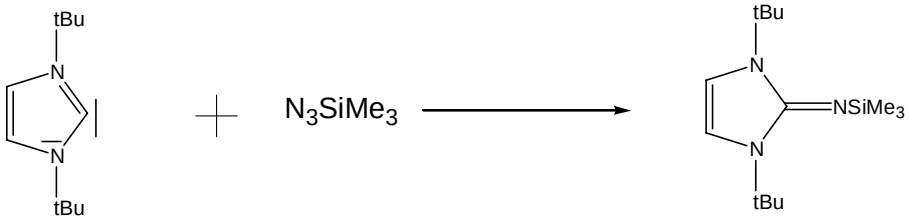
c: R= i-Pr

Carbene **4c** reacts with $POCl_3$ to form the salt **8**, which is transformed into the phosphoric chloride adduct **9** and its acid **10** by stepwise hydrolysis [8].



8 **9** **10**

Reaction between carbene **11** and N_3SiMe_3 produced **12** [9].



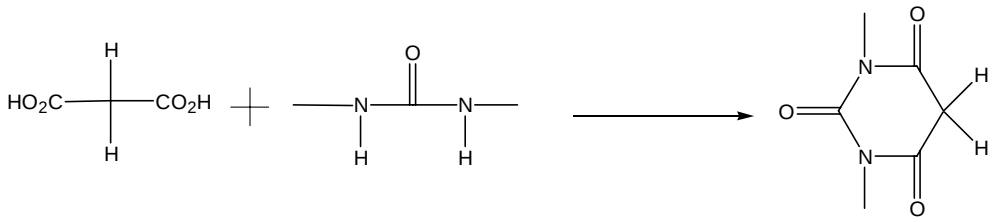
11 **12**

1.2 1.3-dimethylbarbituric acid

1.2.1 Definition

Barbituric acids are heterocyclic derivatives of pyrimidine triones. They are proven to have a wide variety of biological activity [10].

1,3-dimethylbarbituric acid **13** can be easily prepared from malonic acid and 1,3-dimethyl urea with acetic anhydride as condensing agent [11].



13

Ethyl malonate and 1,3-dimethyl urea with sodium alkoxide can also be used for synthesis but in a lower yield [12].

1.2.2 Physical properties

1,3-dimethylbarbituric acid is a white solid compound, m.p.121-123°C.

1,3-dimethylbarbituric acid is considered to be a strong acid with pKa 4.4 relative to acetic acid which has pKa 4.76 [13]. The high acidity can be attributed to the presence of hydrogen atoms attached to a carbon atom which is between two carbonyl groups which gives stability of the resultant anion.

Barbituric acid exists in solution as a single tautomer. The ^{13}C-NMR spectroscopy shows that they are present in the triketo form in a number of polar and non-polar solvents [14].

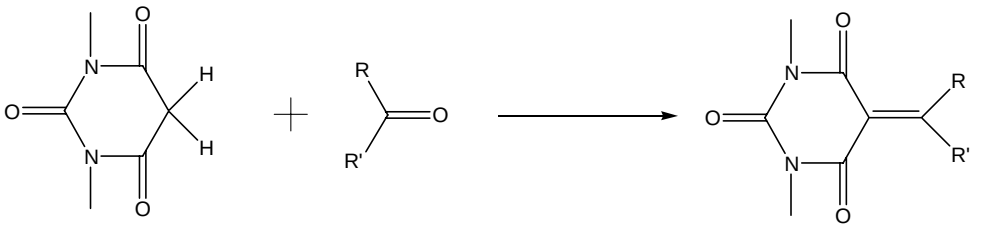
In the IR-spectrum, it shows three carbonyl stretching bands, at 1701 cm^{-1} for (4,6-CO asym. stre.), at 1669 cm^{-1} for (2-CO stre.) and at 1730 cm^{-1} for (4,6-CO sym. stre.) [15].

According to the X-ray crystal structure determination, the ring in barbituric acid is significantly distorted from planarity, in such a way that the methylene part of the ring has a boat-shaped configuration [16].

1.2.3 Chemistry of 1,3-dimethylbarbituric acid

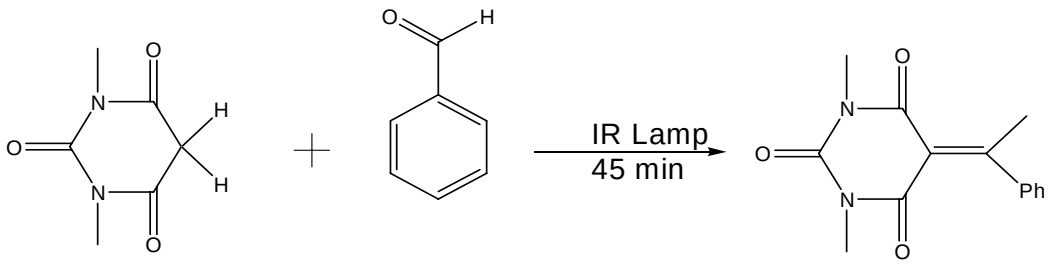
For the last one hundred years, barbituric acids and their simple C5-substituted derivates have had numerous applications in medicinal chemistry [17].

The condensation of 1,3-dimethylbarbituric acid with carbonyl compounds leads to 5-ylidene derivatives of 1,3-dimethylbarbituric acid **14**. The interest to these reactions has been generated mostly by the search of new biologically active substances [18].



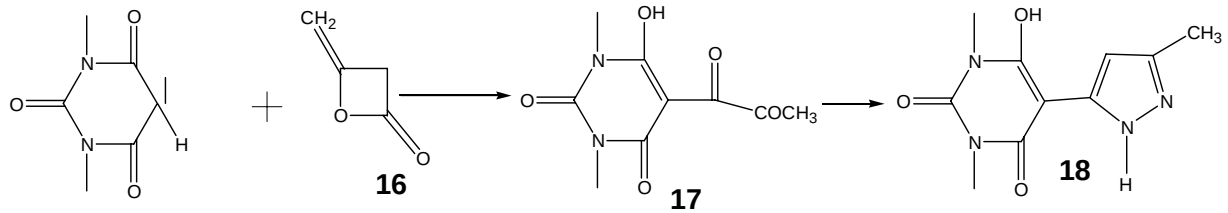
14

Several benzaldehydes can condense with barbituric acid derivatives under infrared irradiation, in absence of solvents affording the corresponding 5-benzylidene barbituric acid **15** [19].



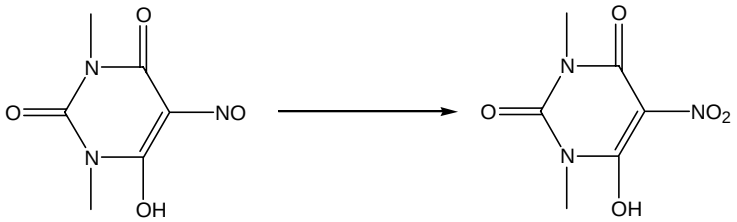
15

Diketene **16** reacted with 1,3-dimethylbarbituric acid in the presence of the basic catalyst and gives the acetoacetyl derivatives **17** in excellent yields. Condensation of the resulting product with hydrazine gave the pyrazole **18** [20].



16 **17** **18**

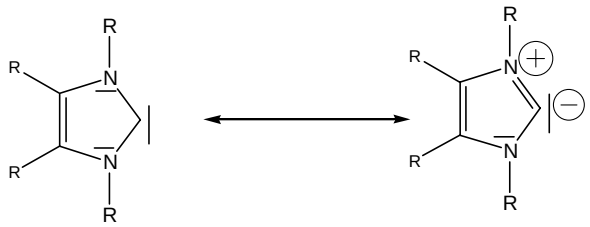
Oxidation of 5-nitroso pyrimidine **19** with 30 % hydrogen peroxide in trifluoroacetic acid produced 5-nitro pyrimidine **20**. The nitro group in the 5-position of a pyrimidine ring activates a 6-substitution towards nucleophilic displacement reactions [21].



19 **20**

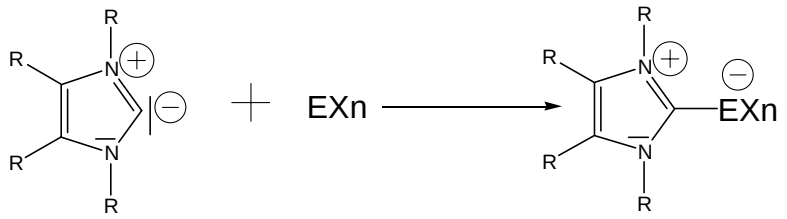
1.3 Nucleophilic and electrophilic carbene fragments

Owing to their highly nucleophilic character, heterocyclic carbenes **21** act as a ligand in complexes of metal and metalloid centres in a manner similar to tertiary phosphanes.

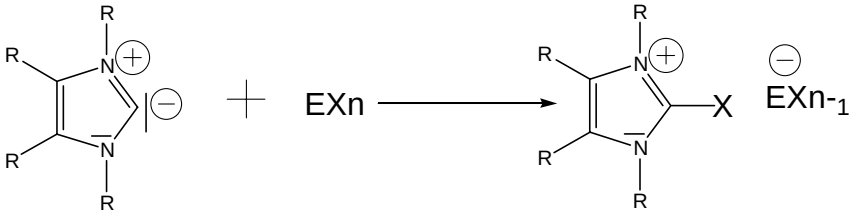


21

It has been reported that they can coordinate through the central element to give compound **22** (EXn = SeI_2 [22], BH_3 [23]), or can abstract a halogen atom to give compound **23** (EXn = SO_2ClF, SO_2F_2 [24]).

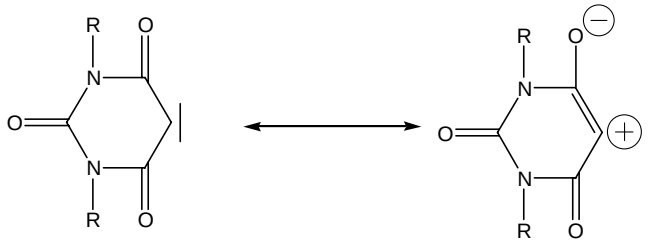


22

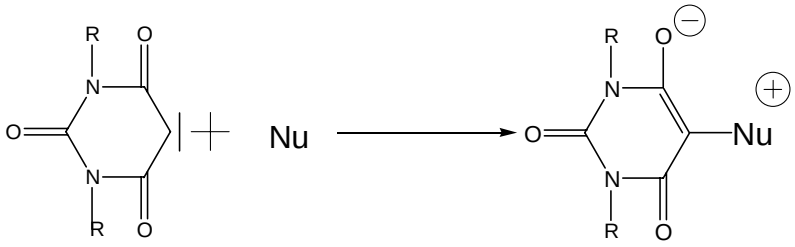


23

In contrast to heterocyclic carbenes, the unstable barbituric acid carbene **24** may has high susceptibility to nucleophilic attack at carbon atom between the two carbonyl groups as well as reported for meldrum's acid carbene **25** [25].



24

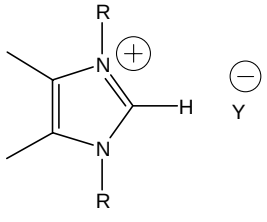


25

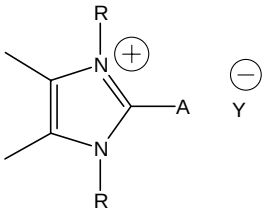
2. Aim of Study

The aims of this study were the preparation of some useful organic and inorganic derivatives of the imidazol-2-ylidenes and 1,3-dimethylbarbituric acid **13** by using new and simple methods. The compounds will be characterized by physical methods in addition to the X-ray structure analysis. The derivatives can be classified into:

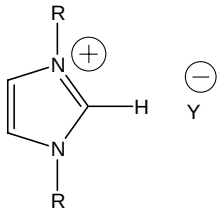
2.1 Salt derivatives of the types of 2,3-dihydroimidazole-2-ylidenes



26 a

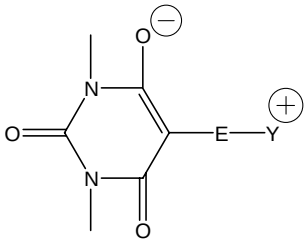


26 b



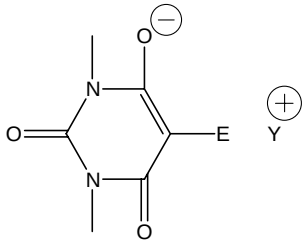
26 c

2.2 Zwitterionic derivatives of 1,3-dimethylbarbituric acid



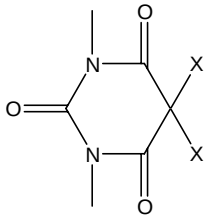
27

2.3 Salt derivatives of 1,3-dimethylbarbituric acid

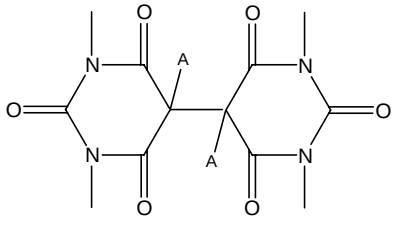


28

2.4 Neutral derivatives of 1,3-dimethylbarbituric acid



29



30

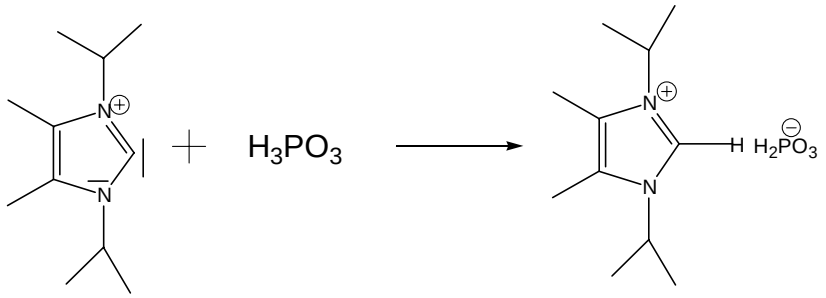
3. Results and Discussion

3.1 Carbenes

3.1.1 Nucleophilic reactions of imidazole-2-ylidenes

3.1.1.1 Synthesis of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium phosphonate (**31**)

The reaction of **4c** with H_3PO_3 gives **31** as stable crystalline solid in good yield.



4c **31**

The molecular structure of **31** was confirmed by X-ray crystallography (tab. 1-3, fig. 1) [26]. **31** crystallizes in the monoclinic space group p2(1)/c. The ^{31}P-NMR spectrum shows a chemical shift of 12.4 ppm relative to 5.0 ppm of H_3PO_3. The two crystallographically independent molecules are found to be similar. The bond angles O(12)-P(1)-O(11) [110.10°], O(12)-P(1)-O(13) [116.78°] and O(11)-P(1)-O(13) [110.28°] are close to those found in the molecular structure of the H_3PO_3 [27] except that the bond angle of OH-P-OH [101°] in H_3PO_3 relative to 110° in **31**.

The bond length P(1)-O(11) [1.554(3) Å] is longer than bond length of each P(1)-O(12) [1.506(3) Å] and P(1)-O(13) [1467(3) Å] due to negative charge distributed at O(12) and O(13). The hydrogen bonds trained in **31** [C(3)-H(3) 0.967 Å, H(3)-O(22) 1.973 Å; C(3)-H(3)….O(22) 169.6°, C(14)-H(14) 0.910 Å, H(14)-O(13) 2.114 Å; C(14)-H(14)….O(13) 179.0°] lie within the standard range of C-H….O bridges.

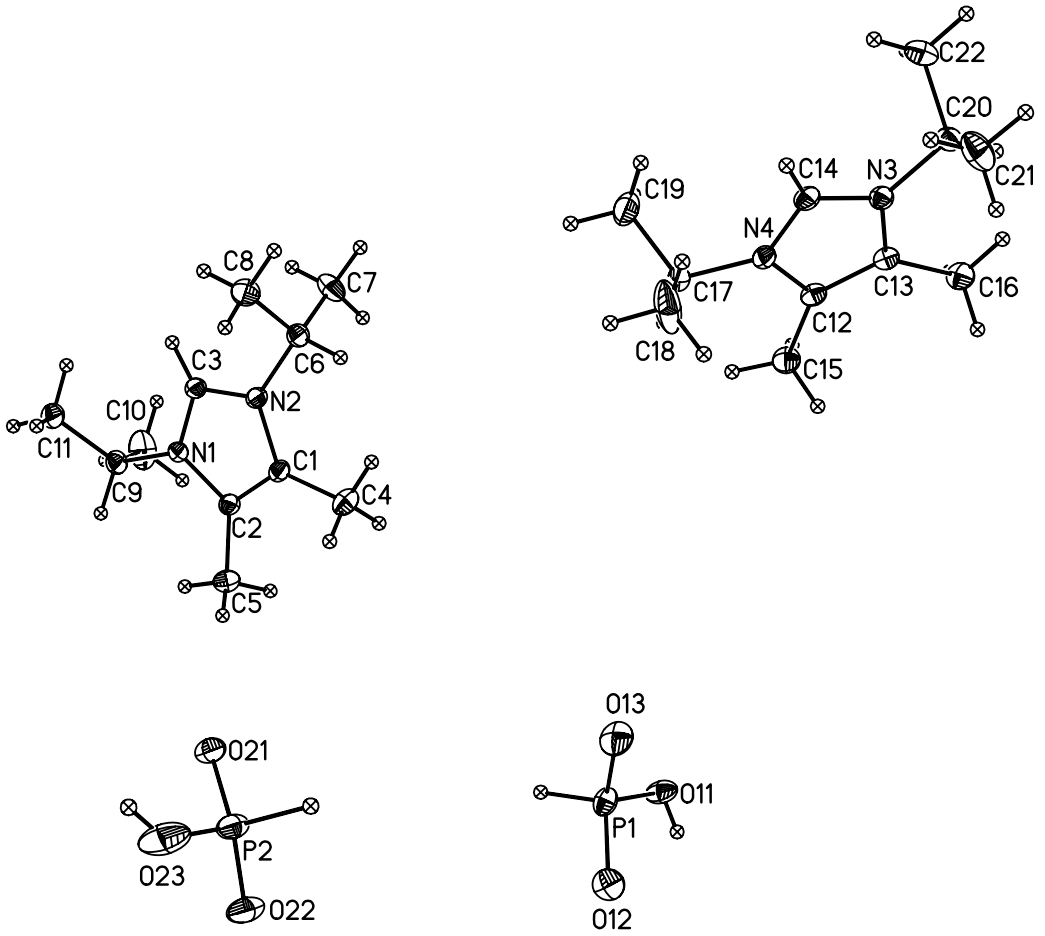
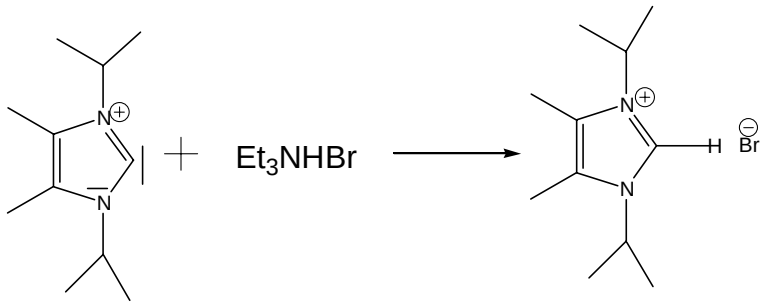


Fig. 1: The crystal structure of **31**

3.1.1.2 Synthesis of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium bromide (**32**)

The reaction of **4c** with Et_3NHBr gives **32** as stable crystalline solid in good yield.



4c **32**

32 crystallizes in the orthorhombic space group C222(1). The molecular structure of **32** was confirmed by X-ray crystallography (tab. 4-6, fig. 2) [28]. The molecular structure of **32** is in agreement with other imidazolium salts which are reported [29]. It proves interionic connection through a hydrogen bridge and the crystal structure reveals the presence of a near linear C-H….Br fragment [C(2)-H(2) 0.950(3) Å, H(2)-Br(2) 2.609(4) Å; C(1)-H(1)-Br(2) 180°].

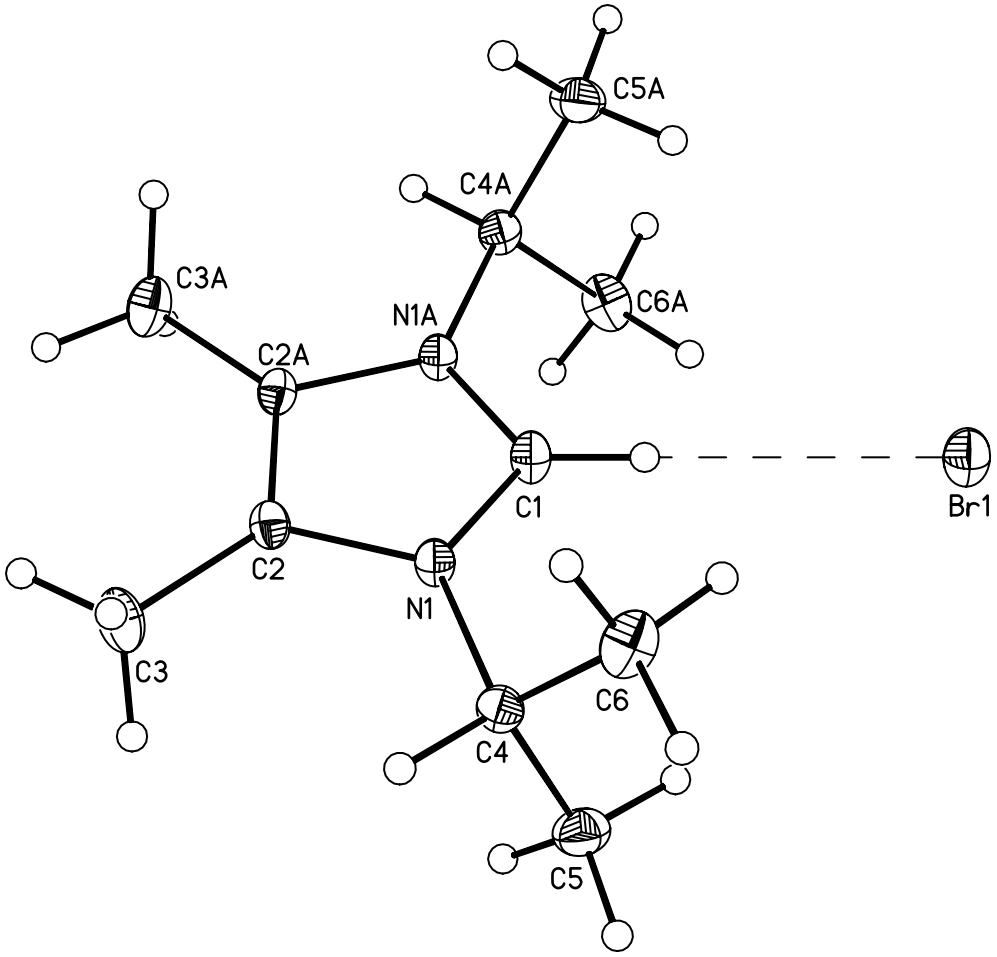
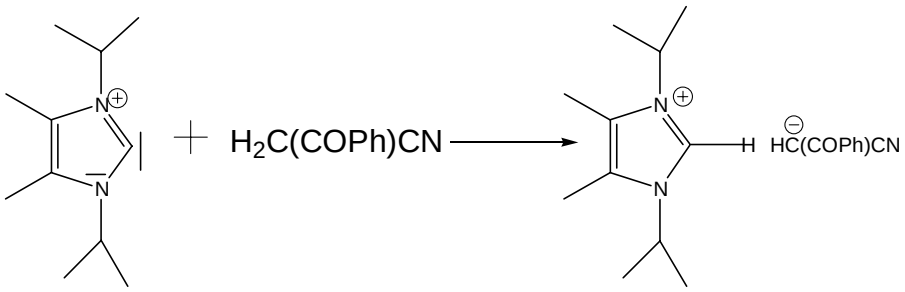


Fig. 2: The crystal structure of **32**

3.1.1.3 Synthesis of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium (*Z*)-2-cyano-1-phenyl-1-ethenolate (**33**)

The reaction of **4c** with $PhCOCH_2CN$ gives **33** as stable crystalline solid in good yield.



4c **33**

The molecular structure of **33** was confirmed by X-ray crystallography (tab. 7-9, fig. 3) [30]. **33** crystallizes in the monoclinic space group P2(1)/c. The hydrogen bonds trained in **33** [C(1)-H(1) 0.969 Å, H(1)-O(1) 2.196 Å; C(1)-H(1)-O(1) 160.3°] lie within the standard range of C-H….O bridges. From the crystal structure analysis, the structure of the anion exhibits the expansion of the C-C double bond with a parallel shortage of the C-O single bond [C(13)-C(14) 1.384(3) Å, C(14)-O(1) 1.265(2) Å]. Despite the π-electron distribution in the enolate, no marked twisting of the fragments was observed along the olefin double bond. The dihedral angle C(12)-C(13)-C(14)-O(1) is [179.1°]. The ^{13}C-NMR spectrum shows a chemical shift of 135.1 ppm for the carbon atom in the CN, this can be attributed due to the presence of the negative charge next to CN. Interestingly, the NMR results confirmed that the anion of **33** has the ***Z***-isomer rather than ***E***-isomer in spite of the ***E***-isomer has more favorable charge compensation when the oxygen atom is trans to the nitril group.

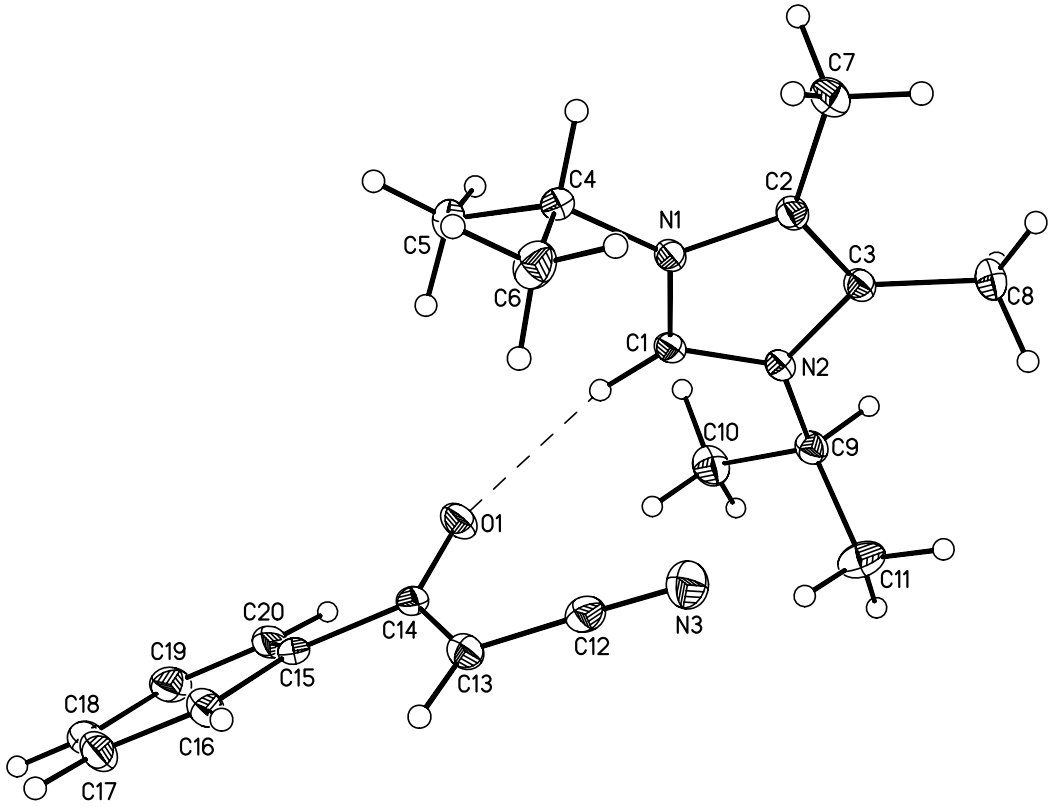
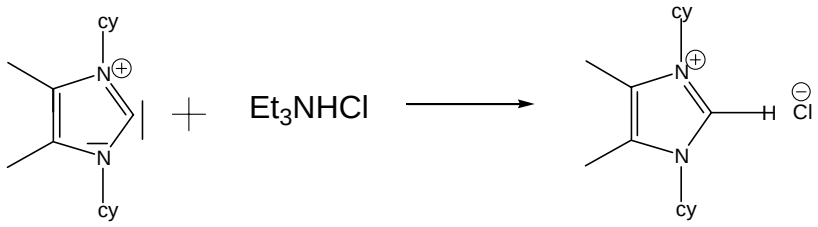


Fig. 3: The crystal structure of **33**

3.1.1.4 Synthesis of 1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium chloride (**35**)

The reaction of **34** with Et_3NHCl gives **35** as stable crystalline solid in good yield.



34 **35**

35 crystallizes in the monoclinic space group P2(1)/n. The molecular structure of **35** was confirmed by X-ray crystallography (tab. 10-12, fig. 4) [31]. The molecular structure of **35** is in agreement with other imidazolium salts which are reported [32]. It proves interionic connection through hydrogen bridges and the crystal structure reveals the presence of a near linear C-H….Cl fragment [C(1)-H(1) 0.950 Å, H(1)-Cl(1) 2.489 Å; C(2)-H(2)-Cl(1) 166.4°]. Interestingly, the presence of the hydrogen bonds to the solvents molecules [H(18C)-Cl(1A) 2.520 Å, H(18D)-Cl(1) 2.602 Å; C(18A)-H(18D)-Cl(1) 157.2°, C(18A)-H(18C)-Cl(1A) 162.2°] could be observed.

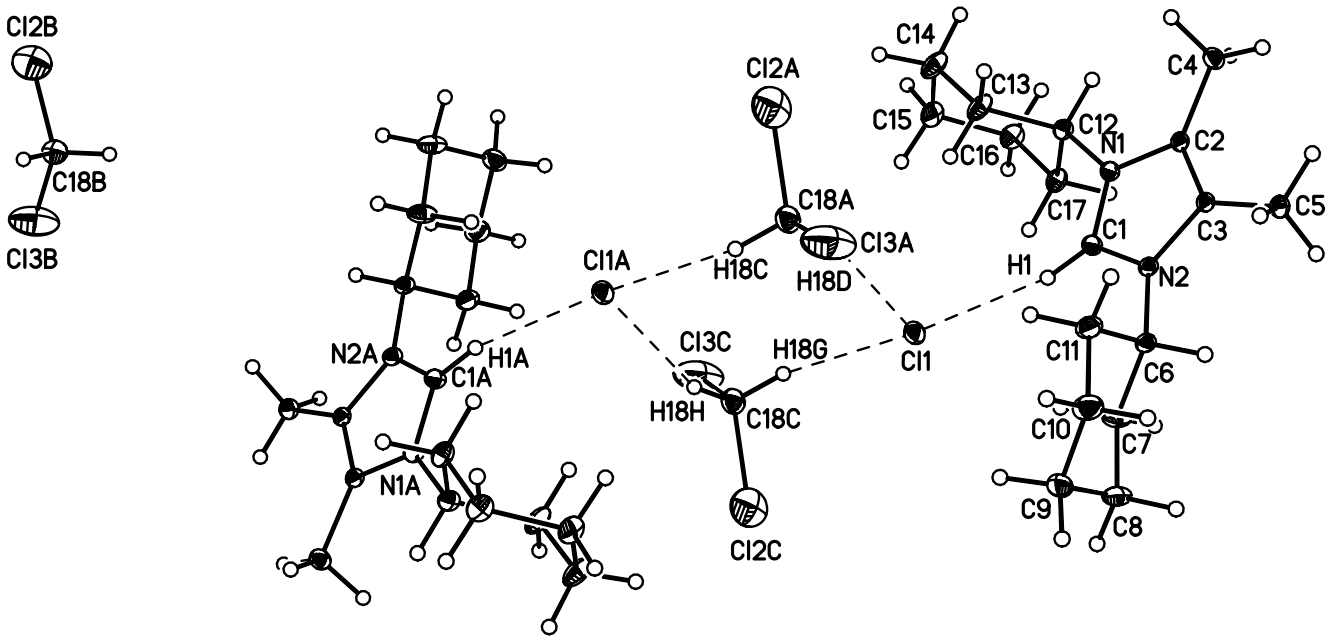
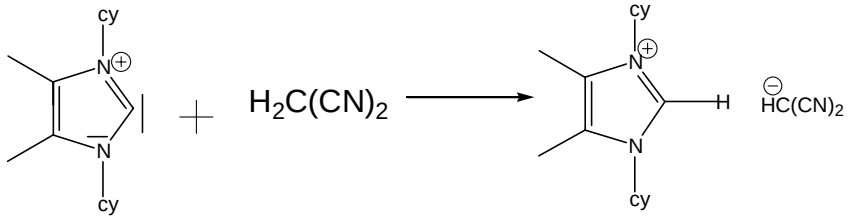


Fig. 4: The crystal structure of **35**

3.1.1.5 Synthesis of 1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium dicyanomethylide (**36**)

The reaction of **34** with $H_2C(CN)_2$ gives **36** as stable crystalline solid in good yield.



34 **36**

36 crystallizes in the monoclinic space group P2(1)/C. The molecular structure of **36** was confirmed by X-ray crystallography (tab. 13-15, fig. 5) [33]. The hydrogen bonds trained in **36** [C(1)-H(1) 0.930(3) Å, H(1)-N(4) 2.280(2) Å; C(1)-H(1)-N(4) 166.5 (1)°] lie within the standard

range of C-H….N bridges. The structure of the anion is a little affected by the monofunctional binding of the cation [C(18)-C(19) 1.383(2) Å, C(18)-C(20) 1.387(2) Å, C(19)-N(4) 1.158(16) Å, C(20)-N(3) 1.157(2) Å; C(19)-C(18)-C(20) 121.81(11)°, C(18)-C(19)-N(4) 178.25(12)°, C(18)-C(20)-N(3) 177.98 (14)°].

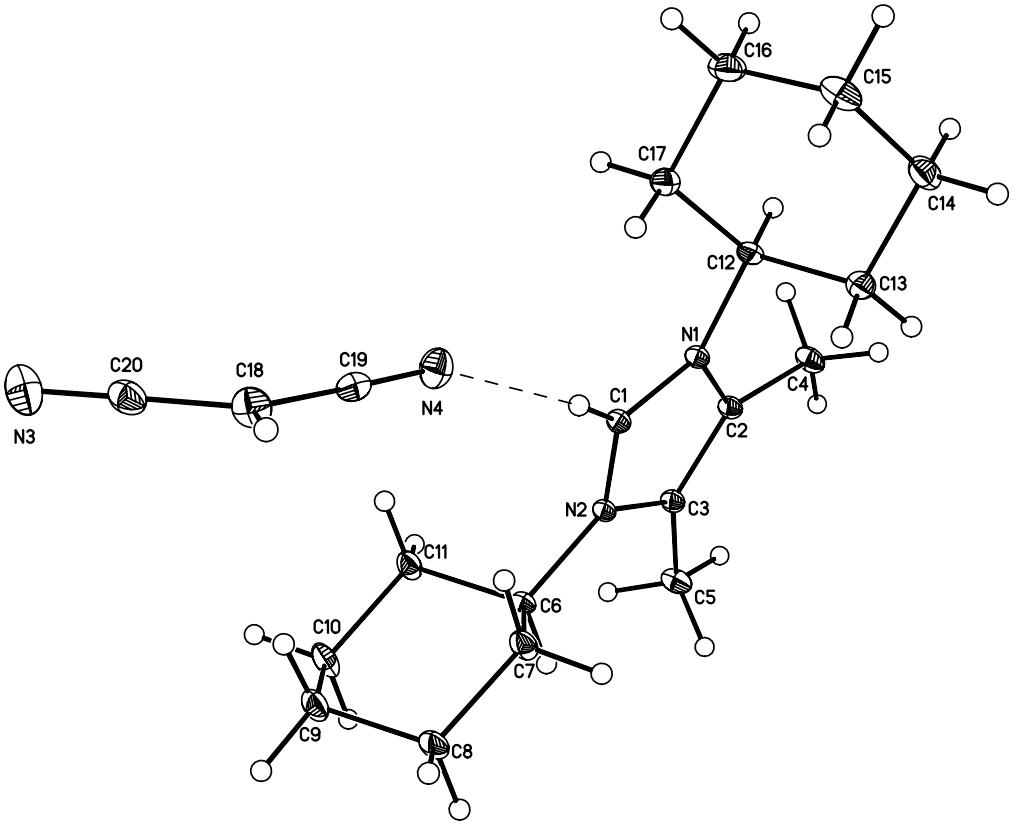
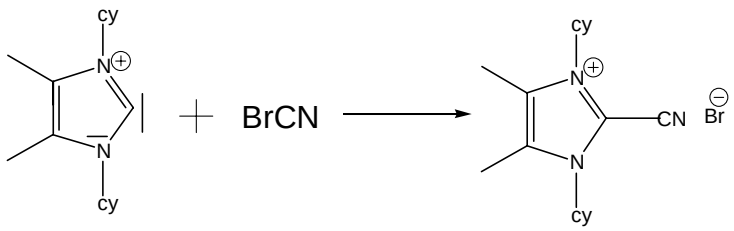


Fig. 5: The crystal structure of **36**

3.1.1.6 Synthesis of 2-cyano-1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium bromide (**37**)

The reaction of **34** with BrCN gives **37** as stable crystalline solid in good yield.



34 **37**

The molecular structure of **37** was confirmed by X-ray crystallography (tab. 16-18, fig. 6) [34]. **37** crystallizes in the orthorhombic space group Pbcn. The bond length C(21)-N(21) [1.19(2) Å] is close to the bond length C(1)-N(3) in **44**, while the bond angle N(21)-C(21)-C(2) [169.1°] has a little deviation from the linearity compared to the bond angle N(3)-C(1)-C(2) [178.5°] in **44**. The characteristic CN vibration band has been found at 2301 cm^{-1}.

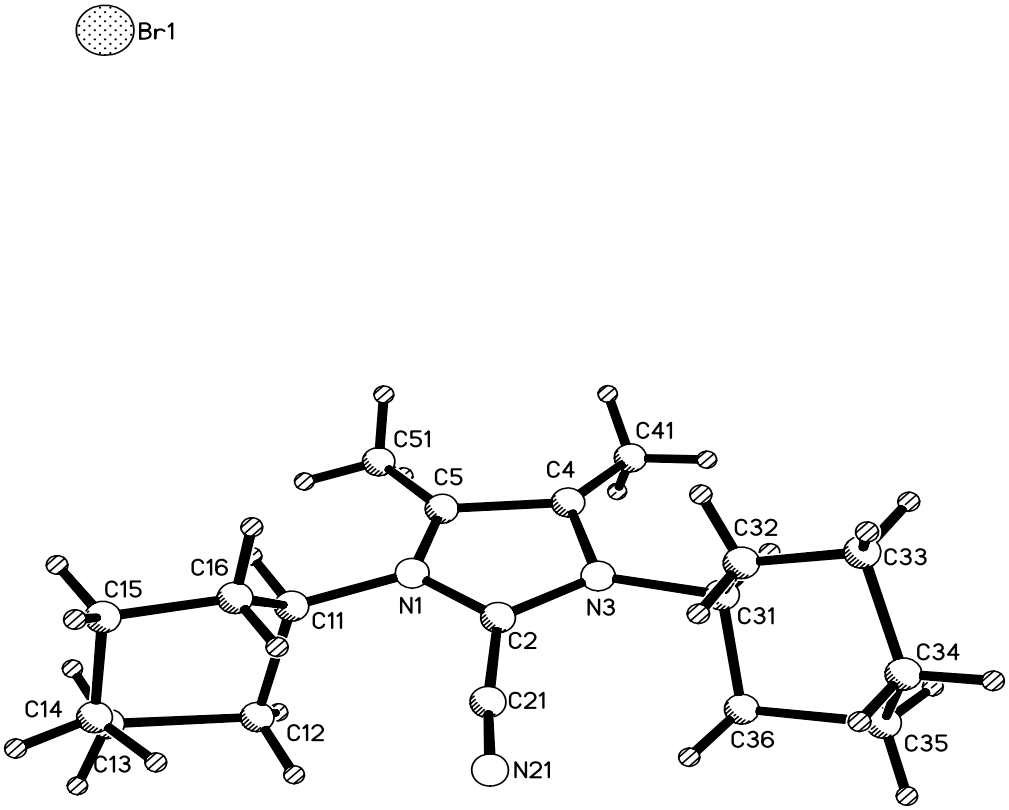
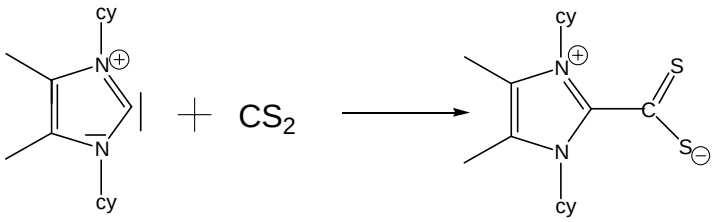


Fig. 6: The crystal structure of **37**

3.1.1.7 Synthesis of 1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium-2-carbodithioate (**38**)

The reaction of **34** with CS_2 gives **38** as stable crystalline solid in good yield.



34 **38**

The molecular structure of **38** was confirmed by X-ray crystallography (tab. 19-21, fig. 7) [35]. **38** crystallizes in the monoclinic space group P21/c. The bond length C(1)-C(2) [1.486(4) Å] lies within the intermediate of the carbon-carbon single and double bonds lengths. The bond

lengths C(1)-S(1) [1.663(4) Å] and C(1)-S(2) [1.660(4) Å] are very close to bond lengths S(1)-C(1), S(2)-C(1) [1.670 (5) Å] in [$ImCS_2$][36]. Parallel to this, the expansion of the bond angle S(1)-C(1)-S(2) [129.5(2)°] and the reduction of the angle N(2)-C(2)-N(1) [108.8(3)°] were observed.

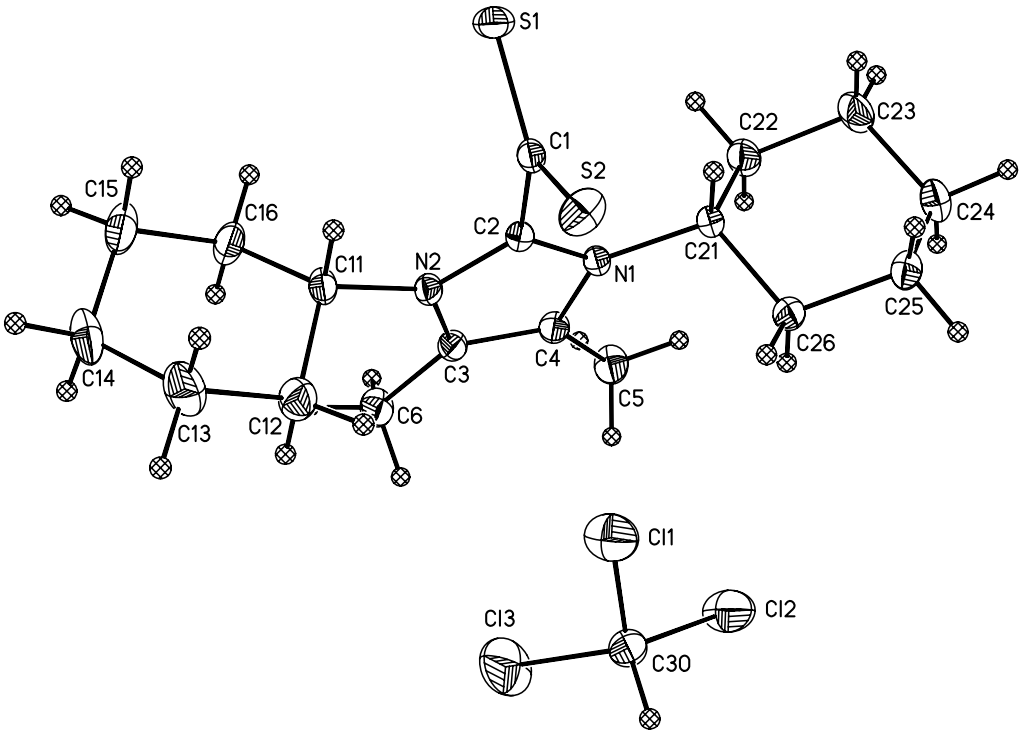
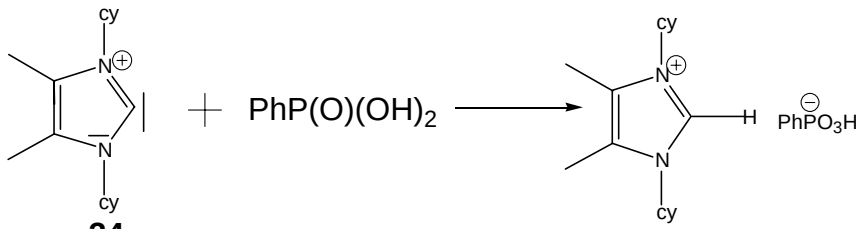


Fig. 7: The crystal structure of **38**

3.1.1.8 Synthesis of 1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium phenylphosphonate (**39**)

The reaction of **34** with $PhP(O)(OH)_2$ gives **39** as stable crystalline solid in good yield .



34 **39**

The molecular structure of **39** was confirmed by X-ray crystallography (tab. 22-24, fig. 8) [37]. **39** crystallizes in the monoclinic space group P2(1)/n. The ^{31}P-NMR spectrum shows a chemical shift of 13.05 ppm. The structure of the imidazolum part is compatible to its structure in **36**. The hydrogen bonds trained in **39** [C(1)-H(1) 0.953 Å, H(1)-O(3) 1.969 Å; C(1)-H(1)-O(3) 170.5°] lie within the standard range of C-H….O bridges. The bond lengths P(1)-O(1) [1.5124(16) Å] and P(1)-O(3) [1.4891(18) Å] are shorter than the bond length P(1)-O(2) [1.5736(18) Å]. The bond angles O(3)-P(1)-O(1) [117.21°], O(3)-P(1)-O(2) [109.71°] and O(1)-P(1)-O(2) [109.25°] are close to those found in the $PhP(O)(OH)_2$ [38].

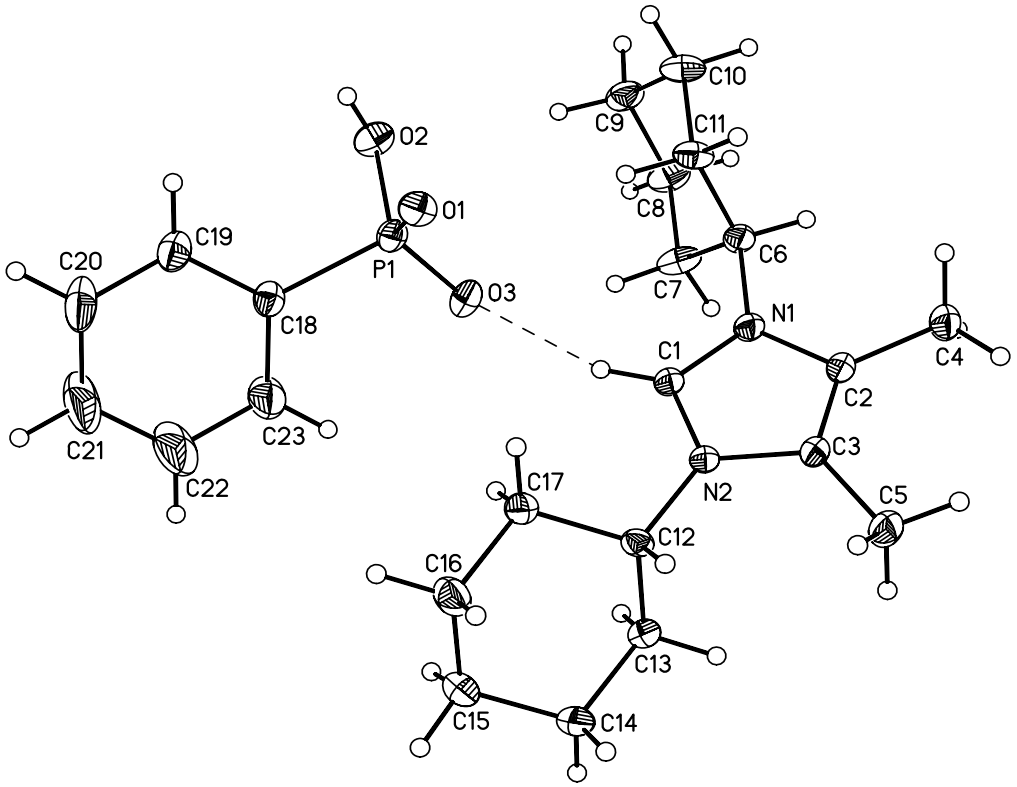
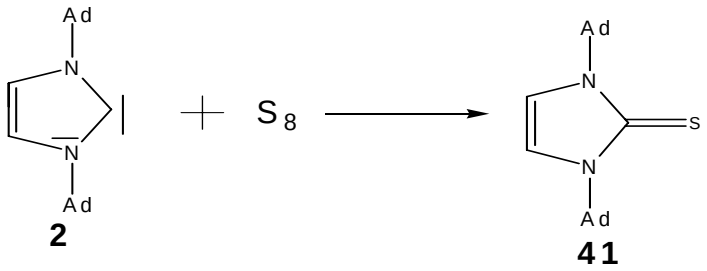


Fig. 8: The crystal structure of **39**

3.1.1.9 Synthesis of 1,3-di(1-adamantyl)-1,3-dihydro-2H-imidazole-2-thione (**41**)

The reaction of **2** with S_8 gives **41** as stable crystalline solid.



The molecular structure of **41** was confirmed by X-ray crystallography (tab. 25-27, fig. 9) [39]. **41** crystallizes in the trigonal space group P3(2)21. The bond length C(1)-S(1) [1.708(8) Å] is shorter than the single bond value of 1.81 Å and greater than the C=S bond value of 1.61 Å. It is representative of the C-S bonding in a selection of molecules containing the grouping –N-CS-X, where X= N or C [40].

The total summation of angles around C1 [S(1)-C(1)-N(1) 126.3°, N(1)-C(1)-N(1A) 107.4°, S(1)-C(1)-N(1A) 126.3°] gives exactly 360° [41], which indicates that the sulfur atom lies in the plane of the imidazolium ring.

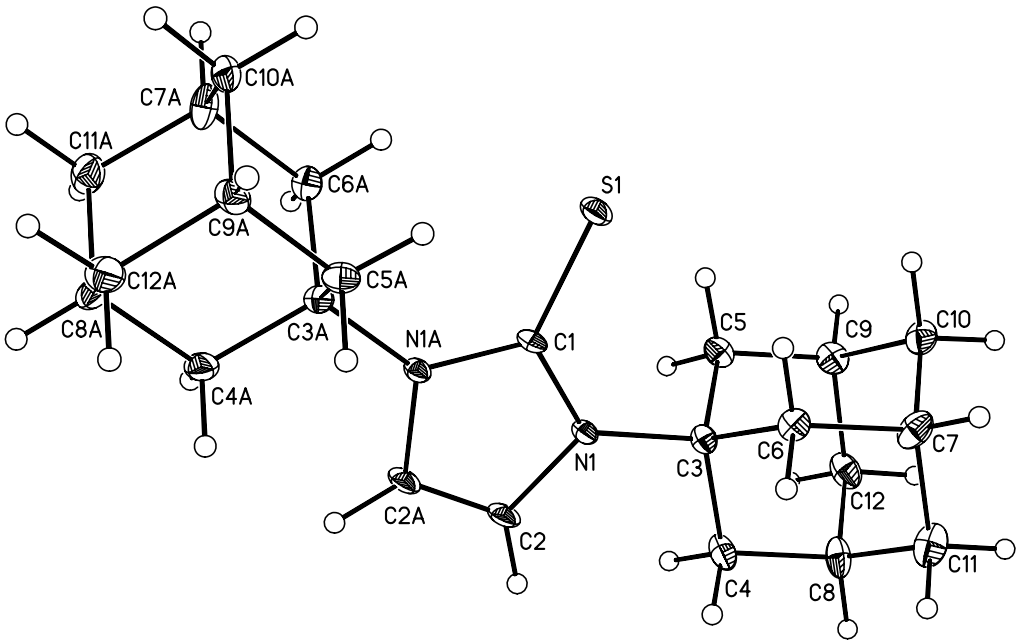
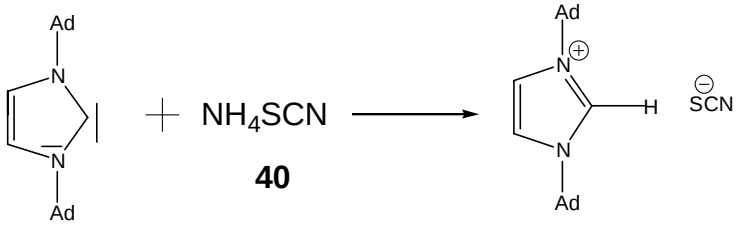


Fig. 9: The crystal structure of **41**

3.1.1.10 Synthesis of 1,3-di(1-adamantyl)-1H-imidazol-3-ium thiocyanate (**42**)

The reaction of **2** with $NH_4(SCN)$ gives **42** as stable crystalline solid in good yield .



2 **42**

The molecular structure of **42** was confirmed by X-ray crystallography (tab. 28-30, fig. 10) [42]. **42** crystallizes in the tricilinc space group P1. The bond angle S(1)-C(47)-N(5) [166.7°] is near linear. The distance between H(24)-S(4) [3.208 Å] lies within the *Van der Waals* distance range. This contact was also observed in other organic salts of the thiocyanate anion [43]. The bonds lengths S(4)-C(50) [1.651(16) Å] and C(50)-N(8) [1.1038(18) Å] lie within the normal range of the S-C≡N bond lengths.

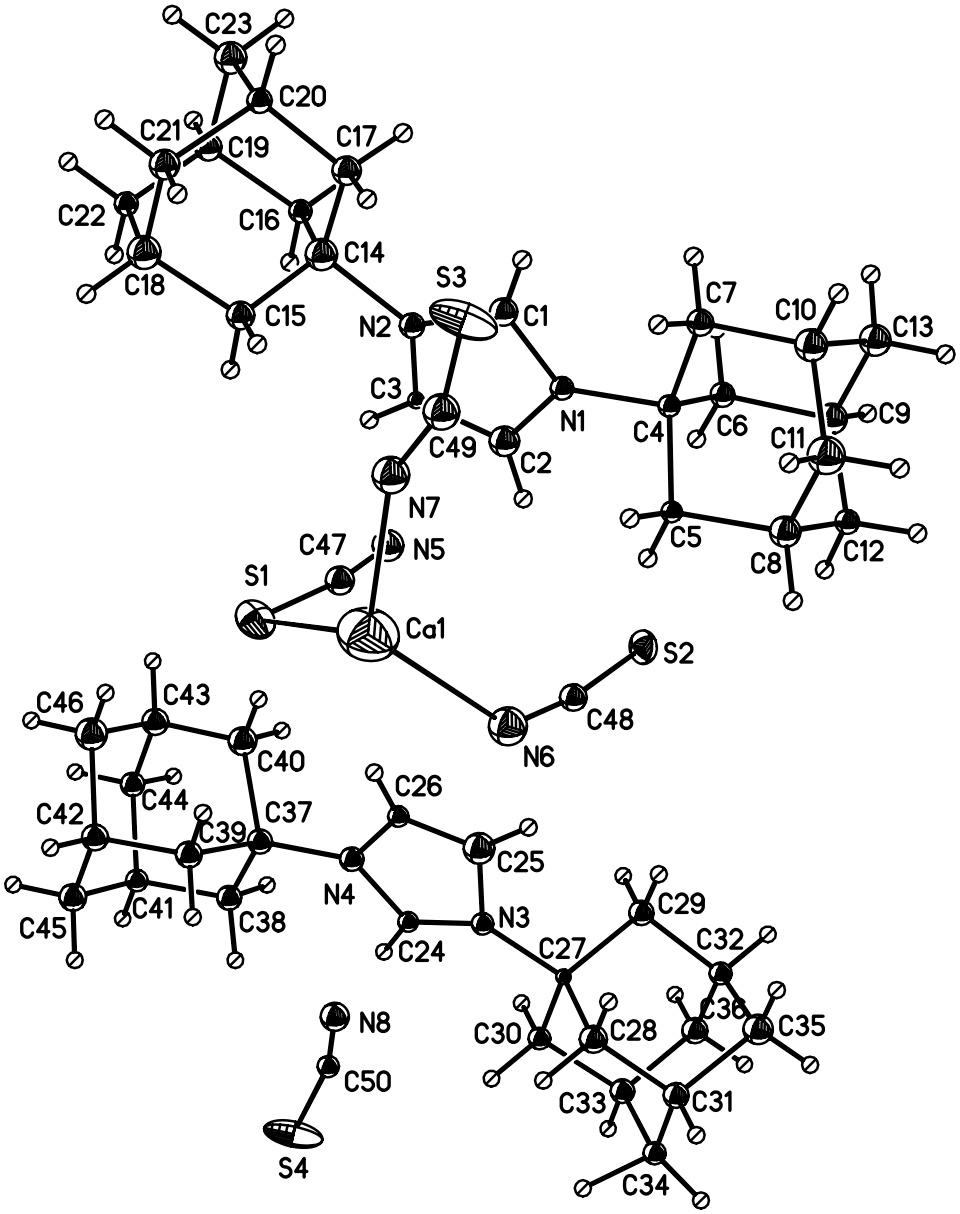
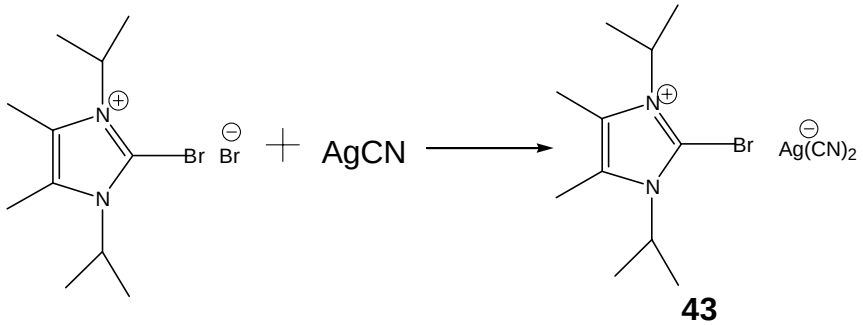


Fig. 10: The crystal structure of **42**

3.1.2 Nucleophilic substitution of 2-halo imidazolium salts

3.1.2.1 Synthesis of 2-bromo-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium dicyanoargentate (**43**)

The reaction of 2-bromo-1,3-diisppropyl-4,5-dimethylimidazolium bromide [44] with AgCN gives **43** as stable crystalline solid in good yield.



43

The molecular structure of **43** was confirmed by X-ray crystallography (tab. 31-33, fig. 11) [45]. **43** crystallizes in the orthorhombic space group P212121. The spectroscopic data of the cation in **43** are close to that observed in 2-bromo-1,3-diisppropyl-4,5-dimethylimidazolium bromide.

The bond lengths and angle C(11)-Ag(1) [2.046(11) Å], C(22)-Ag(1) [2.028(11) Å]; C(11)-Ag(1)-C(22) [177.5°] lie in the range of other Ag compounds [46]. The bond lengths C(22)-N(22) [1.137(11) Å] and C(11)-N(11) [1.114(11) Å] lie within the range of the C-N bond length of the CN group in [ImCl][$Ag(CN)_2$] [47].

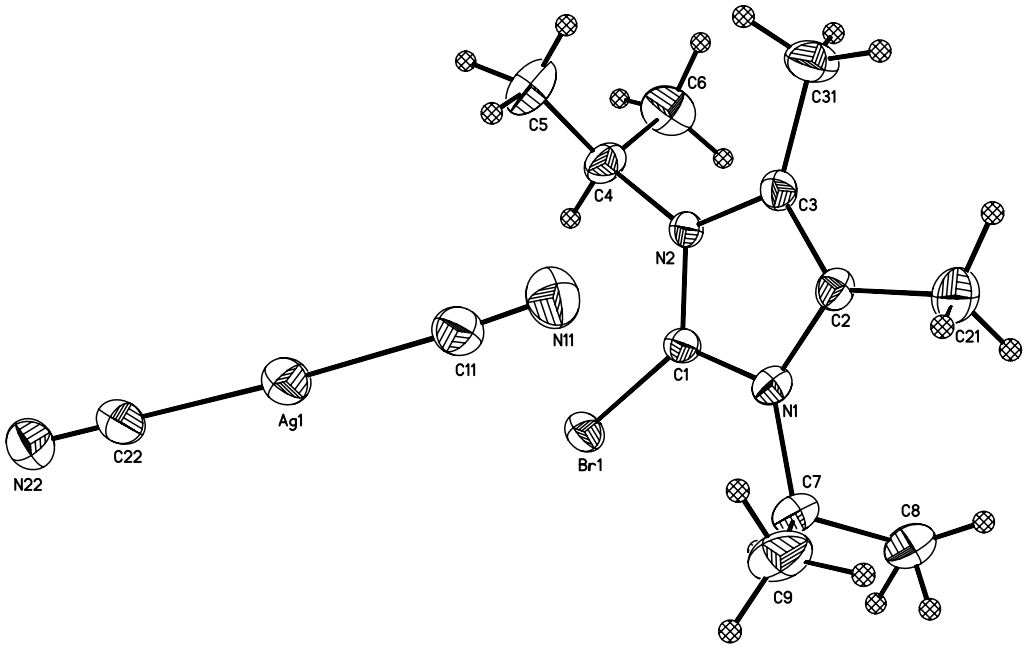
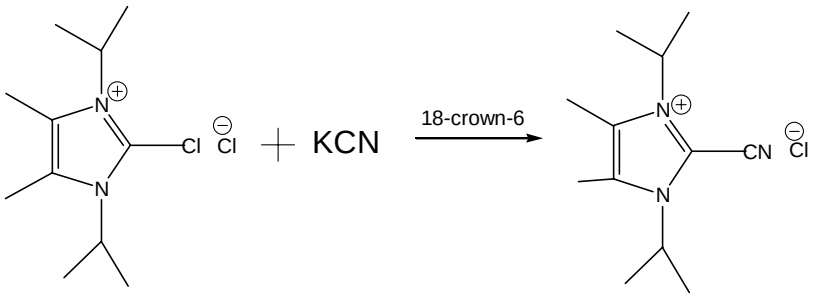


Fig. 11: The crystal structure of **43**

3.1.2.2 Synthesis of 2-cyano-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium chloride (**44**)

The reaction of 2-chloro-1,3-diisppropyl-4,5-dimethylimidazolium chloride [48] with KCN in presence of 18-crown-6 gives **44** as stable crystalline solid in good yield.



44

The molecular structure of **44** was confirmed by X-ray crystallography (tab. 34-36, fig. 12) [49]. **44** crystallizes in the orthorhombic space group Pna2(1). The structure of **44** was initially assigned in solution by ^{13}C-NMR. The NMR spectrum shows a chemical shift of C1 in the CN group of 107.6 ppm.

The characteristic CN vibration band has been found at 2301cm^{-1}. The bond length C(1)-N(3) [1.149(4) Å] lies within the normal range of bond length of the CN group, the bond angle N(3)-C(1)-C(2) is [178.5°] near linear. The presence of the CN group connected to C2 leads to the bond length C(2)-C(1) [1.427 (5) Å] which lies within the intermediate of the carbon-carbon single and double bonds lengths.

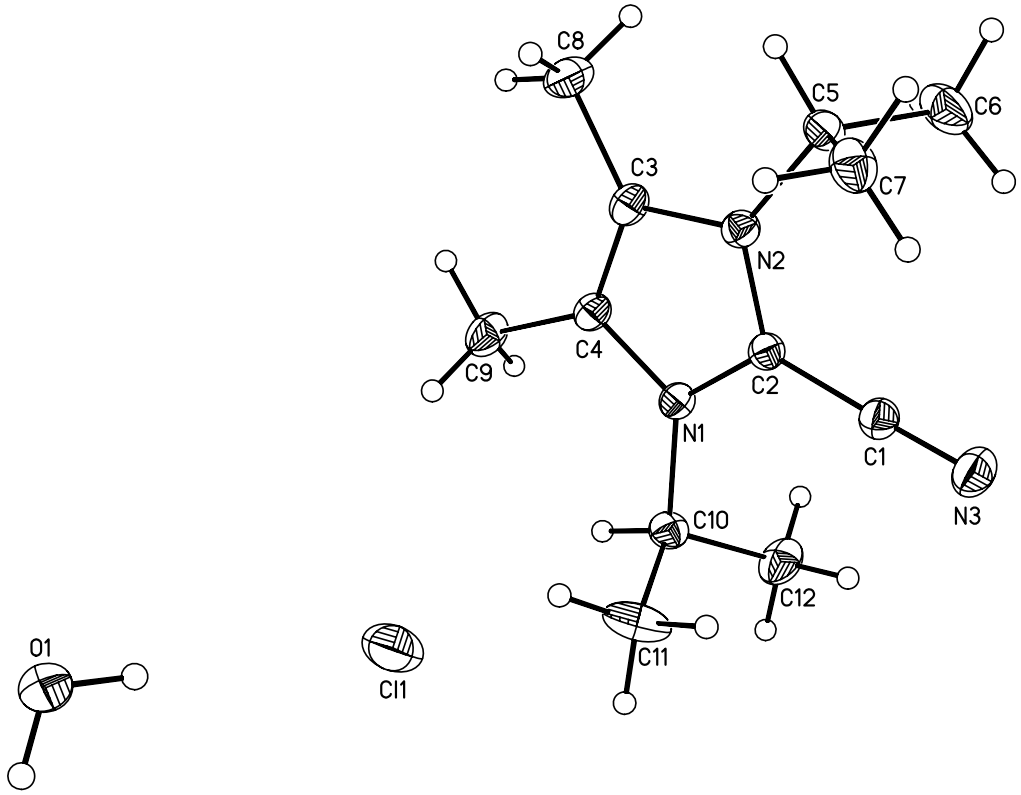
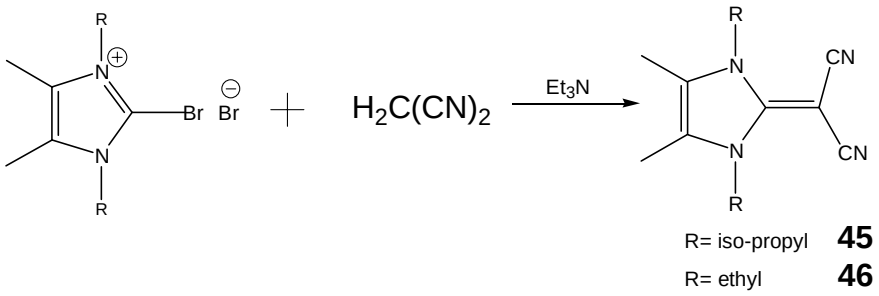


Fig. 12: The crystal structure of **44**

3.1.2.3 Synthesis of 2-(1,3-diethyl-4,5-dimethyl-1,3-dihydro-2H-imidazol-2-ylidene) malononitrile (**46**) and 2-(1,3-diisopropyl-4,5-dimethyl-1,3-dihydro-2H-imidazol-2-ylidene)malononitrile (**45**)

The reaction of 2-bromo-1,3-diethyl-4,5-dimethylimidazolium bromide [44] and 2-bromo-1,3-diisppropyl-4,5-dimethylimidazolium bromide [44] with $H_2C(CN)_2$ gives **45** and **46** respectively, as stable crystalline solids in good yield.



The molecular structures of **45** and **46** were confirmed by X-ray crystallography (tab. 37-42, fig. 13-14) [50]. **46** crystallizes in the monoclinic space group C2/c and **45** crystallizes in the monoclinic space group P2(1)/c. The crystal structure analyses of **45** and **46** show the influence of the steric requirement of the substituents in 1,3-position on the state of exocyclic double bond.

For both **45** and **46**, there is a tendency for the coplanar orientation of the five membered ring and the dicyano methylene fragments. The steric requirements actually force in 1,3-position to make a twisting of the molecule along the exocyclic olefinic double bond. The interplanar angle of **46** N(1)-C(1)-N(2)/C(11)-C(10)-C(12) is [34.7(2)°] and N(1)-C(1)-N(2)/C(13)-C(12)-C(14) is [48.6 (2)°] for **45**.

Due to this, the contact length C(14)-C(11) is [3.072 Å] in **46** and [3.108 Å] for C(4)-C(11) in **45**. This contact expands the carbon-carbon olefin double bond C(1)-C(10) [1.435(2) Å] in **46** and C(1)-C(12) [1.4455(10) Å] in **45**. A comparison of the structures between **45** and **46** shows the expected relationship between the interplanar angle between the five membered ring and the dicyanomethylene fragments and the bond length [**46**/**45**: (34.7/48.6); (1.435/1.445 Å].

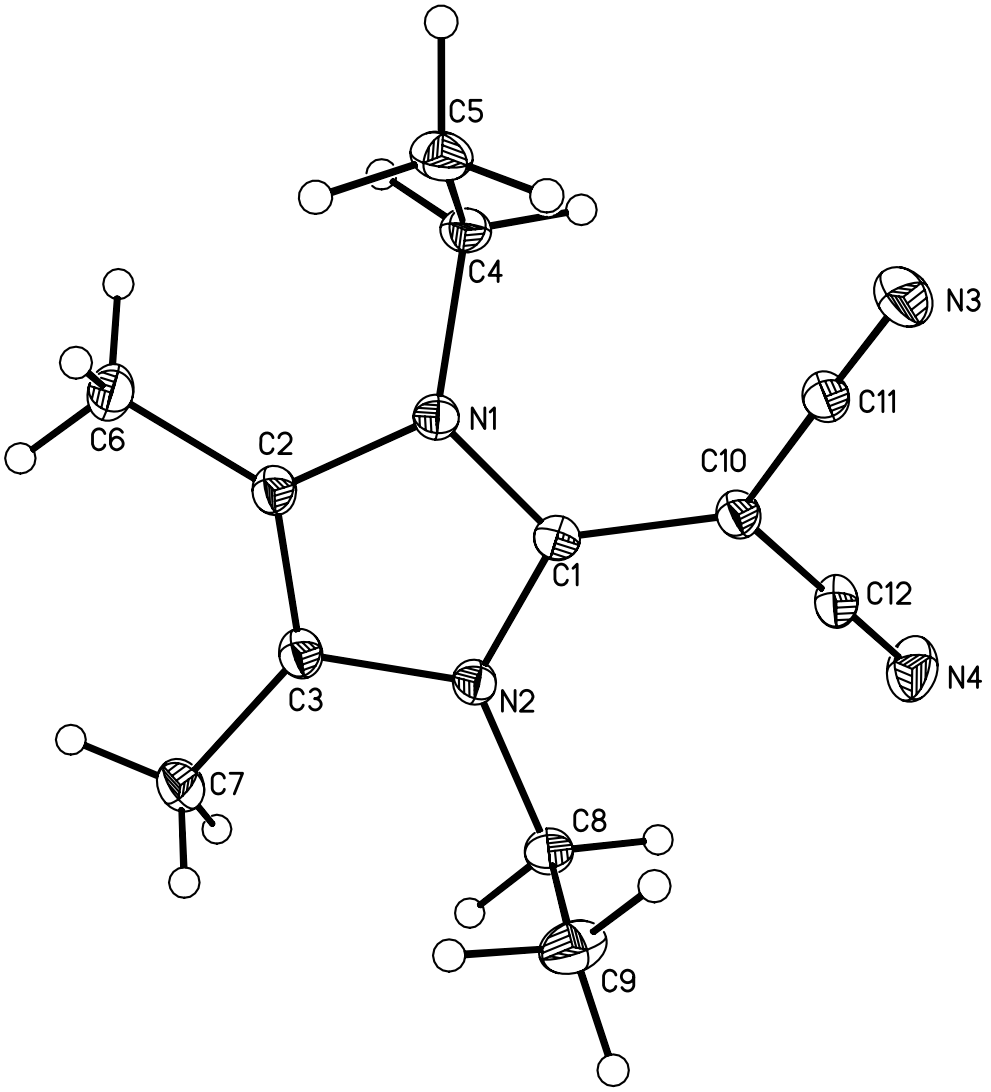


Fig. 13: The crystal structure of **46**

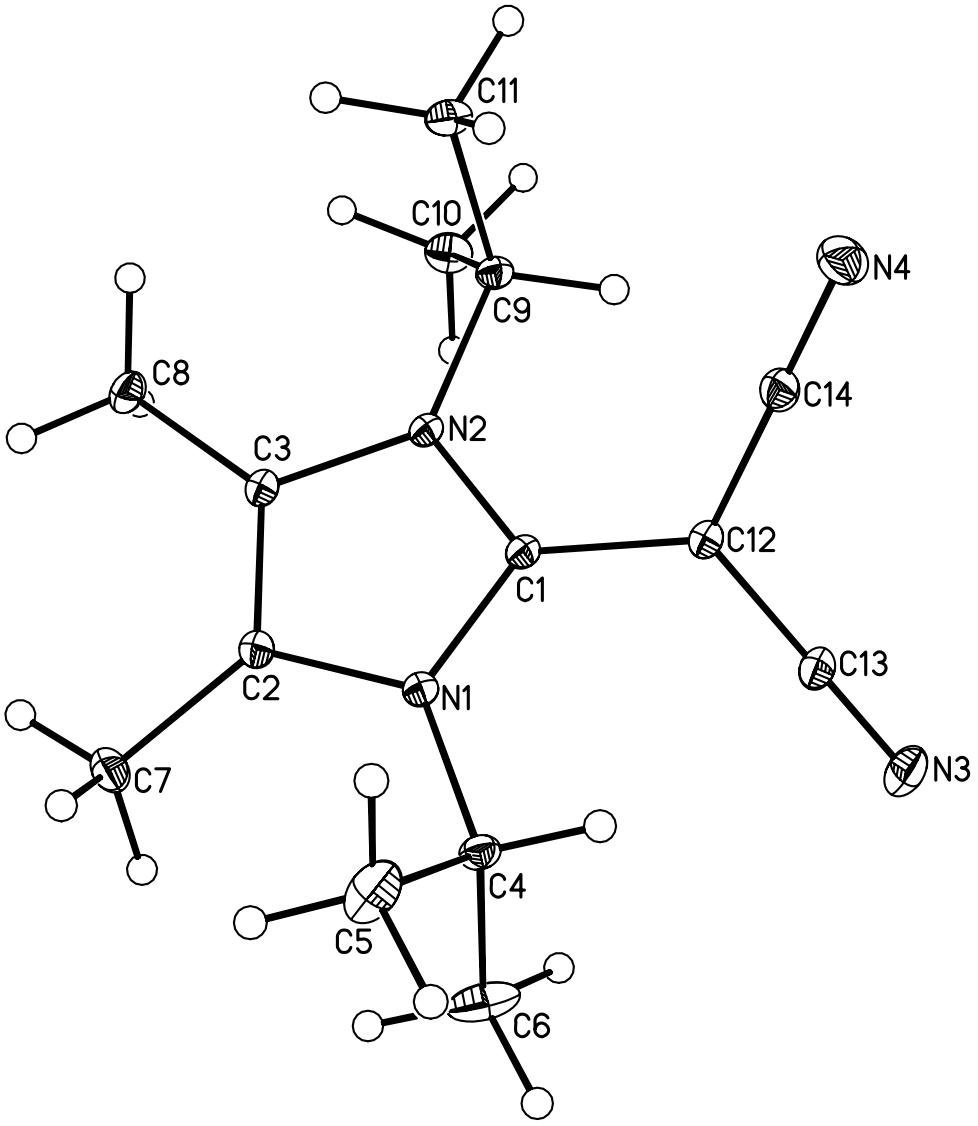
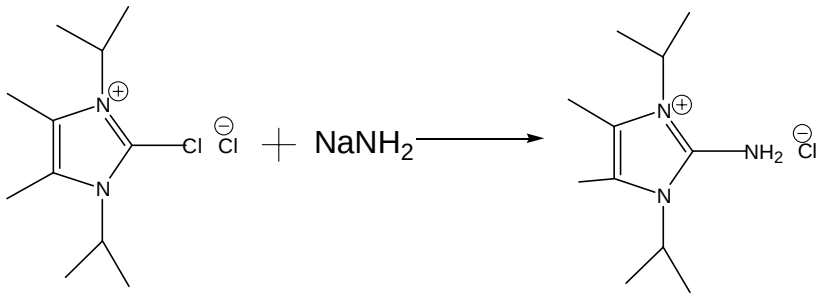


Fig. 14: The crystal structure of **45**

3.1.2.4 Synthesis of 2-amino-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium chloride (**47**)

The reaction of 2-chloro-1,3-diisppropyl-4,5-dimethylimidazolium chloride [48] with $NaNH_2$ gives **47** as stable crystalline solid.



47

47 crystallizes in the orthorhombic space group Pbca. The molecular structure of **47** was confirmed by X-ray crystallography (tab. 43-45, fig 15) [51]. The unit cell is composed of two cations and two anions.

The contacts between Cl(1)-H(1A) [2.012 Å], H(1b)-Cl(1A) [2.336 Å] lie within the *Van der Waals* distance range. The angle H(1A)-Cl(1)-H(1bb) is [78.2°], while there are no other contacts between N(1)-N(1A) or Cl(1)-Cl(1A). The bond lengths C(1)-N(1) [1.331(4) Å], C(1)-N(2) [1.356(4) Å] and C(1)-N(3) [1.339(4) Å] are close to each other, which indicates the partial double bond among them. The total summation of angles around C1 [N(1)-C(1)-N(3) 125.8°, N(1)-C(1)-N(2) 126.0°, N(3)-C(1)-N(2) 108.2°] gives exactly 360°, which indicates that the imidazole ring and the amino group are coplanar.

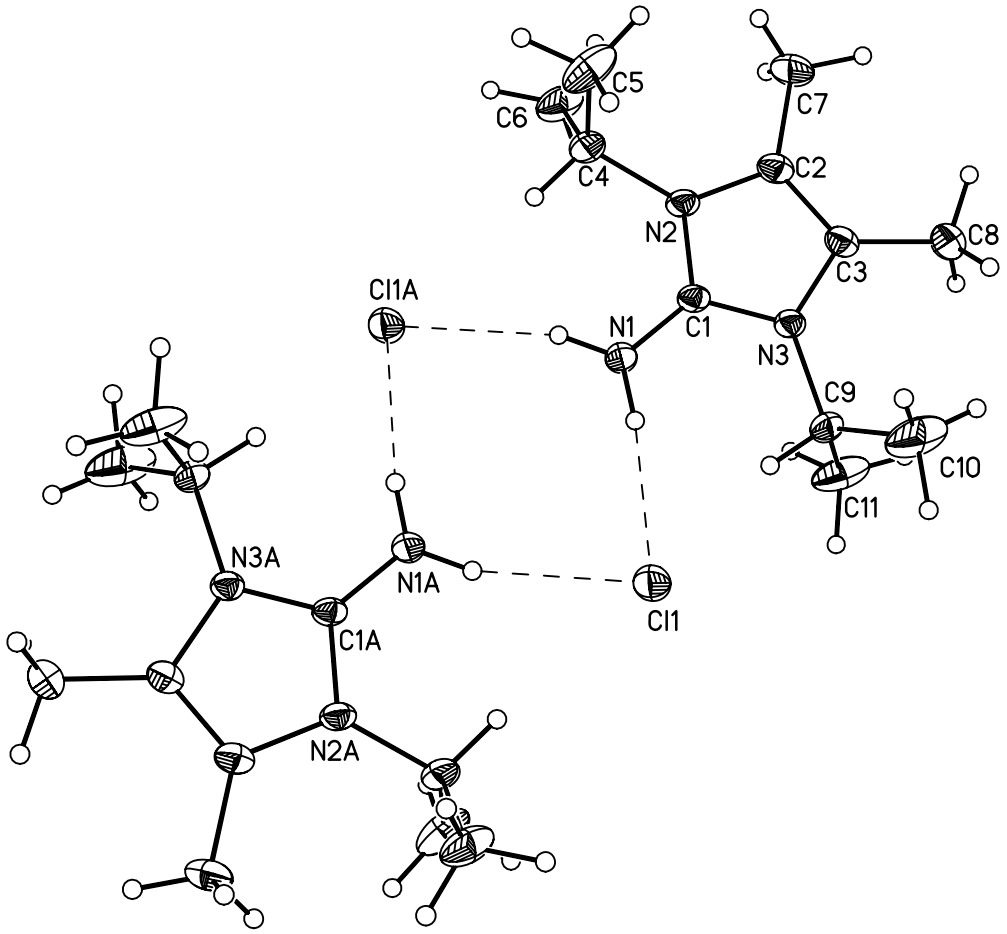
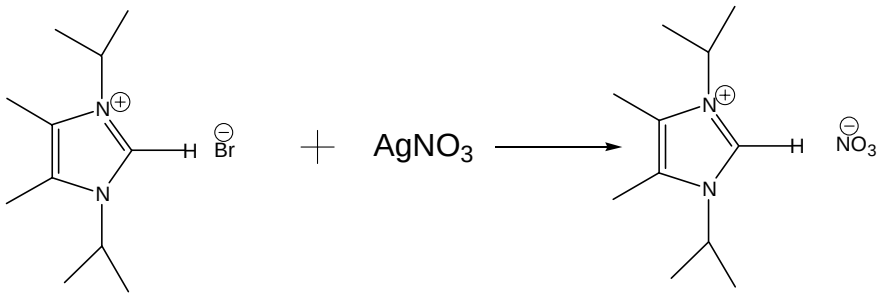


Fig. 15: The crystal structure of **47**

3.1.2.5 Synthesis of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium nitrate (**48**)

The reaction of **32** with $AgNO_3$ gives **48** as stable crystalline solid in good yield.



32 **48**

48 crystallizes in the tetragonal space group P4(3)2(1)2. The molecular structure of **48** was confirmed by X-ray crystallography (tab. 46-48, fig. 16) [52].The crystal structure analysis reveals the presence of ion pairs in which the ions are linked together by weak C-H-O hydrogen bonds. The nitrate ions are connected to the hydrogen atoms in 2-position of the cations in a bifurcated mode [C(1)-H(1A) 0.929(3) Å, H(1A)-O(2) 2.367(3) Å; O(2)-H(1A)-O(2A) 54.1(1)°].

Surprisingly, the protons of the methyl substituents in 4,5-positions are also capable to form hydrogen bonds [C(6)-H(6B) 0.988(3) Å, H(6B)-O(1A) 2.574(3) Å, C(6)-H(6B)-O(1A) 176.5(4)°; H(6B)-O(1A)-H(6BA) 53.4(4)°]. The bond lengths and angles in the nitrate ion indicate a weak and electrostatic interactions [N(2)-O(1) 1.225(2) Å, N(2)-O(2) 1.2463(14) Å, O(1)-N(2)-O(2) 120.26(7)°, O(2)-N(2)-O(2A) 119.48(15)°]. The planes of the cations and anions are orientated perpendicularly to each other forming infinite chains of ions similar to those obtained in the organic nitro derivatives [53], no stacking being present there. The planes of the neighboured five-membered rings exhibit parallel orientation. From the crystallographic reasons, the distances between the planes [4.407 Å] and the centers of the rings [7.967 Å] are outside the *Van* der *Waals* range.

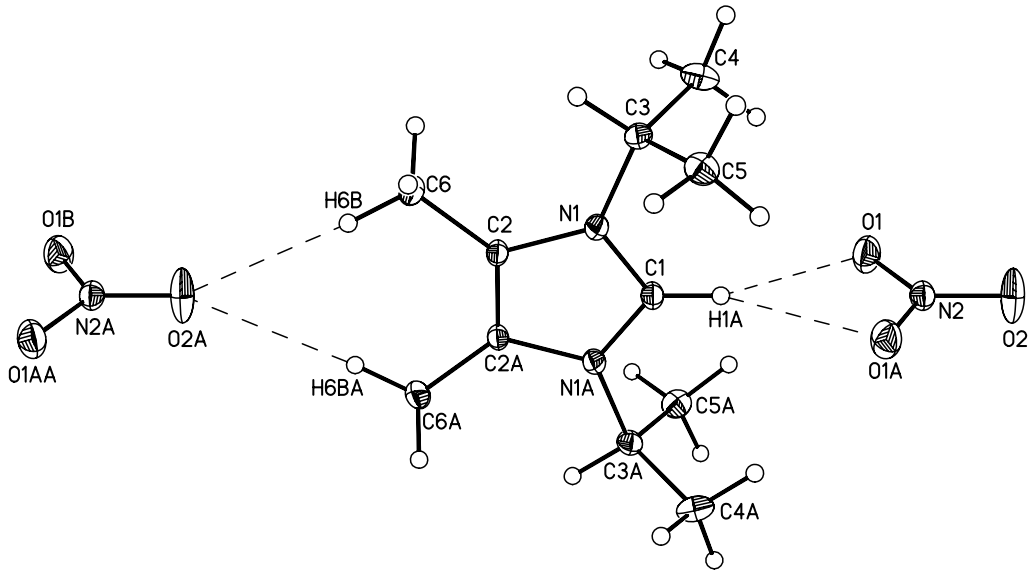
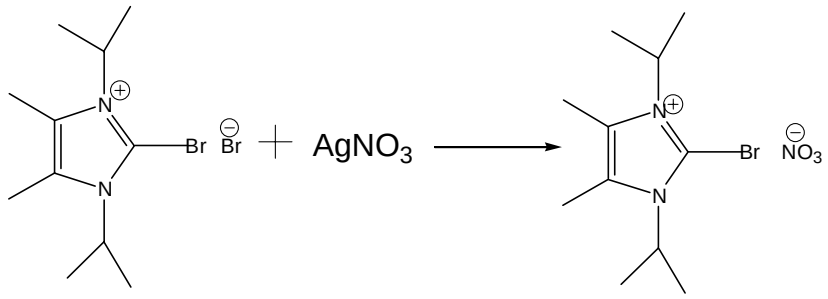


Fig. 16: The crystal structure of **48**

3.1.2.6 Synthesis of 2-bromo-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-iumnitrate (**49**)

The reaction of 2-bromo-1,3-diisppropyl-4,5-dimethylimidazolium bromide [44] with $AgNO_3$ gives **49** as stable crystalline solid in good yield.



49

49 crystallizes in the orthorhombic space group Cmcm. The molecular structure of **49** was confirmed by X-ray crystallography (tab. 49-51, fig. 17) [52].The crystal structure of **49** consists of coplanar orientated cations and anions which form stacks of alternating ions additionally connected by interactions between the ions of adjacent stacks. The very close contacts between the planes inside the stack [interplanar distance 3.364 Å] caused by the electrostatic interaction are influenced, in addition, by hydrogen bonds between methyl protons of the isopropyl substituents and oxygen atoms of the anions [H(5BB)-O(1C) 2.722(4) Å, H(5BB)-O(2B) 2.930(4) Å]. The contacts between the neighboured stacks are performed by the hydrogen bonds between the methyl substituents in 4,5-positions of the heterocyclic ring and one of the nitrate oxygen atom [C(4)-H(4B) 1.02(1) Å, H(4B)-O(2A) 2.735(4) Å; C(4)-H(4B)-O(2A) 167.8(1)°, H(4B)-O(2A)-H(4BA) 44.5(1)°], surprisingly, by close Br-O contact which is inside the *Van der Waals* range [Br(1)-O(1) 3.005(4) Å; O(1)-Br(1)-O(1A) 41.8 (1)°].

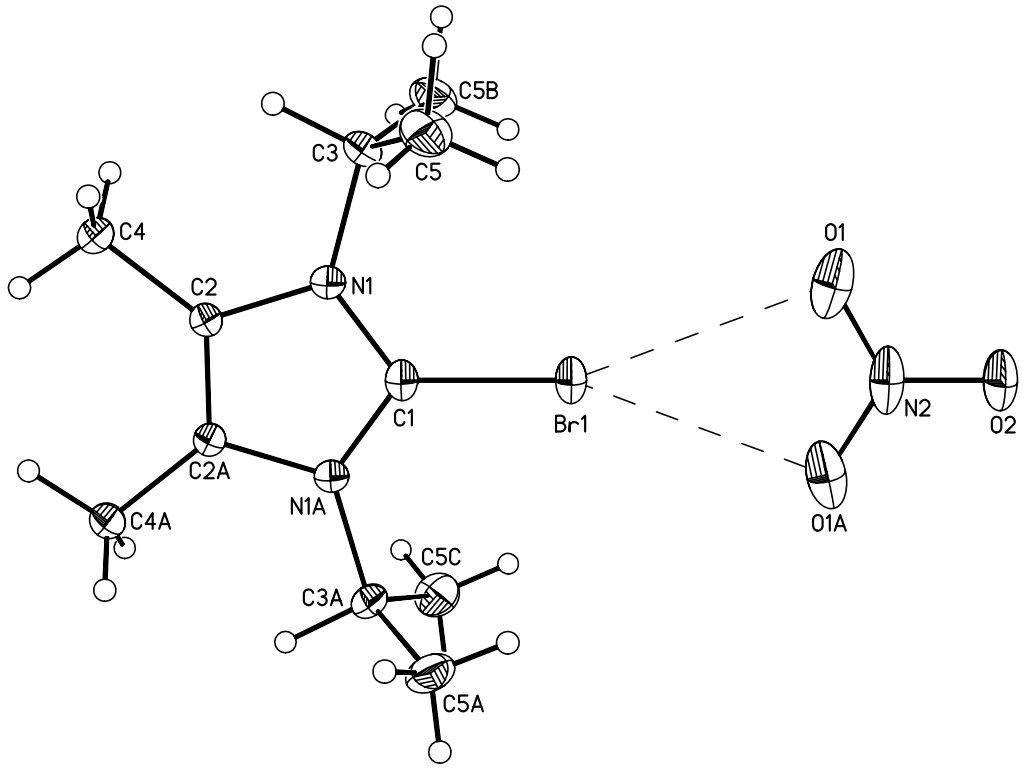


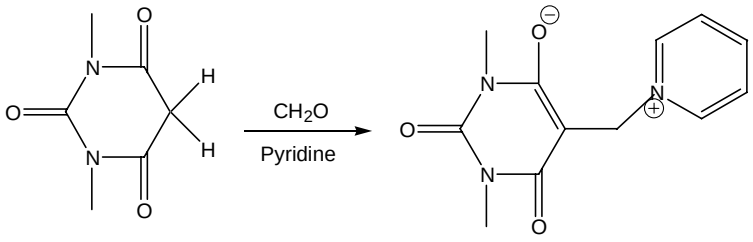
Fig. 17: The crystal structure of **49**

3.2 1,3-Dimethylbarbituric acid

3.2.1 Derivatives of methylene 1,3-dimethylbarbituric acid

3.2.1.1 Synthesis of 1,3-dimethyl-2,6-dioxo-5-(1-pyridiniumylmethyl)-1,2,3,6-tetrahydro-4-pyrimidinolate (**50**)

The condensation of 1,3-dimethylbarbituric acid **13** with aqueous formaldehyde in the presence of pyridine gives **50** in good yield.

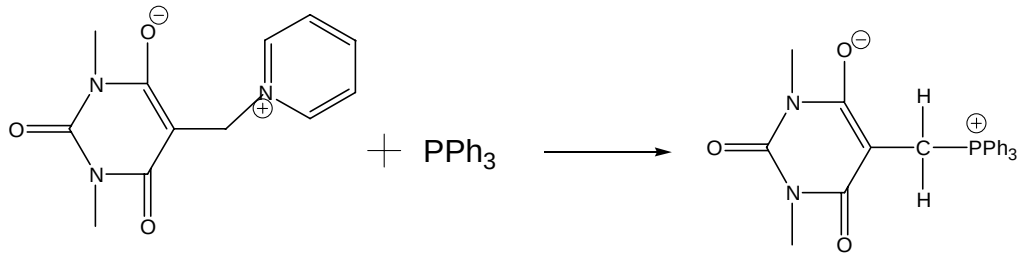


13 **50**

The zwitterionic pyridine adduct should open new synthetic possibilities by the exchange of the pyridine fragment against other bases. The ^{1}H and ^{13}C-NMR spectra show the chemical shifts for the central methylene group [^{1}H 5.24, ^{13}C 51.8] which lie in the same range of the chemical shift of the methylene group in meldrum's pyridine adduct [54].

3.2.1.2 Synthesis of 1,3-dimethyl-2,6-dioxo-5-[(triphenylphosphonio)methyl]-1,2,3,6-tetrahydro-4-pyrimidinolate (**51**)

The reaction of **50** with PPh_3 gives **51** as stable crystalline solid in good yield.



50 **51**

The molecular structure of **51** was confirmed by X-ray crystallography (tab. 52-54, fig. 18) [55]. **51** crystallizes in the monoclinic space group P2(1)/n.

The crystal structure analysis of **51** indicates its structure as to be zwitterionic. The bond lengths of the barbiturate part [C(1)-C(2) 1.394(3) Å, C(1)-C(4) 1.403(3) Å, C(2)-O(3) 1.242(3) Å, C(4)-O(1) 1.242(3) Å] lie in the range corresponding to the barbiturate part in the structure of **52**. The expected phosphonium character of the zwitterionic nature appears in the dimension of the Ph_3PCH_2-fragments P(1)-C(7) [1.792(2) A°], P(1)-C(13) [1.800(2) Å], P(1)-C(19) [1.793(2) Å], P(1)-C(25) [1.852(2) Å]; C(7)-P(1)-C(13) [111.79(11)°], C(7)-P(1)-C(19) [108.96(11)°, C(7)-P(1)-C(25) [108.75(11)°], C(13)-P(1)-C(19) [108.25(11)°], C(13)-P(1)-C(25) [108.66(11)°] and C(19)-P(1)-C(25) [110.43(11)°]. The structure of the central methyl fragments is close to that in structure **52** [C(1)-C(25) 1.492(3) Å; C(1)-C(25)-P(1) 112.99(17)°]. This result was also observed in the methylene meldrum's derivatives [56].

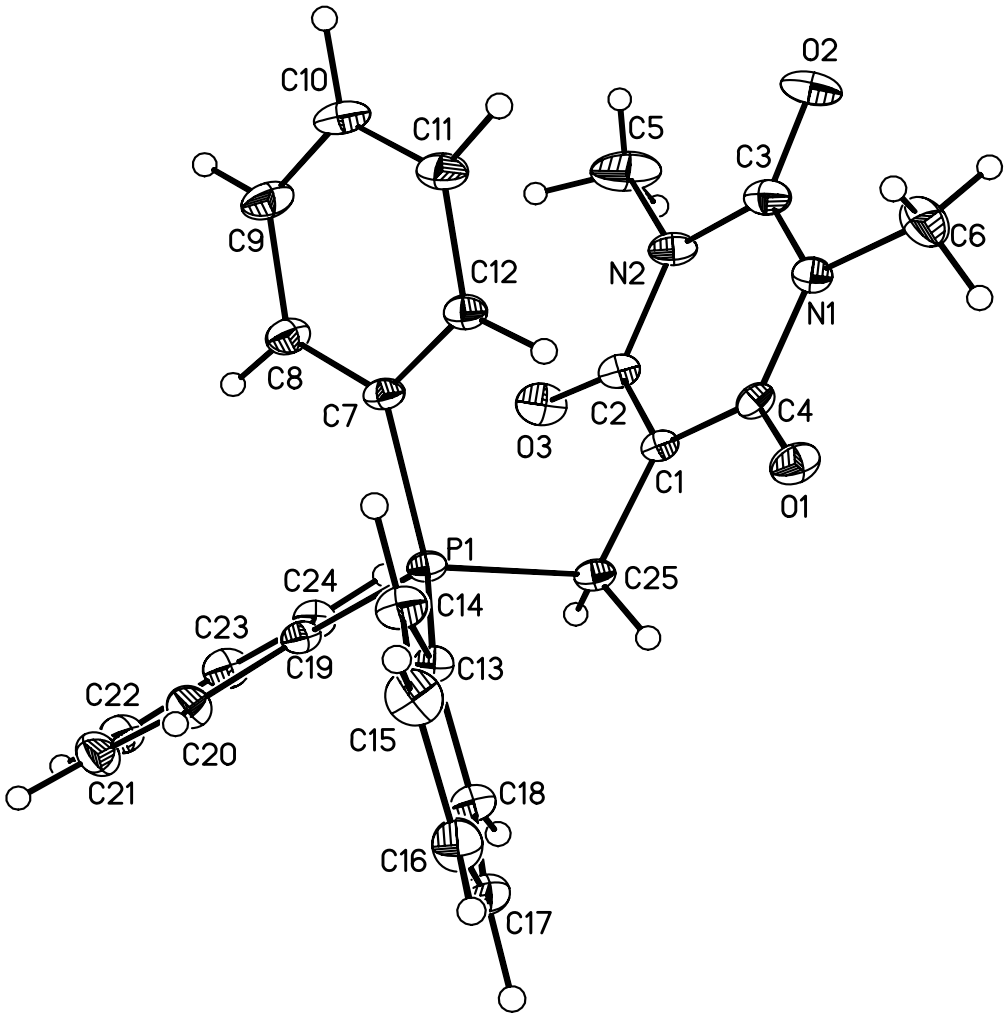
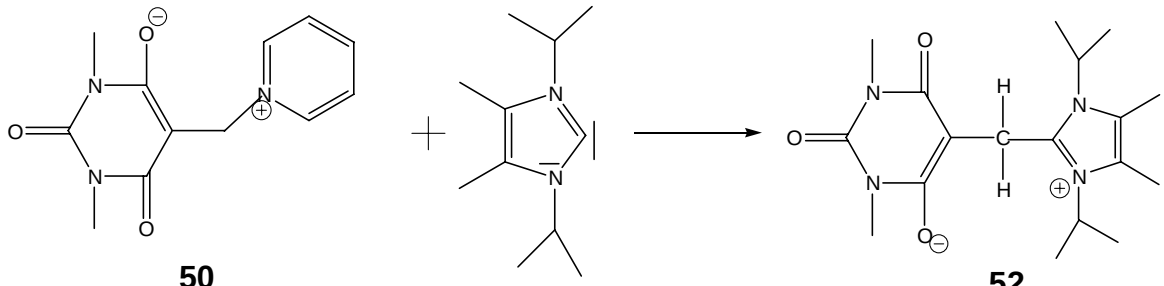


Fig. 18: The crystal structure of **51**

3.2.1.3 Synthesis of 5-[(1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium-2-yl)methyl] 1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate (**52**)

The reaction of **50** with carbene **4c** gives **52** as stable crystalline solid in good yield.



The molecular structure of **52** was confirmed by X-ray crystallography (tab. 55-57, fig. 19) [55]. **52** crystallizes in the monoclinic space group p2(1)/n.

The structure characteristics of the heterocyclic rings are observed in both of the imidazolium fragments and the barbiturate rings, due to the delocalization of the π–electrons, this allows to a geometric comparison with the barbituric acid [57] and its anion [58]. The structure of the imidazolium part is compatible to its structure in the numerous imidazolium derivatives [4]. The heterocyclic rings bonded to the methylene group show the expansion of C(1)-C(18)-C(7) [113.53 (12)°], due to steric requirements. A small difference was observed in the C-C bond lengths C(1)-C(18) [1.509(2) Å] and C(18)-C(7) [1.494(2) Å] due to the different S-proportions.

As expected, the presence of the water molecule in the crystal shows hydrogen bonds with the neighbouring zwitterionic part [O(4)-H(4A) 0.959(3) Å, H(4A)-O(2) 1.903(4) Å, O(4)-H(4B) 0.874(3) Å, H(4B)-O(1) 2.009(4) Å; O(4)-H(4A)-O(2) 172.4(1)°, O(4)-H(4B)-O(1) 170.0(1)°].

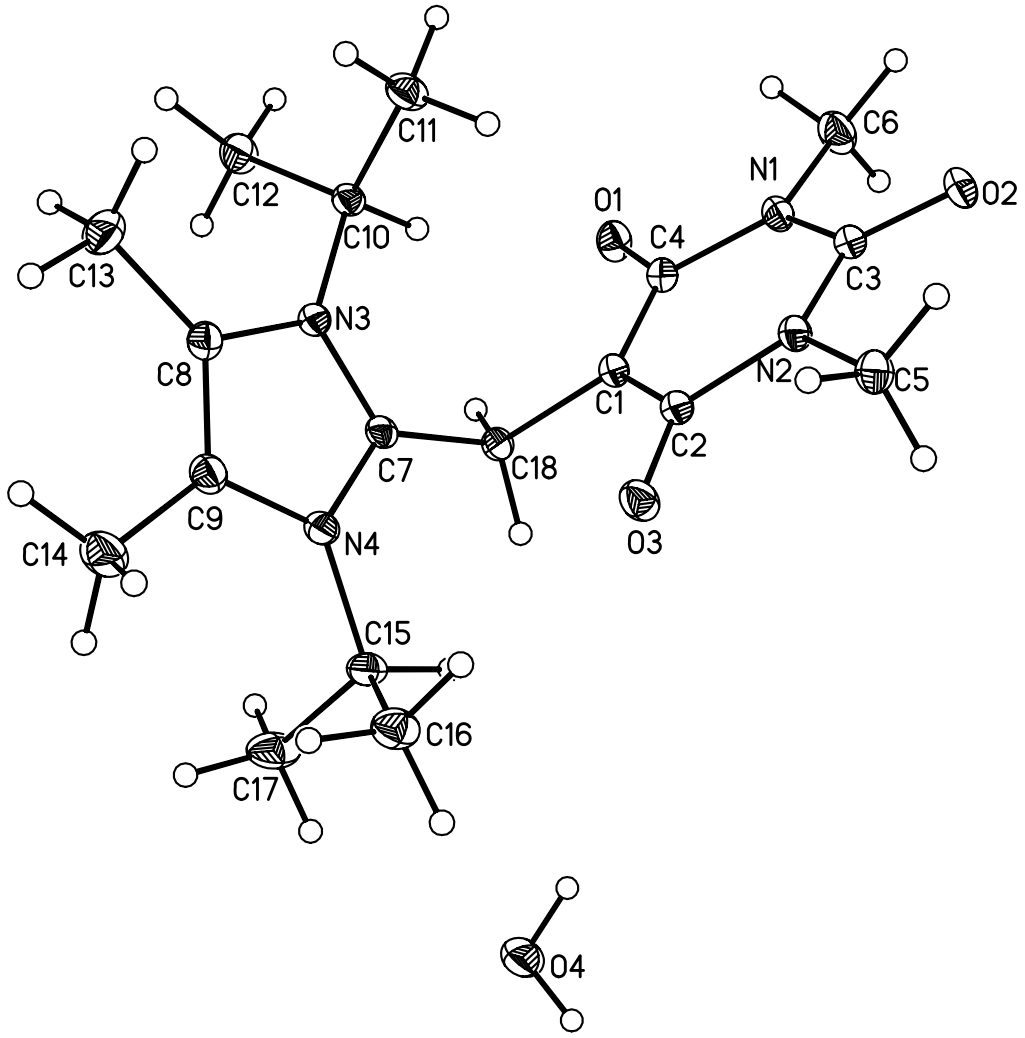
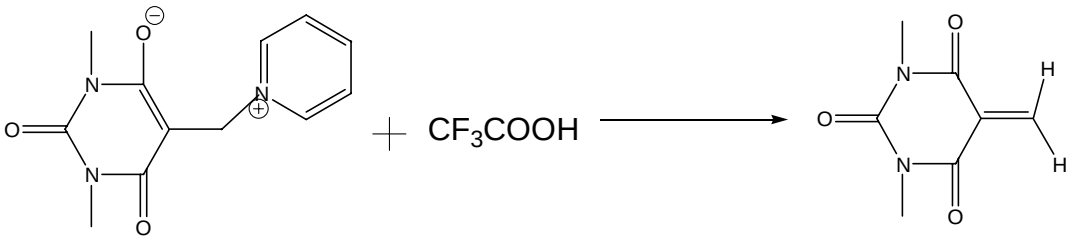


Fig. 19: The crystal structure of **52**

3.2.1.4 Synthesis of 1,3-dimethyl-5-methylene-2,4,6(1H,3H,5H)-pyrimidinetrione (**53**)

The reaction of **50** with CF_3COOH gives **53** as stable crystalline solid.



50 **53**

The molecular structure of **53** was confirmed by X-ray crystallography (tab. 58-60, fig. 20) [59]. **53** crystallizes in the monoclinic space group P2(1)/C. The bond lengths H(7BA)-O(3) [2.873 Å], C(7A)-H(7BA) [0.960 Å] and C(7A)-H(7BA)….O(3) [145.5°] lie within the *Van der Waals* contact range. The bond length C(3A)-C(6A) [1.441(9) Å] lies within the intermediate of the carbon-carbon single and double bonds lengths. The unexpected result was the absence of hydrogen bonds between any of the hydrogen atoms at the exocyclic carbon atom with any of the oxygen atoms. Surprisingly, the ^{1}H-NMR spectrum shows a chemical shift of 2.11 ppm for the two exocyclic hydrogen atoms in contrast to the chemical shift of the hydrogen atoms in the methylene meldrum's derivatives of 7.18 ppm [54].

53 has high susceptibility to the nucleophilic attack at the carbon atom of the methylene group, the reaction of **53** with PPh_3 produced **51**. In the case of the meldrum's pyridine adduct, this compound was prepared only in situ without any isolation [54].

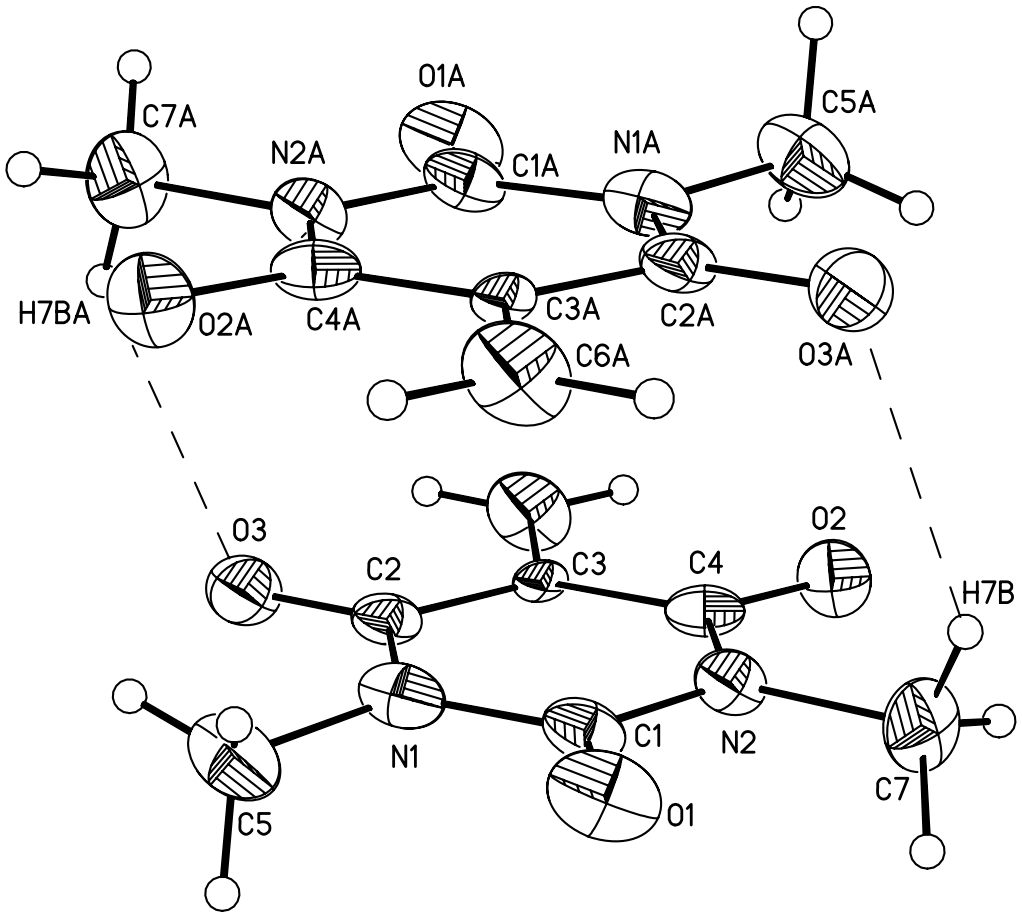
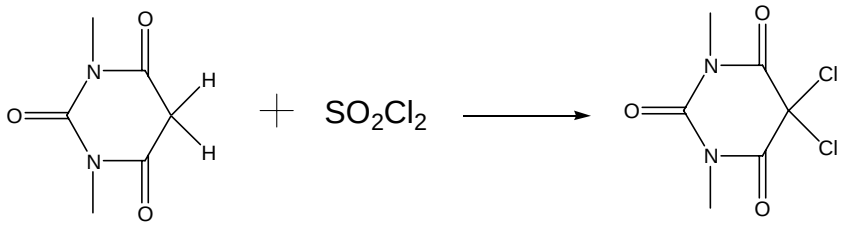


Fig. 20: The crystal structure of **53**

3.2.2 1,3-Dimethylbarbituric acid derivatives consisting of nucleophilic attack at 5-halo position

3.2.2.1 Synthesis of 5,5-dichloro-1,3-dimethyl-2,4,6(1H,3H,5H)-pyrimidinetrione (**54**)

The reaction of **13** with SO_2Cl_2 gives **54** as stable crystalline solid in good yield.



13 **54**

The structure of **54** was initially assigned in solution by ^{1}H and ^{13}C-NMR analysis. The ^{1}H spectrum shows a large chemical shift difference of C5 (C1 in the structure) of 72.2 ppm relative to its position in **13** of 39.8 ppm. The molecular structure of **54** was confirmed by X-ray crystallography (tab. 61-63, fig. 21) [60]. **54** crystallizes in the monoclinic space group $P2_1/c$.

The total summation of angles around C2 [O(1)-C(2)-C(1) 120.3°, C(1)-C(2)-N(1) 116.5°, N(1)-C(2)-O(1) 123.2°] gives 360°. The influence of the Cl-atoms in **54** is evident by the comparison to the barbituric acid. The C-C bond length in **54** is considerably longer than the C-C bond length in the barbituric acid [1.53 versus 1.48 Å] [61].

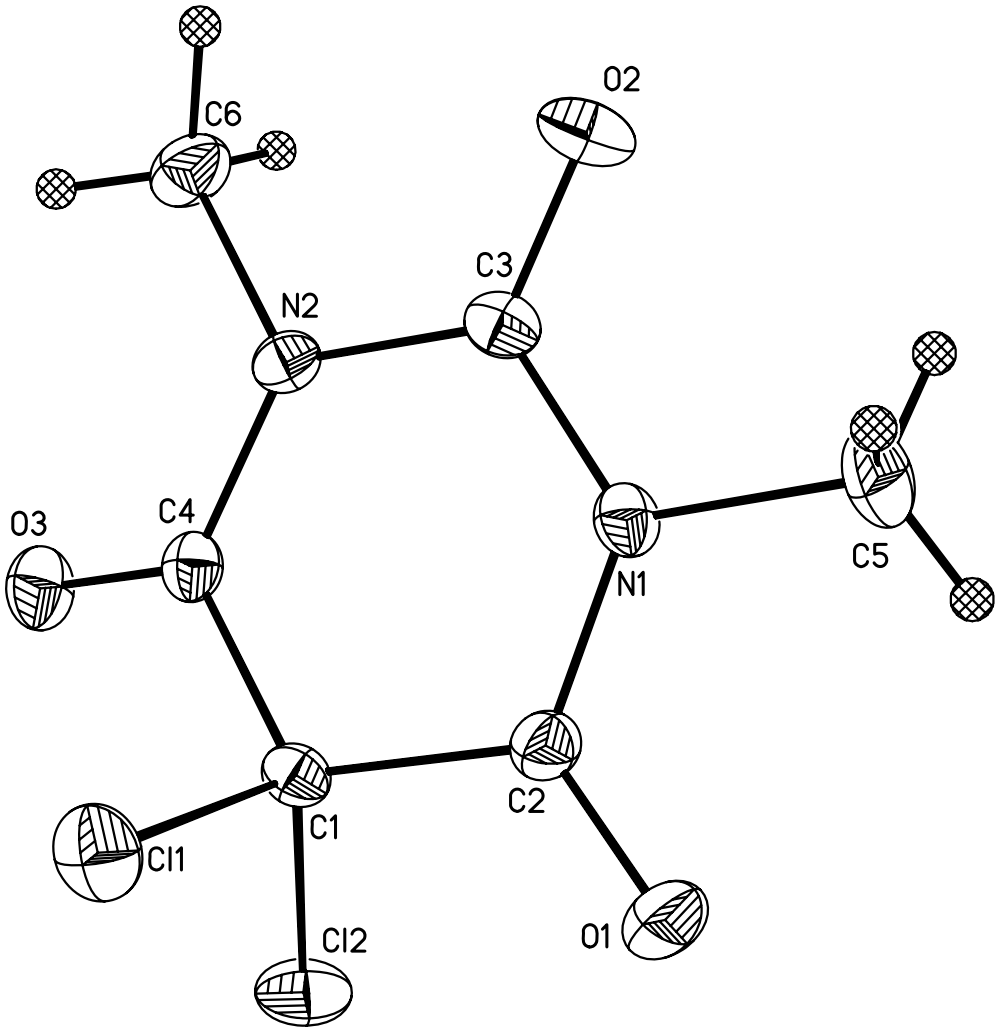
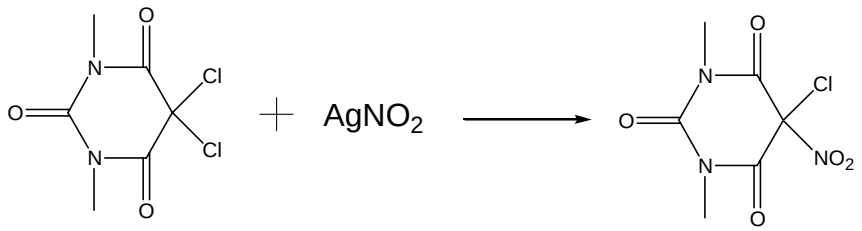


Fig. 21: The crystal structure of **54**

3.2.2.2 Synthesis of 5-chloro-1,3-dimethyl-5-nitro-2,4,6(1H,3H,5H)-pyrimidinetrione (**55**)

The reaction of **54** with $AgNO_2$ gives **55** as stable crystalline solid in good yield.



54 **55**

The ^{13}C-NMR spectrum shows a large chemical shift difference of C5 (C1 in the structure) of 89.2 ppm relative to its position in **54** of 72.2 ppm. The molecular structure of **55** was confirmed by X-ray crystallography (tab. 64-66, fig. 22) [62]. **55** crystallizes in the monoclinic space group Cc. The bond angles around C5 (C1 in the structure) lie in the range of 106.4-116.6°, which indicates the sp^3-hybridization of the carbon. The bond length C(1)-Cl(1) [1.752(3) Å] is close to the bond lengths C(1)-Cl(1) and C(1)-Cl(2) in **54** [1.784(2) Å and 1.748(2) Å], respectively. The bond length C(1)-N(3) [1.523(4) Å] is longer than the bond lengths C(5)-N(1) [1.82(4) Å] or C(6)-N(2) [1.473(5) Å]. This can be attributed due to the presence of the electron-withdrawing groups bonded to C5 in **55**.

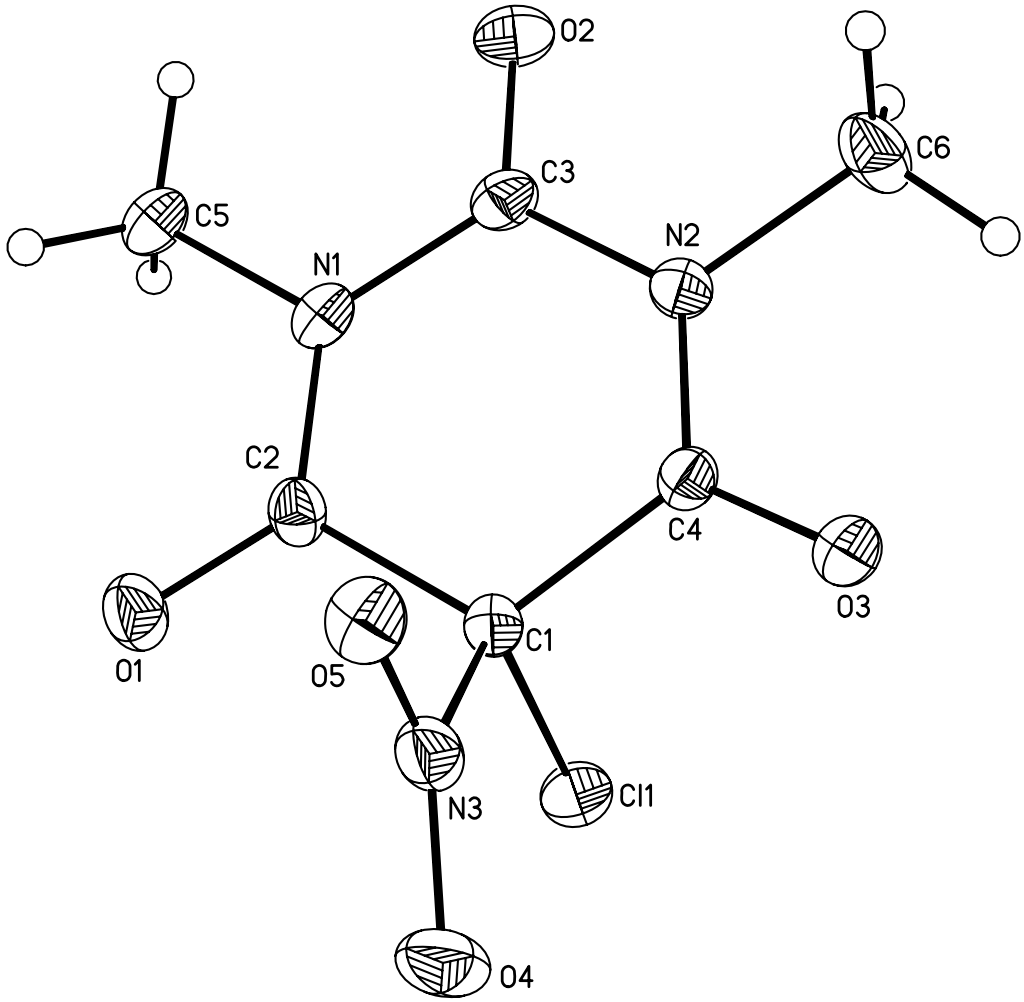
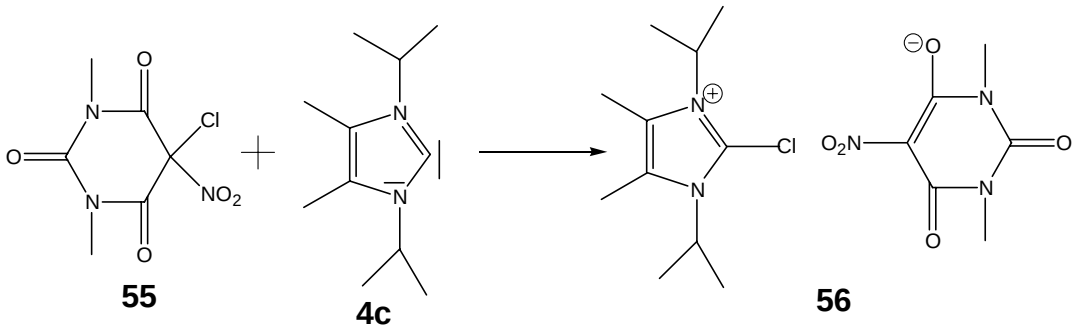


Fig. 22: The crystal structure of **55**

3.2.2.3 Synthesis of 2-chloro-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium 1,3-dimethyl-5-nitro-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate (**56**)

The reaction of **55** with carbene **4c** gives **56** as stable crystalline solid in good yield.



The spectroscopic data of the cation (ImCl) are close to that observed in other chloroimidazolium salts [48]. The ^{13}C-NMR spectrum of the anion shows a large chemical shift difference of C5 (C11 in the structure) of 114.2 ppm relative to its position in **55** of 89.2 ppm.

The molecular structure of **56** was confirmed by X-ray crystallography (tab. 67-69, fig. 23) [63]. **56** crystallizes in the monoclinic space group P2(1)/m. The contacts Cl(1)-O(4A) [3.803 Å] and Cl(1)-O(5) [2.880 Å] lie within the *Van der Waals* distance range, while there is no contact between Cl(1) and each of O(1A) or O(2A) or O(3A). The bond angle C(1)-Cl(1)-O(5A) [172.7°] reveals the presence of near linear Cl-C-O contacts.

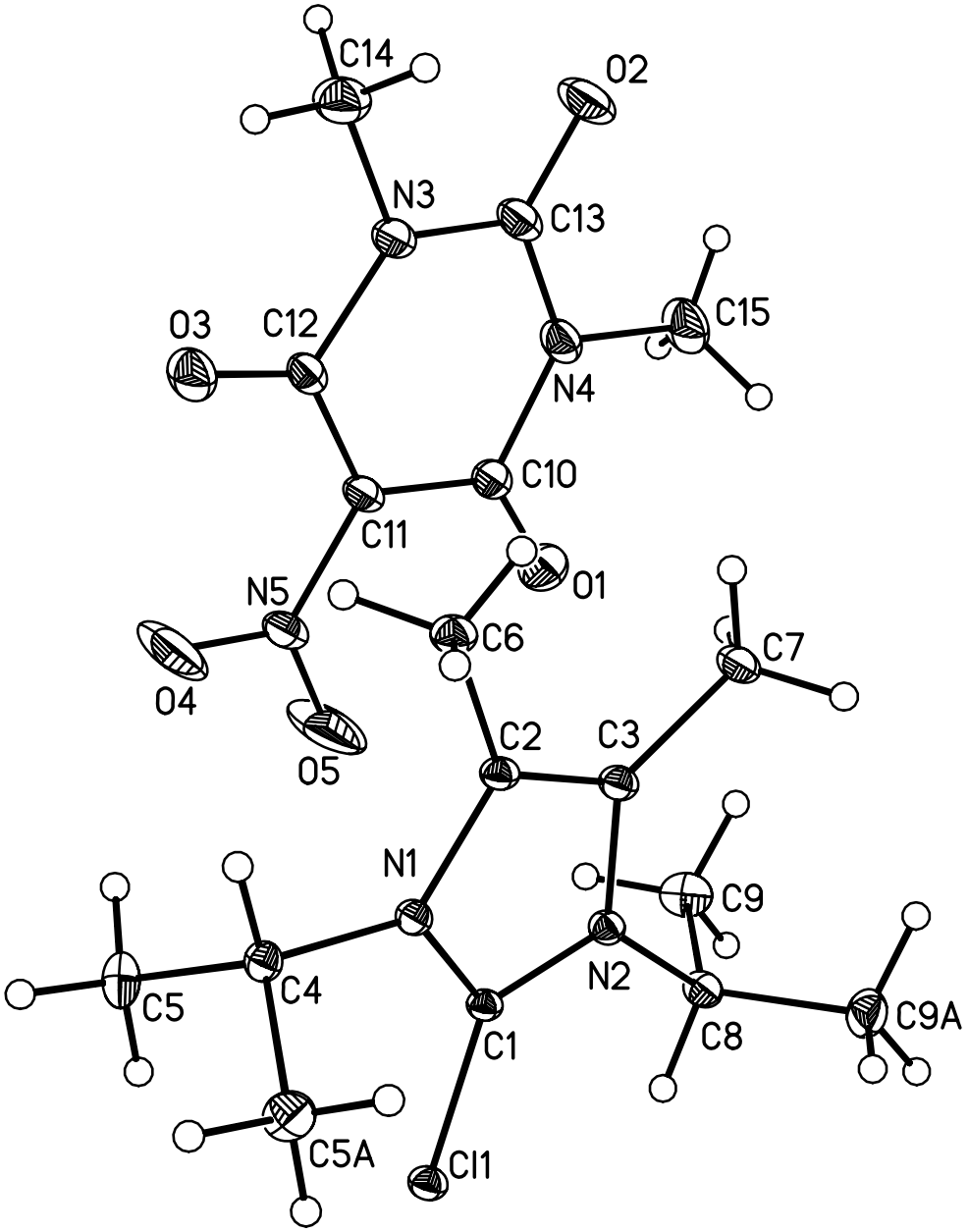
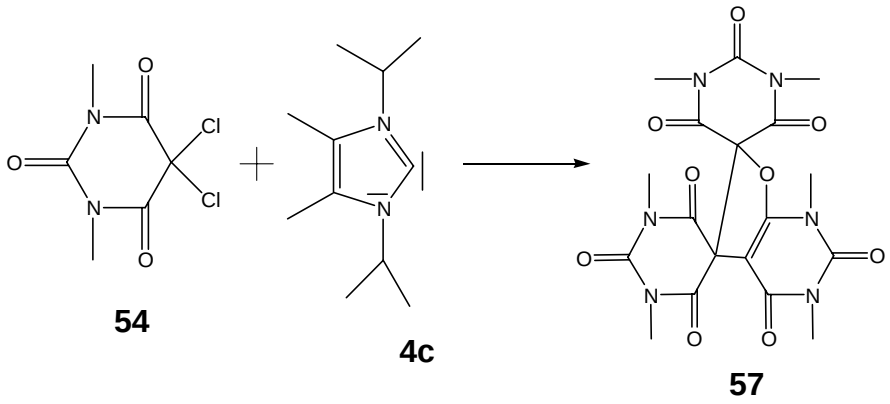


Fig. 23: The crystal structure of **56**

3.2.2.4 Synthesis of 5,6-dihydro-1,3-dimethyl-5,6-bis-[1',3'-dimethyl]-2',4',6'-trioxopyrimid (5',5') yl[2,3-d] uracil **(57)**

The reaction of **54** with carbene **4c** gives **57** as stable crystalline solid in good yield.



The molecular structure of **57** was confirmed by X-ray crystallography (tab. 70-72, fig. 24) [64]. **57** crystallizes in the orthorhombic space group Pbca. **57** is a trimeric form of 1,3-dimethylbarbituric acid with two spiro linkages and a central dihydrofuran fragment. The bond distance between the C-atoms of the two spiro-linked barbiturate molecules C(1)-C(11) is quite long [1.613 Å]. The five membered ring is in an envelope conformation [64].

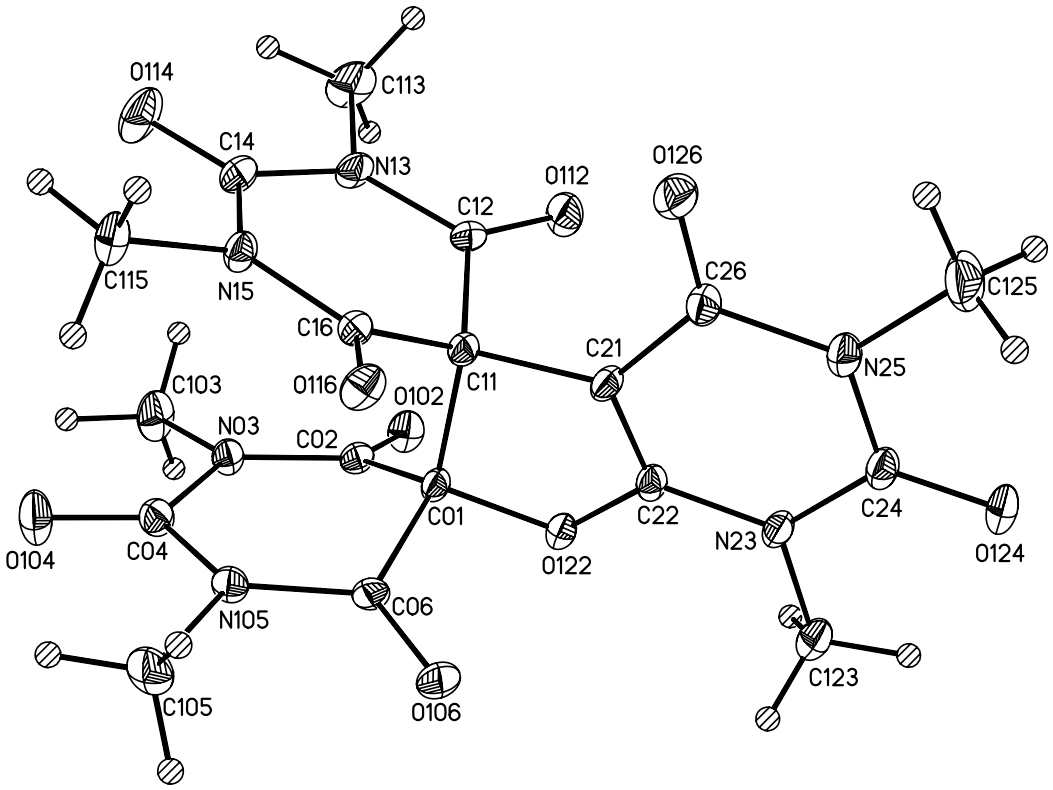
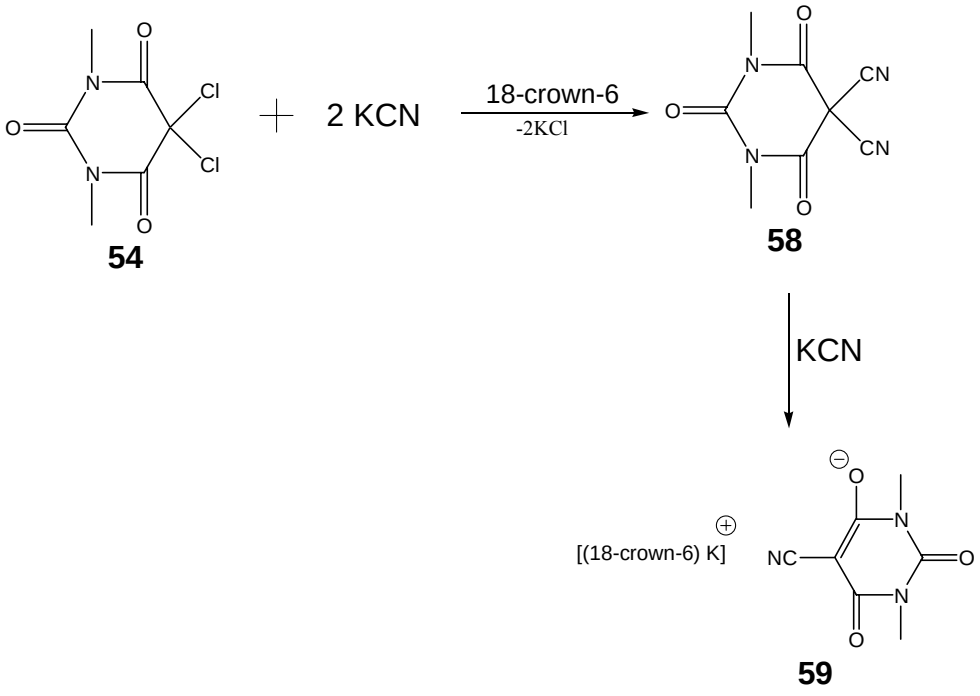


Fig. 24: The crystal structure of **57**

3.2.2.5 Synthesis of potassium 5-cyano-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate 1,4,7,10,13,16-hexaoxacyclooctadecane (**59**)

The reaction of **54** with KCN in presence of 18-crown-6 gives **59** as stable crystalline solid.



54 **58** **59**

The molecular structure of **59** was confirmed by X-ray crystallography (tab. 73-75, fig. 25) [65]. **59** crystallizes in the monoclinic space group P2(1). The ^{13}C-NMR spectrum shows a large chemical shift difference of C5 of 41.4 ppm relative to its position in **54** of 72.2 ppm. The C-C bond lengths C(14)-C(15) [1.415(5) Å] and C(15)-C(16) [1.417(5) Å] lie within the intermediate of the carbon-carbon single and double bonds lengths. The bond lengths C(7)-K(1) [2.687(3) Å], C(13)-O(7) [1.229(5) Å] lie within the range of normal covalent distances.

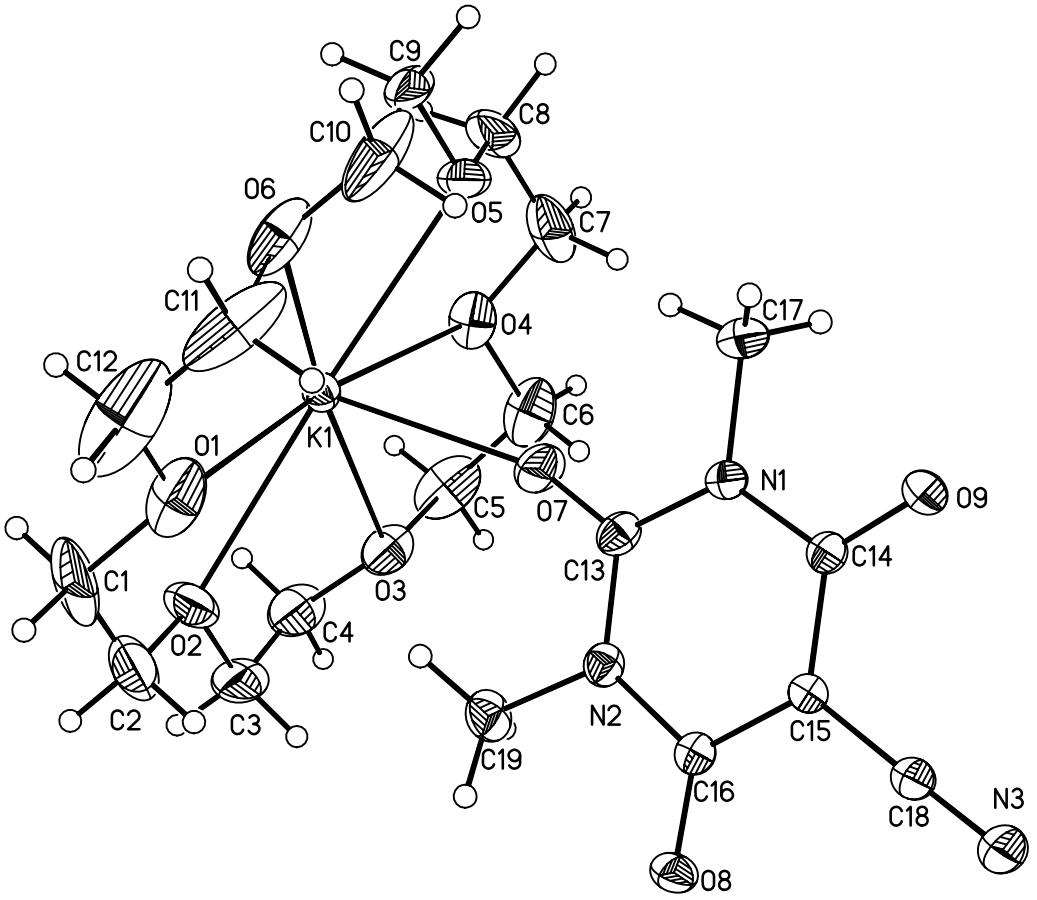
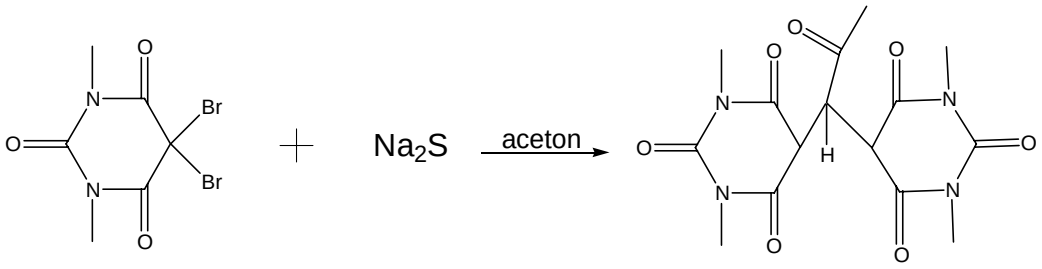


Fig. 25: The crystal structure of **59**

3.2.2.6 Synthesis of 5-[1-(1,3-dimethyl-2,4,6-trioxohexahydro-5-pyrimidinyl)-2-oxopropyl]-1,3-dimethyl-2,4,6(1H,3H,5H)-pyrimidinetrione (**60**)

The reaction of 5,5-dibromo-1,3-dimethyl-2,4,6-pyrimidinetrione [66] with Na_2S in acetone gives **60** as stable crystalline solid in low yield.



60

The molecular structure of **60** was confirmed by X-ray crystallography (tab. 76-78, fig. 26) [67]. **60** crystallizes in the orthorhombic space group P2(1)2(1)2(1). The structure of **60** was assigned in solution by ^{1}H and ^{13}C-NMR analysis. The ^{1}H-NMR spectrum shows a chemical shift for hydrogen atoms in the acetyl group of 1.62 ppm and the ^{13}C-NMR spectrum shows a chemical shift for the carbon atom in the carbonyl group of 200.1 ppm. The bond lengths C(2)-C(13) [1.537(4) Å] and C(13)-C(8) [1.543(4) Å] lie within the normal range of the C-C single bond length.

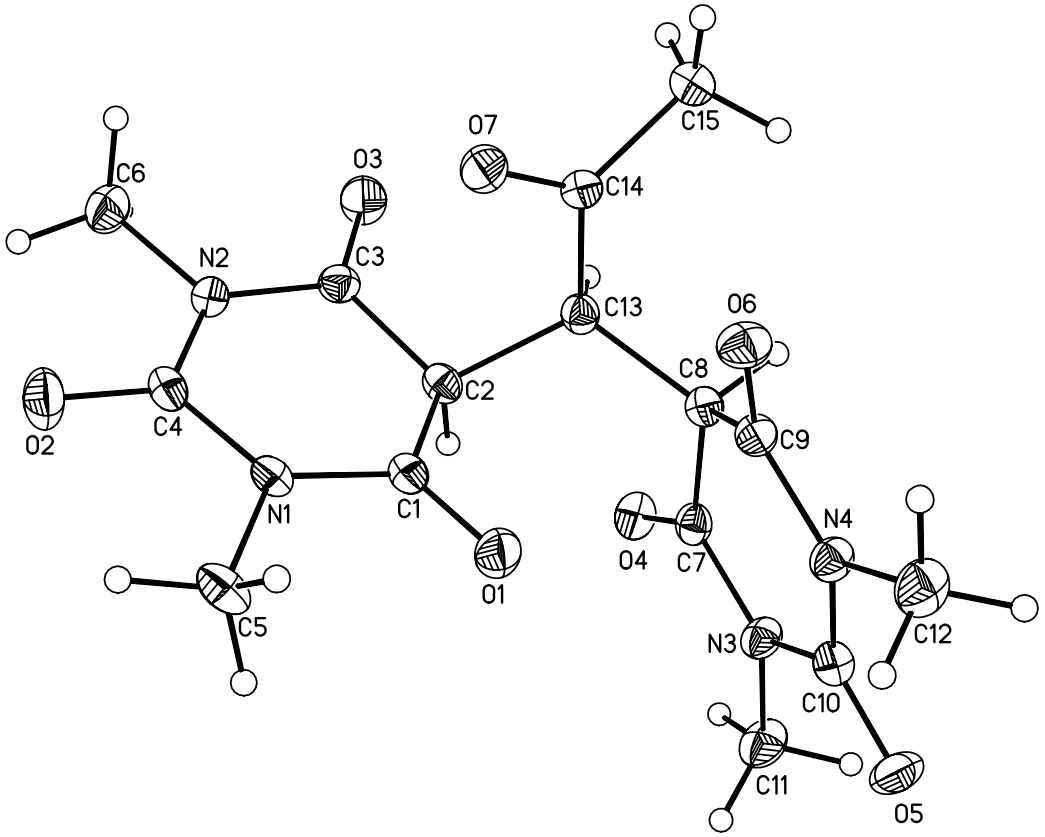
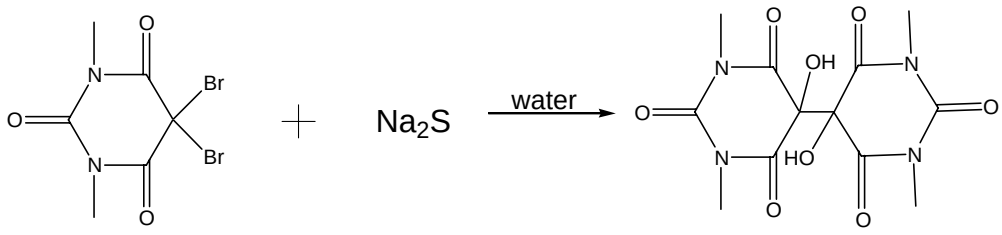


Fig. 26: The crystal structure of **60**

3.2.2.7 Synthesis of 5,5'-dihydroxy-1,1',3,3'-tetramethyl [5,5'-Bipyrimidine] 2,2',4,4',6,6'(1H,1'H,3H,3'H,5H,5'H)-hexone (**61**)

The reaction of 5,5-dibromo-1,3-dimethyl-2,4,6-pyrimidinetrione [66] with Na_2S in water gives **61** as stable crystalline solid in low yield.



61

The molecular structure of **61** was confirmed by X-ray crystallography (tab. 79-81, fig. 27) [68]. **61** crystallizes in the orthorhombic space group P2(1)2(1)2(1). The central C-C single bond C(1)-C(7) [1.610(4) Å] is significantly elongated relative to carbon-carbon single bond length [1.54 Å]. The two heterocyclic ring fragments are near coplanar, the twist angle between the planes C(4)C(1)C(2) and C(10)C(7)C(8) being 6.3°. Interestingly, the presence of hydrogen bonds to the solvents molecules H(1)-O(9A) [1.860 Å]; O(1)-H(1)-O(9A) [162.0°] and H(5)-O(10A) [1.956 Å]; O(5)-H(5)-O(10A) [171.8°] could be observed.

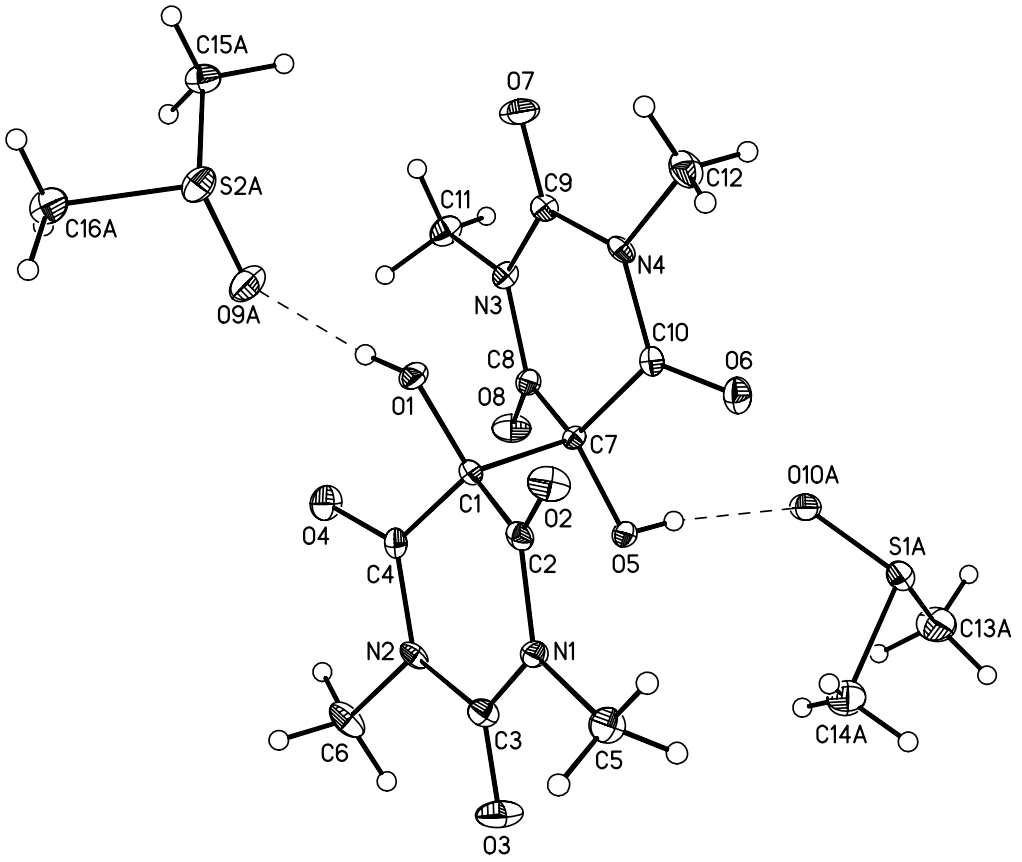
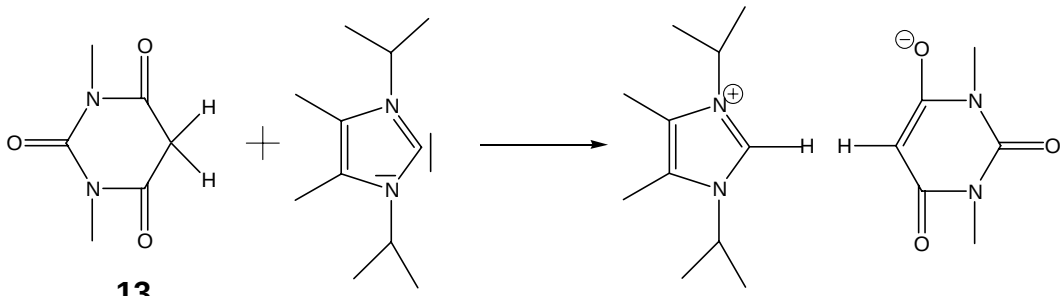


Fig. 27: The crystal structure of **61**

3.2.3 1,3-dimethylbarbituric acid salts

3.2.3.1 Synthesis of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium 1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate (**62**)

The reaction of **13** with carbene **4c** gives **62** as stable crystalline solid in good yield.



13 **4c** **62**

The molecular structure of **62** was confirmed by X-ray crystallography (tab. 82-84, fig. 28) [58]. **62** crystallizes in the monoclinic space group P2(1)/c. The ^{13}C-NMR spectrum shows a large chemical shift difference of C5 (C2 in the structure) of 76.8 ppm relative to its position in **13** of 39.8 ppm. The anion is connected wit the cation by one of the exocyclic oxygen atoms.

The hydrogen bonds trained in **62** [C(7)-H(7A) 0.955 Å, O(1)-H(7A) 2.048 Å, O(1)-C(1) 1.247(2) Å; C(7)-H(7A)….O(1) 163.6°] lie within the standard range of the C-H….O bridges, the presence of the weak C-H….O hydrogen contacts cause the construction of the polymer system in the crystal, the ring plains of the ion pair are twisted against each other by 74.5°.

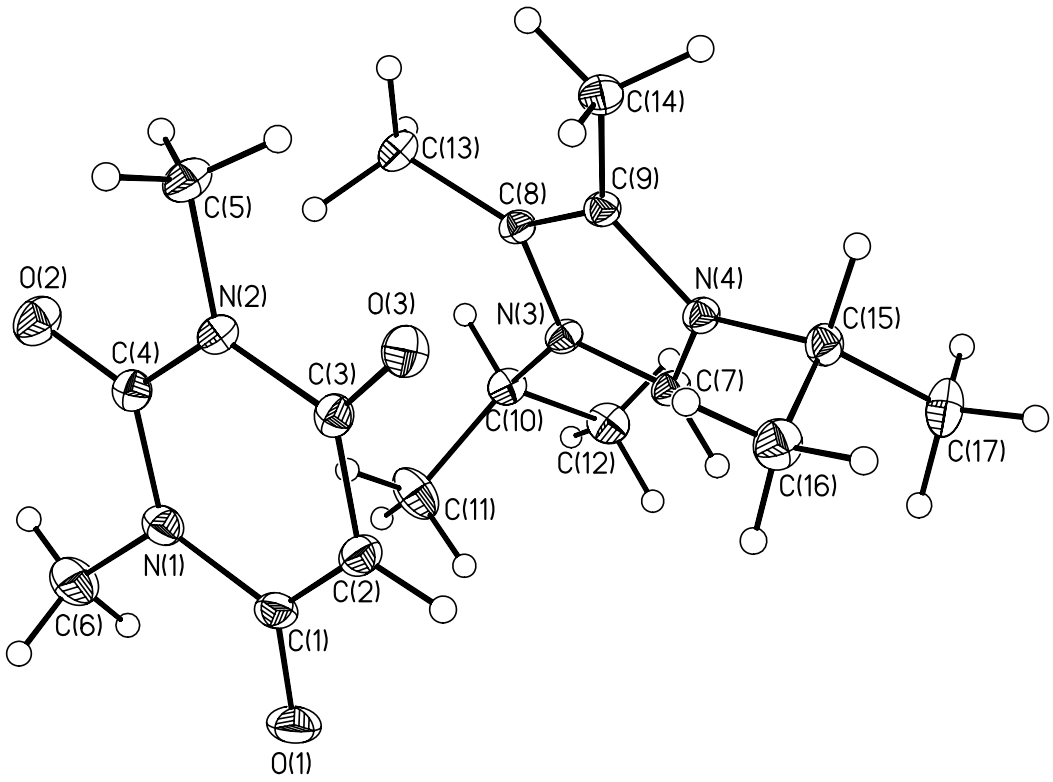
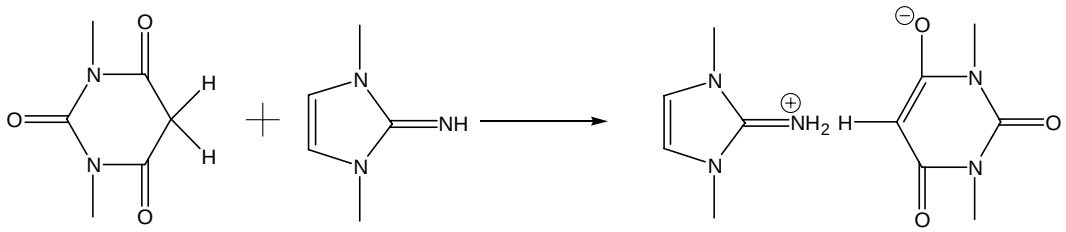


Fig. 28: The crystal structure of **62**

3.2.3.2 Synthesis of 1,3-dimethyl-1,3-dihydro-2H-imidazol-2-iminium 1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate (**64**)

The reaction of **13** with **63** [69] gives **64** as stable crystalline solid in good yield.



13 **63** **64**

The molecular structure of **64** was confirmed by X-ray crystallography (tab. 85-87, fig. 29) [58]. **64** crystallizes in the orthorhombic space group Pbca. The anion is connected with the cation by the exocyclic oxgen atoms. The strong interionic N-H-O hydrogen bonds between the two oxygen atoms (O4,O6) in the barbiturate anion and the two hydrogen atoms on the amino group in the cation give the dimeric ion-pair [N(5)-H(5A) 0.873 Å, H(5A)-O(3A) 2.006 Å, N(5)-H(5B) 0.980 Å, H(5B)-O(1) 1.978 Å, O(3A)-C(3A) 1.245(2) Å, O(1)-C(1) 1.247(2) Å; H(5A)-N(5)….H(5B) 117.8°, N(5)-H(5A)….O(3A) 170.1°].

The presence of the weak bonds between the oxygen atom (O2) in the barbiturate anion and the hydrogen atoms at (C4, C5) and the N-methyl in imidazolium cation causes the construction of the space network structure. In the dimeric unit, the distance between the cation layers is 3.77 Å.

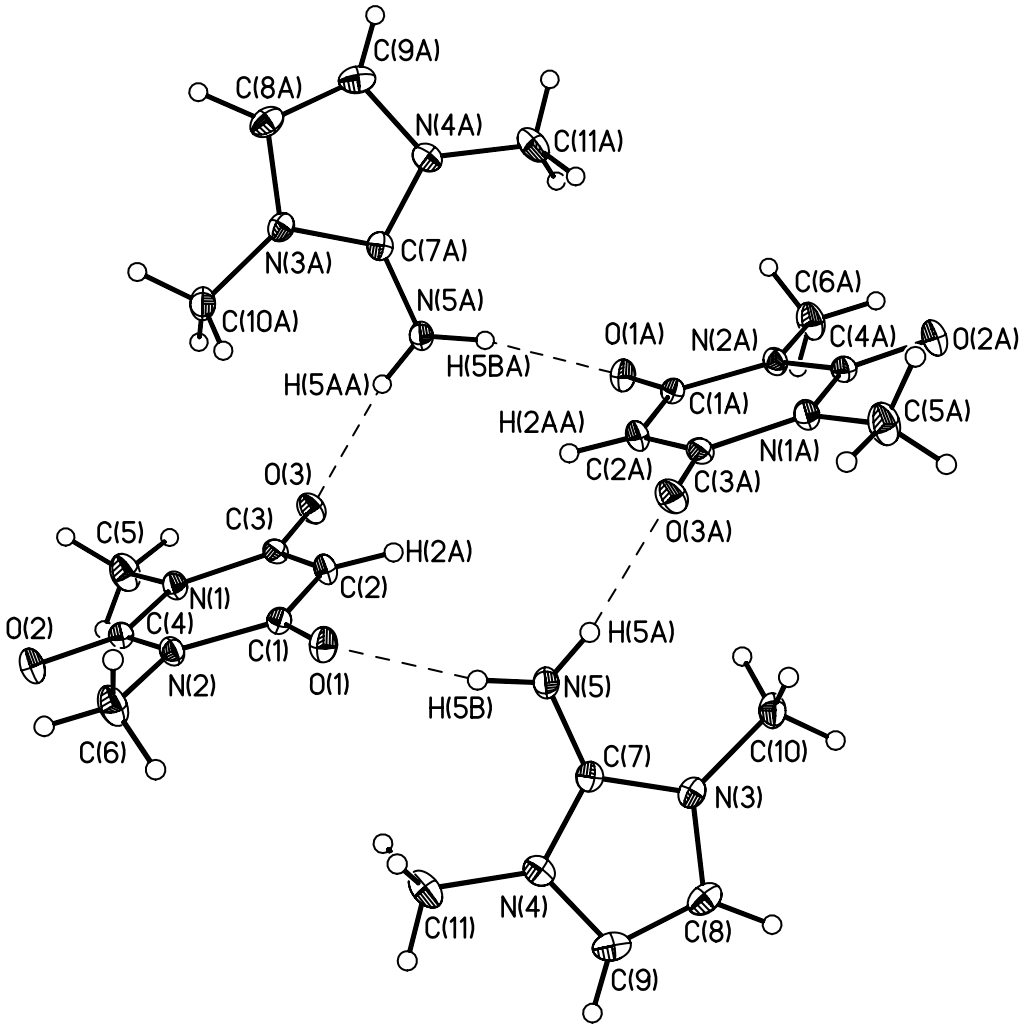
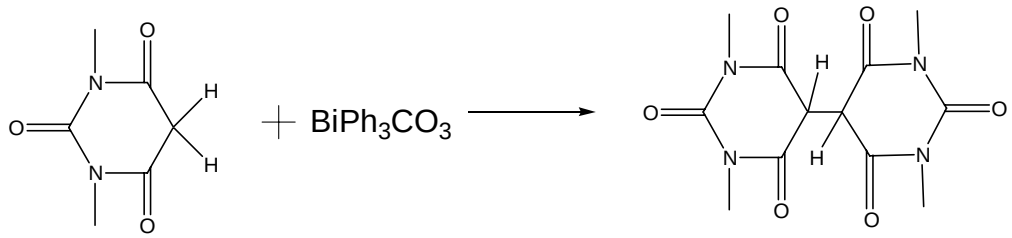


Fig. 29: The crystal structure of **64**

3.2.4 Synthesis of 1,3-dimethylbarbituric acid dimer

3.2.4.1 Synthesis of 1,1',3,3'-tetramethyl [5,5'-Bipyrimidine] 2,2',4,4',6,6' (1H,1'H,3H,3'H,5H,5'H)-hexone, (**65**)

The reaction of **13** with $BiPh_3CO_3$ [70] gives **65** as stable crystalline solid.



13 **65**

The molecular structure of **65** was confirmed by X-ray crystallography (tab. 88-90, fig. 30) [71]. **65** crystallizes in the tetragonal space group I4(1)/a. The ^{13}C-NMR spectrum shows a small chemical shift difference of C5 (C2 and C2A in the structure) of 46.9 ppm relative to its position in 1,3-dimethylbarbituric acid **13** of 39.8 ppm. The structure of **65** adopts the keto-form.

The total summation of angles around C1 or C3 [C(2)-C(3)-N(2) 115.99°, C(2)-C(3)-O(2) 121.90°, O(2)-C(3)-N(2) 121.91°] is near 360°. The center of the C(2)-C(2A) single bond lies on the crystallographic inversion center and the C-C single bond C(2)-C(2A) [1.522 Å] is very close to the expected C-C single bond value [1.54 Å].

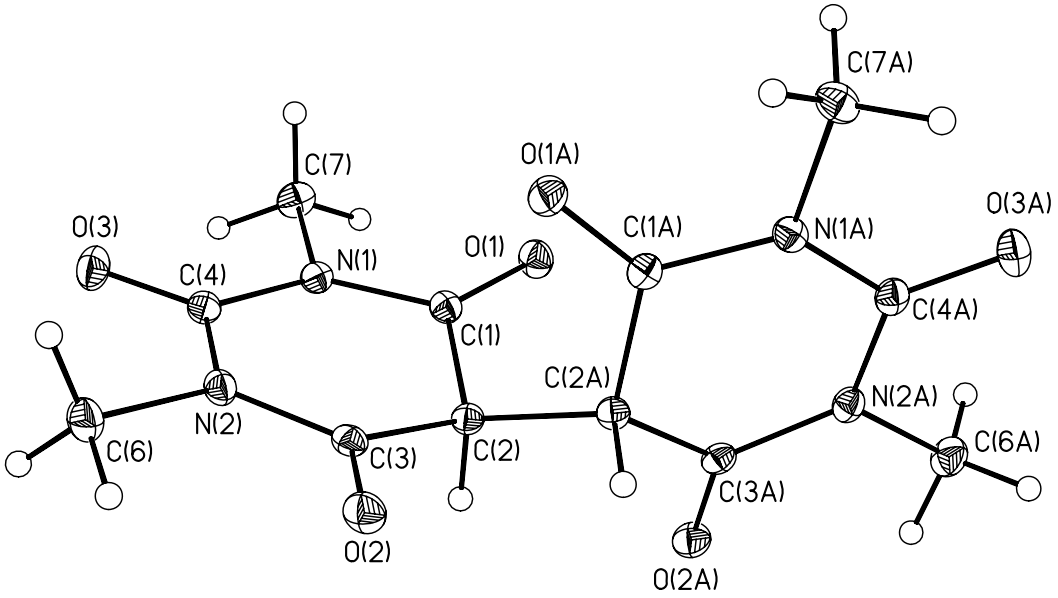
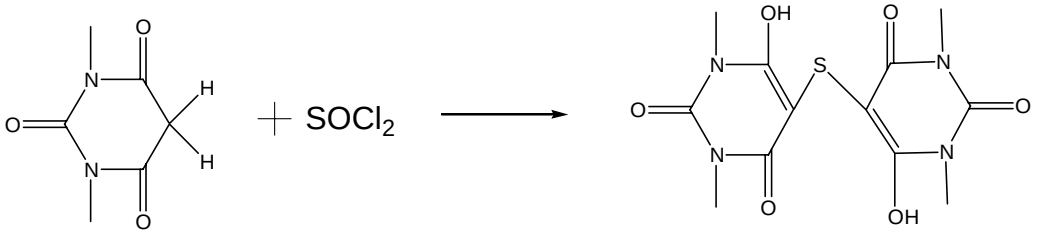


Fig. 30: The crystal structure of **65**

3.2.5 The 1,3-dimethylbarbituric acid thion and its derivatives

3.2.5.1 Synthesis of 2,6-hydroxy-5-[(6-hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydro-5-pyrimidinyl)sulfanyl]-1,3-dimethyl-2,4(1H,3H)-pyrimidinedione (**66**)

The reaction of **13** with $SOCl_2$ gives **66** as stable crystalline solid in good yield.



13 **66**

The molecular structure of **66** was confirmed by X-ray crystallography (tab. 91-93, fig. 31) [72]. **66** crystallizes in the triclinic space group P-1. The structure of **66** adopts the enol-form. The total summation of angles around C2 or C8 [C(3)-C(2)-C(1) 120.14°, S(1)-C(2)-C(1) 119.30°, S(1)-C(2)-C(3) 120.41°] is near 360°. The C-S bond lengths C(2)-S(1) [1.749(19) Å] and S(1)-C(8) [1.755(2) Å] lie in the normal range of the C-S single bond value. The bond angle

C(2)-S(1)-C(8) [101.71°] is larger than the C-S-C bond angle in the dimer of the thioxo meldrum's acid [73]. The hydrogen bonds trained in **66** [O(2)-H(2) 0.933 Å, H(2)-O(5) 1.793 Å; O(2)-H(2)….O(5) 162.1°, O(4)-H(4) 0.813 Å, H(4)-O(1) 1.913 Å, O(4)-H(4)….O(1) 167.2°] lie within the standard range of O-H….O bridges. The bond lengths C(2)-C(3) [1.370(3) Å] and C(7)-C(8) [1.372 Å] lie in the range of the carbon-carbon double bond length.

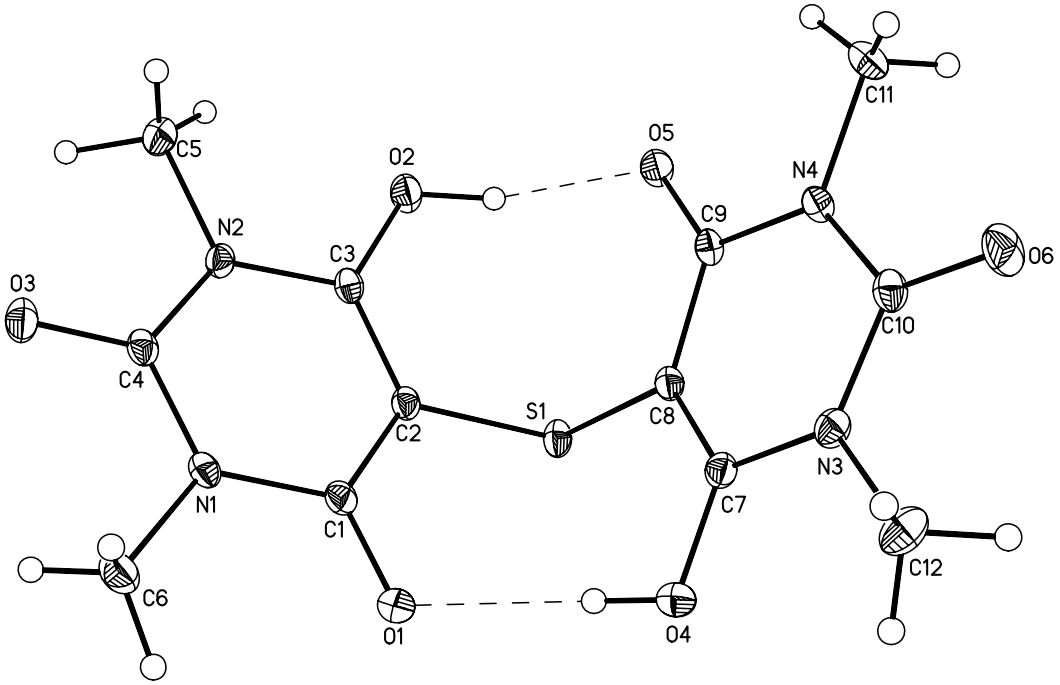
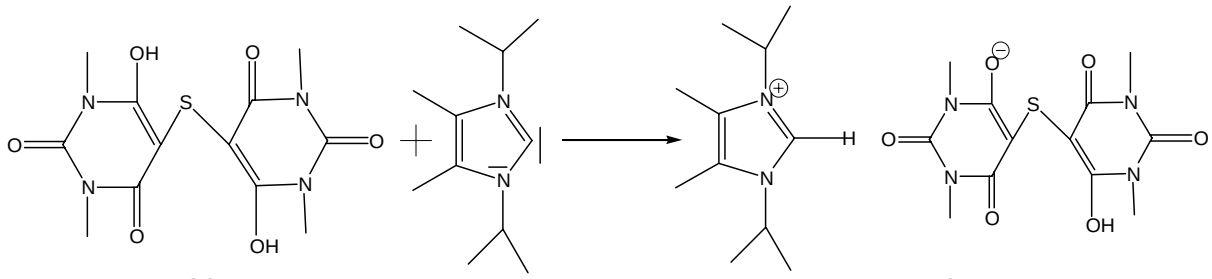


Fig. 31: The crystal structure of **66**

3.2.5.2 Synthesis of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium 5-[(6-hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydro-5-pyrimidinyl)sulfanyl]-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate (**67**)

The reaction of **66** with carbene **4c** in a molar ratio (1:1) gives **67** as stable crystalline solid in good yield.



66 **4c** **67**

The molecular structure of **67** was confirmed by X-ray crystallography (tab. 94-96, fig. 32) [74]. **67** crystallizes in the monoclinic space group P2(1)/c. The spectroscopic data of the cation ImH (exp. part) are close to that observed in the other salts of this cation [4].

The C-S bond lengths C(2)-S(1) [1.767(5) Å] and S(1)-C(8) [1.758(6) Å] lie in the same range of the C-S bond lengths of **66**. The cation is connected with the anion by hydrogen bonds through the H atom of the cation with the two exocyclic oxygen atoms at the anion. The hydrogen bonds trained in **67** [C(2)-O(1) 1.199(7) Å, C(9)-O(9) 1.259(6) Å, O(1)-H(14) 2.363 Å, O(6)-H(14) 2.387 Å, O(1)….H(14)….O(6) 100.0°] lie within the standard range of C-H….O bridges. An other bridge exists in the anion itself (O-H….O) which is stronger than the C-H….O bridge between the cation and the anion [O(4)-H(4) 1.197 Å, O(3)-H(4) 1.289 Å, C(1)-O(3) 1.300 Å, C(7)-O(4) 1.262 Å; O(4)-H(4)….O(3) 160.3°].

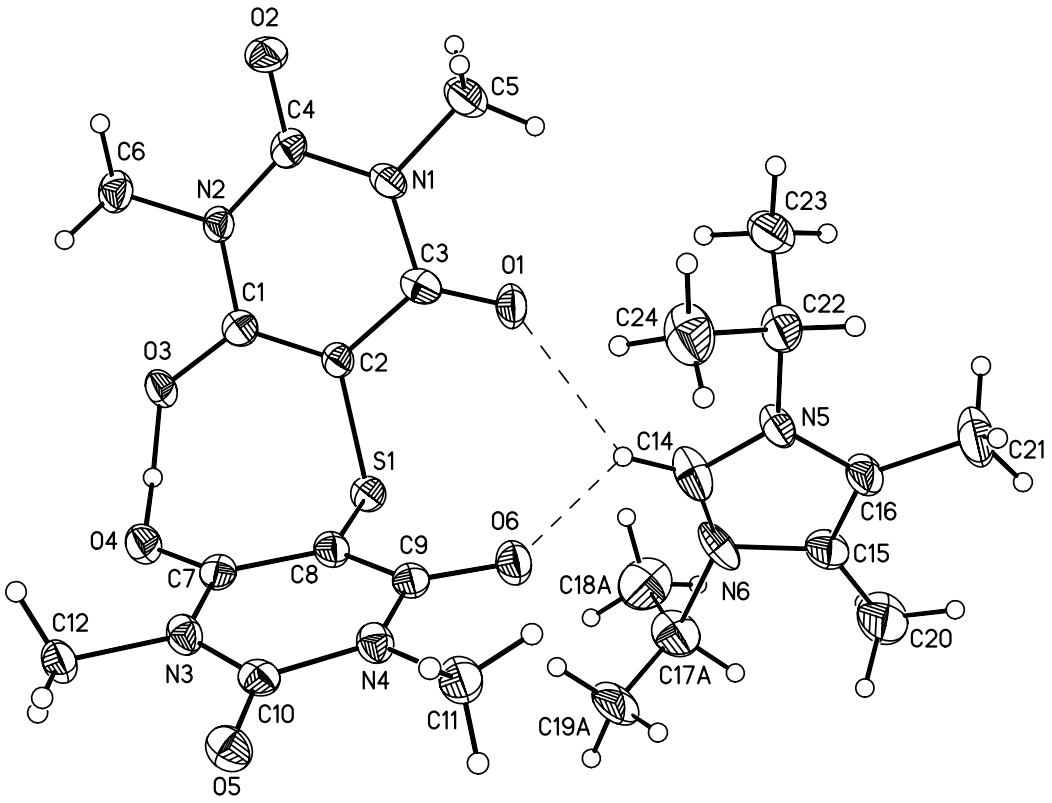
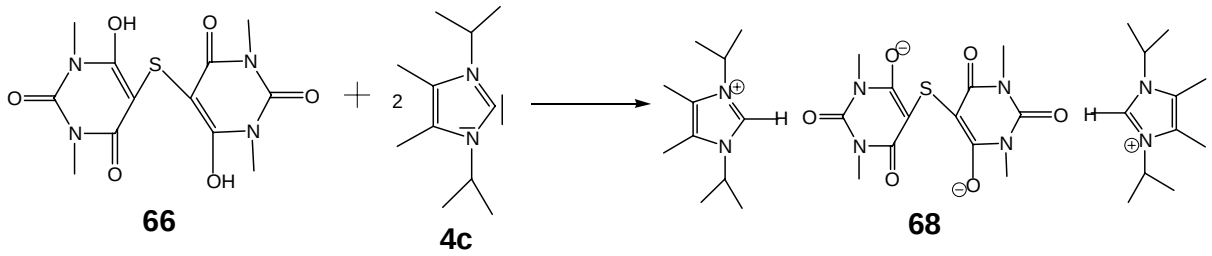


Fig. 32: The crystal structure of **67**

3.2.5.3 Synthesis of di(1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium) 5-[(1,3-dimethyl-6-oxido-2,4-dioxo-1,2,3,4-tetrahydro-5-pyrimidinyl)sulfanyl]-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate (**68**)

The reaction of **66** with carbene **4c** in a molar ratio (1:2) gives **68** as stable crystalline solid in good yield.



66 **4c** **68**

The molecular structure of **68** was confirmed by X-ray crystallography (tab. 97-99, fig. 33) [75]. **68** crystallizes in the monoclinic space group P2(1)/n. The spectroscopic data of the cation ImH (exp. Part) are close to that observed in the other salts of this cation [4].

The C-S bond lengths C(23)-S(1) [1.763(19 Å] and S(1)-C(29) [1.759(19) Å] lie in the same range of the C-S bond lengths of **66** and **67**. The anion is connected with the cations by the exocyclic oxygen atoms. The hydrogen bonds trained in **68** [H(1)-O(4) 2.220 Å; C(1)-H(1a)….O(4) 163.2°; H(12)-O(3) 2.213 Å, C(12)-H(12a)….O(3) 150.3°] lie within the standard range of C-H….O bridges. The bond lengths C(23)-C(26) [1.398(3) Å], C(23)-C(24) [1.409(3) Å], C(29)-C(30) [1.398(3) Å] and C(29)-C(32) [1.405(3) Å] lie within the intermediate of the carbon-carbon single and double bonds lengths, which indicates the enolate nature of the barbiturate anion.

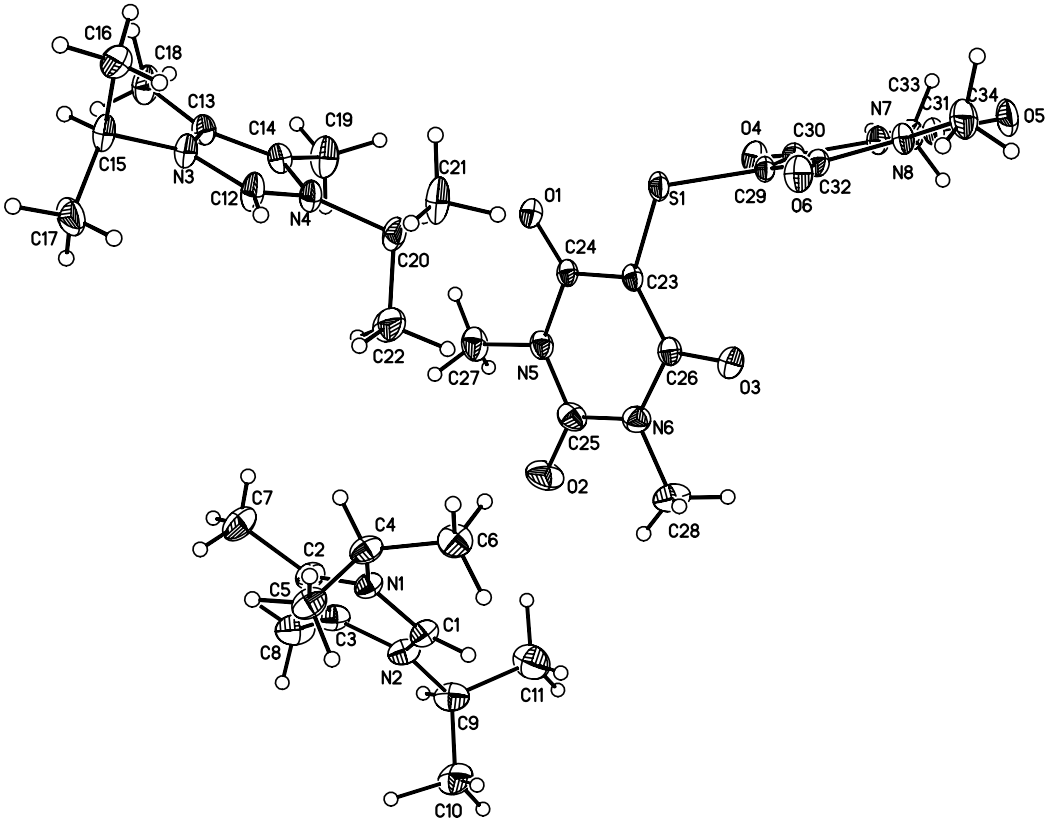
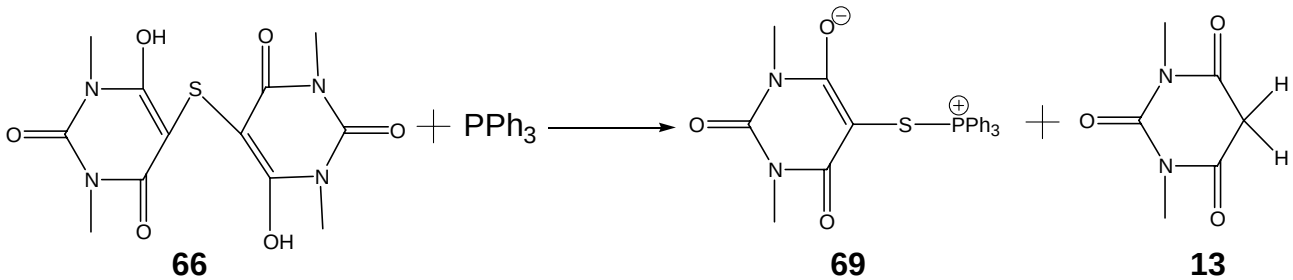


Fig. 33: The crystal structure of **68**

3.2.5.4 Synthesis of 1,3-dimethyl-2,6-dioxo-5-[(triphenylphosphonio)sulfanyl]-1,2,3,6-tetrahydro-4-pyrimidinolate (**69**)

The reaction of **66** with PPh_3 gives the zwitterionic compound **69** as stable crystalline solid in good yield.



66 **69** **13**

The molecular structure of **69** was confirmed by X-ray crystallography (tab. 99-102, fig. 34) [76]. **69** crystallizes in the orthorhombic space group P2(1)2(1)2(1). The ^{13}C-NMR spectrum shows a large chemical shift difference of C5 (C1 in the structure) of 60.8 ppm relative to its position in **66** of 81.7 ppm. In the ^{31}P-NMR spectrum, the chemical shift of 46.7 ppm is in the range of the phosphonium salts as expected. The central C(1)-S(1)-P(1) angle is 100.8°, which is close to that of the phosphonium zwitteronic compound of the meldrum's acid [73]. The C-S and S-P bond lengths are in the range of the normal bond lengths with a very small shortage [C(1)-S(1) 1.740 (2) Å, S(1)-P(1) 2.095 (9) Å]. The bond lengths C(1)-C(2) [1.420(4) Å] and C(1)-C(4) [1.411(4) Å] lie within the intermediate of the carbon-carbon single and double bonds lengths which indicates the enolate nature of the barbiturate anion.

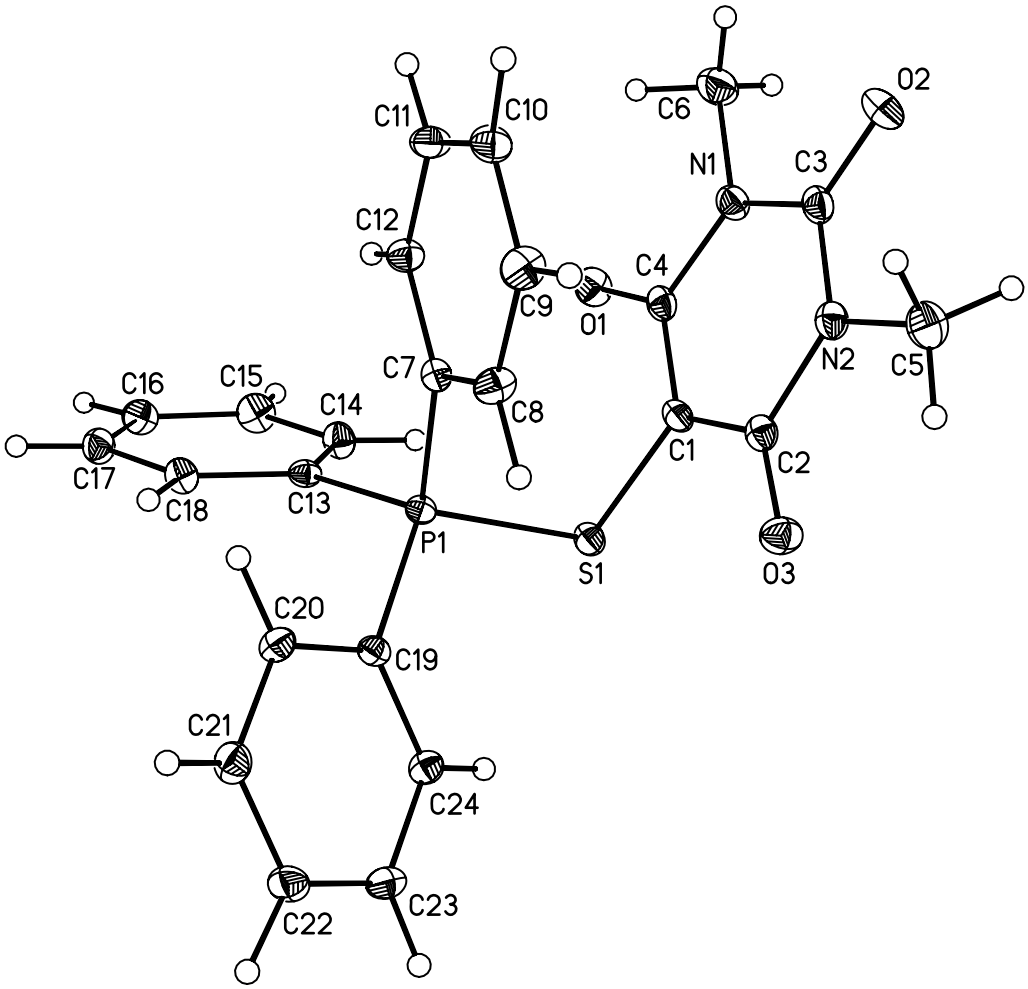


Fig. 34: The crystal structure of **69**

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: ± 0,5%

Hydrogen: ± 0,3%

Nitrogen: ± 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*). The FD spectra were required on a Finnigan MAT 711A at 30°C.

4.1.4 NMR spectra

The high resolution NMR spectra were acquired by a Bruker DRX 250 NMR spectrometer operating at ^{1}H: 250,13 MHz; ^{13}C: 62,90 MHz; ^{31}P: 101,20 MHz. The spectra were measured relative to TMS (^{1}H, ^{13}C) and 85% H_3PO_4 (^{31}P) as external 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 (**32, 35, 36, 41, 45, 47, 54**), a Nonius Kappa CCD diffractometer (**31, 33, 37, 38, 39, 43, 46, 48, 49, 51, 52, 53, 55, 56, 57, 59, 60, 61, 62, 64, 65, 66, 67, 69**), and a Stoe IPDS diffractometer (**42, 44, 68**) 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-dimethylbarbituric acid	**13** [Aldrich]
1,3-diisopropyl-4,5-dimethyl-2-ylidene	**4c** [3]
1,3-dicyclohexyl-4,5-dimethyl-2-ylidene	**34** [3]
1,3-diadamantyl-4,5-dihydro-2-ylidene	**2** [2]

4.5 Preparation of the compounds

4.5.1 Preparation of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium phosphonate **(31)**

0.3 g (3.7 mmol) of H_3PO_3 was added to a solution of 0.67 g (3.7 mmol) of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene in 10 ml Et_2O at -15°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from dichloromethane/diethylether 0.74 g (76 %), as colourless crystals. Elemental analysis for $C_{11}H_{23}N_2PO_3$ (262.29 g/mol): found (calc.) C 50.83 (50.37), H 8.39 (8.84), N 10.38 (10.68) %.

^{1}H-NMR (CD_2Cl_2):

δ = 1.61	[d, 12 H, 1,3-CH*Me*$_2$, ^{3}J=7.0 Hz]
δ = 2.32	[s, 6 H, 4,5-Me]
δ = 4.60	[sept, 2 H, C*H*Me$_2$]
δ = 9.24	[s, 1 H, 2-H$_{Im}$]
δ = 6.78	[d, 1 H, HP]
Not observed	[OH]

^{13}C-NMR (CD_2Cl_2):

δ = 7.3	[4,5-Me]
δ = 21.4	[1,3-CH*Me*$_2$]
δ = 49.9	[1,3-*C*HMe$_2$]
δ = 125.9	[C4,5]
δ = 130.7	[C2]

^{31}P-NMR (CD_2Cl_2):

δ = 12.39	[HPO_2OH]

4.5.2 Preparation of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium bromide **(32)**

A solution of 0.80 g (4.4 mmol) of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene in 5 ml THF was added to 0.72 g (4.0 mmol) of triethylammonium bromide in 15 ml of THF at r.t., after the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from dichloromethan/diethylether 0.97 g (84 %), as colourless crystals. Elemental analysis for $C_{11}H_{21}N_2Br$ (261.21 g/mol): found (calc.) C 50.17 (50.58), H 8.26 (8.10), N 10.37 (10.72) %.

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

δ = 1.64	[d, 12 H, 1,3-CH*Me*$_2$, ^{3}J=7.1 Hz]
δ = 2.20	[s, 6 H, 4,5-Me]
δ = 4.54	[sept, 2 H, C*H*Me$_2$]
δ = 10.09	[s, 1 H, 2-H$_{Im}$]

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

δ = 8.5	[4,5-Me]
δ = 22.6	[1,3-CH*Me*$_2$]
δ = 50.8	[1,3-*C*HMe$_2$]
δ = 125.4	[C4,5]
δ = 132.4	[C2]

4.5.3 Preparation of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium (*Z*)-2-cyano-1-phenyl-1-ethenolate **(33)**

A solution of 0.56 g (3.9 mmol) of benzoylacetonitril in 10 ml THF was added to 0.70 g (3.9 mmol) of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene in 15 ml of THF at r.t., after the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from acetonitrile/diethylether 1.15 g (91 %), as red crystals; m.p. 109°C. Elemental analysis for $C_{20}H_{27}N_3O$ (325.45 g/mol): found (calc.) C 73.43 (73.85), H 8.02 (8.31), N 12.71 (12.92) %.

^{1}H-NMR (CD_2Cl_2):

δ = 1.60	[d, 12 H, 1,3-CH*Me*$_2$, ^{3}J=7.0 Hz]

δ = 4.84	[sept, 2 H, C*H*Me$_2$]
δ = 2.23	[s, 6 H, 4,5-Me]
δ = 4.15	[s, 1 H, NCCH]
δ = 7.25-7.71	[m, 5 H, Ph]
δ = 10.37	[s, 1 H, 2-H$_{Im}$]

^{13}C-NMR (CD_2Cl_2):

δ = 8.3	[4,5-Me]
δ = 22.1	[1,3-CH*Me$_2$*]
δ = 50.7	[1,3-*C*HMe$_2$]
δ = 54.9	[*C*CN]
δ = 125.2, 125.8, 127.1, 127.7	[Ph]
δ = 128.0	[C4,5]
δ = 138.2	[C2]
δ = 135.1	[CN]
δ = 182.9	[CO]

4.5.4 Preparation of 1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium chloride **(35)**

A solution of 0.64 g (2.5 mmol) of 1,3-dicyclohexyl-4,5-dimethylimidazol-2-ylidene in 5 ml THF was added to 0.34 g (2.5 mmol) of triethylammonium chloride in 15 ml of THF at r.t., after the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from dichloromethane/diethylether 0.64 g (86 %), as colourless crystals. Elemental analysis for $C_{17}H_{29}N_2Cl$ (296.88 g/mol): found (calc.) C 68.24 (68.78), H 9.46 (9.85), N 9.37 (9.44) %.

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

δ = 1.43, 1.82, 1.98	[m,m,m, 4,8,8 H, cy]
δ = 2.45	[s, 6 H, 4,5-Me]
δ = 3.75	[sept, 2 H, C*H*Me$_2$]
δ = 8.30	[s, 1 H, 2-H$_{Im}$]

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

δ = 9.3	[4,5-Me]
δ = 25.2, 25.9, 34.6, 57.7	[C_6H_{11}]
δ = 128.1	[C4,5]
δ = 131.5	[C2]

4.5.5 Preparation of 1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium dicyanomethylide **(36)**

0.15 ml (2.3 mmol) of malononitrile was added to a solution of 0.6 g (2.3 mmol) of 1,3-dicyclohexyl-4,5-dimethylimidazol-2-ylidene in 30 ml of THF at -10°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from acetonitrile/diethylether 0.66 g (87 %), as colourless crystals; m.p. 142°C. Elemental analysis for $C_{20}H_{30}N_4$ (326.48 g/mol): found (calc.) C 73.17 (73.58), H 9.76 (9.26), N 17.37 (17.16) %.

^{1}H-NMR (CD_3CN):

δ = 1.31, 1.72, 1.86	[m,m,m, 4,8,8 H, cy]
δ = 2.20	[s, 6 H, 4,5-Me]
δ = 4.18	[m, 2 H, 1,3-CH]
δ = 2.3	[s, 1 H, $CH(CN)_2$]

^{13}C-NMR (CD_3CN):

δ = 7.3	[4,5-Me]
δ = 24.3, 24.7, 32.6, 56.7	[C_6H_{11}]
δ = 45.0	[$C(CN)_2$]
δ = 126.1	[C4,5]
δ = 129.5	[C2]
δ = 163.4	[$C(CN)_2$]

4.5.6 Preparation of 2-cyano-1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium bromide **(37)**

A solution of 0.34 g (3.2 mmol) of BrCN in 10 ml THF was added to a solution of 0.83 g (3.2 mmol) of 1,3-dicyclohexyl-4,5-dimethylimidazol-2-ylidene in 20 ml of THF at -76°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from methanol/diethylether 0.74 g (63 %), as red crystals. Elemental analysis for $C_{18}H_{28}N_3Br$ (366.34 g/mol): found (calc.) C 59.17 (59.01), H 7.26 (7.70), N 11.37 (11.47) %.

^{1}H-NMR (CD_2Cl_2):

δ = 1.48, 1.97, 2.24	[m,m,m, 4,8,8 H, cy]
δ = 2.54	[s, 6 H, 4,5-Me]
δ = 4.49	[m, 2 H, 1,3-CH]

^{13}C-NMR (CD_2Cl_2):

δ = 11.6	[4,5-Me]
δ = 25.3, 26.4, 32.4, 62.5	[C_6H_{11}]
δ = 126.3	[C4,5]
δ = 133.9	[C2]
δ = 107.8	[CN]

MS (FAB): m/z (%) = 286 [100, M^+], 261 [84, M^+ - CN] and further fragments.

5.4.7 Preparation of 1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium-2-carbodithioate **(38)**

0.34 (6.0 mmol) of CS_2 was added to a solution of 1.56 g (6.0 mmol) of 1,3-dicyclohexyl-4,5-dimethylimidazol-2-ylidene in 20 ml of THF at -15°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from methanol/diethylether 1.72 g (85 %), as red crystals. Elemental analysis for $C_{18}H_{28}N_2S_2$ (336.56 g/mol): found (calc.) C 64.07 (64.24), H 8.26 (8.39), N 8.77 (8.32) %.

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

δ = 2.27	[s, 6 H, 4,5-Me]
δ = 4.50	[m, 2 H, 1,3-CH]

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

δ = 10.9	[4,5-Me]
δ = 25.4, 26.1, 31.2, 59.6	[C_6H_{11}]
δ = 123.4	[C4,5]
δ = 150.3	[C2]
δ = 230.1	[CS_2]

4.5.8 Preparation of 1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium phenylphosphonate **(39)**

A solution of 0.25 g (1.6 mmol) of $PhP(O)(OH)_2$ in 10 ml THF was added to a solution of 0.42 g (1.6 mmol) of 1,3-dicyclohexyl-4,5-dimethylimidazol-2-ylidene in 20 ml of THF at -10°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from methanol/diethylether 0.60 g (90 %), as colourless crystals. Elemental analysis for $C_{23}H_{35}N_2O_3P$ (418.51 g/mol): found (calc.) C 66.47 (66.01), H 8.06 (8.43), N 6.27 (6.69) %.

^{1}H-NMR (CD_3OD):

δ = 1.31, 1.72, 1.86	[m,m,m, 4,8,8 H, cy]
δ = 2.11	[s, 6 H, 4,5-Me]
δ = 4.24	[m, 2 H, 1,3-CH]
δ = 8.92	[s, 1 H, 2-H_{Im}]
δ = 7.55-8.15	[m, 15 H, Ph]
Not observed	[OH]

^{13}C-NMR (CD_3OD):

δ = 8.6	[4,5-Me]
δ = 26.3, 26.8, 34.6, 58.9	[C_6H_{11}]
δ = 128.8-132.2	[Ph]
δ = 132.3	[C2]

^{31}P-NMR (CD_3OD):

δ = 13.05	[$PhPO_2OH$]

4.5.9 Preparation of 1,3-di(1-adamantyl)-1,3-dihydro-2H-imidazole-2-thione **(41)**

A solution of 0.42 g (1.25 mmol) of 1,3-diadamantyl-4,5- dihydroimidazol-2-ylidene and 0.04 g S in 25 ml THF was refluxed for 8 hr. After cooling, solvent was removed in vacuo at r.t. to dryness. The residue was recrystallised from dichloromethane/diethylether to give 0.15 g (33 %), as yellow crystals; m.p. 157°C. Elemental analysis for $C_{23}H_{32}N_2S$ (368.58 g/mol): found (calc.) C 74.45 (74.95), H 8.31 (8.75), N 7.15 (7.60) %.

^{1}H-NMR (d_8-toluene):

δ = 1.56-2.57	[$C_{10}H_{15}$]
δ = 6.36	[d, 2 H, 4,5-Me]

^{13}C-NMR (d_8-toluene):

δ = 30.4, 36.4, 39.6, 60.1	[$C_{10}H_{15}$]
δ = 112.6	[C4,5]
δ = 188.1	[C2]

MS (FAB): m/z (%) = 369 [100, M^+], 341 [24, $M^+ - 2CH_2$] and further fragments.

4.5.10 Preparation of 1,3-di(1-adamantyl)-1H-imidazol-3-ium thiocyanate **(42)**

A solution of 0.59 g (1.75 mmol) of 1,3-diadamantyl-4,5- dihydroimidazol-2-ylidene in 5 ml THF was added to 0.13 g (1.75 mmol) of H_4NSCN in 15 ml of THF at r.t., after the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from dichloromethane/diethylether 0.58 g (84 %), as colourless crystals. Elemental analysis for $C_{24}H_{33}N_3S$ (395.61 g/mol): found (calc.) C 72.34 (72.86), H 8.86 (8.41), N 10.37 (10.62) %.

^{1}H-NMR (CD_3OD):

δ = 1.56-3.74	[$C_{10}H_{15}$]
δ = 7.91	[d, 2 H, 4,5-Me]
δ = 8.94	[s, 1 H, 2-H_{Im}]

^{13}C-NMR (CD_3OD):

δ = 31.4, 36.8, 43.8, 61.8	[$C_{10}H_{15}$]
δ = 131.3	[C4,5]
δ = 120.9	[C2]
Not observed	[S*C*N]

4.5.11 Preparation of 2-bromo-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium dicyanoargentate **(43)**

1.3 g (9.7 mmol) of silver cyanide was added to a solution of 1.1 g (3.2 mmol) of 2-bromo-1,3-diisopropyl-4,5-dimethylimidazoliumbromide in 30 ml of acetonitrile at r.t., the mixture was stirred for 48 hr. The solvent was removed in vacuo and 20 ml of dichloromethane was added. The resulting solution was filtered off and solvent was removed in vacuo. Yield after recrystallisation from dichloromethane/diethylether 0.98 g (73 %), as colourless crystals. Elemental analysis for $C_{13}H_{20}N_4BrAg$ (420.10 g/mol): found (calc.) C 37.54 (37.17), H 4.38 (4.80), N 13.01(13.34) %.

^{1}H-NMR (CD_3CN):

δ = 1.59	[d, 12 H, 1,3-CH*Me*$_2$, ^{3}J=6.9 Hz]
δ = 2.33	[s, 6 H, 4,5-Me]
δ = 4.89	[sept, 2 H, C*H*Me$_2$]

^{13}C-NMR (CD_3CN):

δ = 9.3	[4,5-Me]
δ = 19.4	[1,3-CH*Me*$_2$]
δ = 53.3	[1,3-*C*HMe$_2$]
δ = 129.0	[C4,5]
δ = 142.0	[Ag(CN)$_2$]
Not observed	[C2]

4.5.12 Preparation of 2-cyano-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium chloride **(44)**

A mixture solution of 0.20 g (3.1 mmol) of KCN and 0.82 g (3.0 mmol) 18-crown-6-ether in 15 ml CH_2Cl_2 was added to solution of 0.78 g (3.1 mmol) of 2-chloro-1,3-diisopropyl-4,5-dimethylimidazoliumchloride in 15 ml of CH_2Cl_2 at r.t., the solution was refluxed for 8 hr. After cooling, solvent was removed in vacuo to dryness. The residue was recrystallised from dichloromethane/THF to give 0.47 g (65 %), as colourless crystals. Elemental analysis for $C_{12}H_{20}N_3Cl$ (241.76 g/mol): found (calc.) C 59.23 (59.62), H 8.24 (8.34), N 17.88 (17.38) %.

^{1}H-NMR (d_6-DMSO):

δ = 1.61	[d, 12 H, 1,3-CH*Me*$_2$, ^{3}J=6.9 Hz]
δ = 2.48	[s, 6 H, 4,5-Me]
δ = 4.94	[sept, 2 H, C*H*Me$_2$]

^{13}C-NMR (d_6-DMSO):

δ = 9.69	[4,5-Me]
δ = 21.2	[1,3-CH*Me*$_2$]
δ = 53.7	[1,3-*C*HMe$_2$]
δ = 132.2	[C4,5]
δ = 112.4	[C2]
δ = 107.6	[*C*N]

MS (FAB): m/z (%) = 206 [100, M^+], 164 [23, M^+ - Me] and further fragments.

4.5.13 Preparation of 2-(1,3-diisopropyl-4,5-dimethyl-1,3-dihydro-2H-imidazol-2-ylidene)malononitrile **(45)** and 2-(1,3-diethyl-4,5-dimethyl-1,3-dihydro-2H-imidazol-2-ylidene) malononitrile **(46)**

Preparation of **(45)**:

0.24 ml (3.8 mmol) of malononitrile was added to a solution of 1.3 g (3.8 mmol) of 2-bromo-1,3-diisopropyl-4,5-dimethylimidazoliumbromide in 30 ml of acetonitrile at -30 °C. Then 7 ml of triethylamine was added slowly and the mixture was stirred for 24 hr at r.t., the solvent was removed in vacuo and the residue was dissolved in 15 ml of dichloromethane. The resulting solution was extracted with 30 ml of water. The organic layer was dried over Na_2SO_4 and

evaporated in vacuo to dryness. The residue was recrystallised from dichloromethane/ diethylether to give 0.59 g (64 %), colourless crystals. Elemental analysis for $C_{14}H_{20}N_4$ (244.34 g/mol): found (calc.) C 68.11(68.82), H 7.89 (8.25), N 22.72 (22.93) %.

^{1}H-NMR (CD_2Cl_2):

δ = 1.46	[d, 12 H, 1,3-CH*Me$_2$*, ^{3}J=6.9 Hz]
δ = 2.18	[s, 6 H, 4,5-Me]
δ = 4.96	[sept, 2 H, C*H*Me$_2$]

^{13}C-NMR (CD_2Cl_2):

δ = 10.5	[4,5-Me]
δ = 21.2	[1,3-CH*Me$_2$*]
δ = 51.5	[1,3-*C*HMe$_2$]
δ = 125.4	[C4,5]
δ = 144.4	[C2]
δ = 14.0	[*C*(CN)$_2$]
δ = 122.9	[C(*C*N)$_2$]

MS (FAB): m/z (%) = 245 [100, M^+], 267 [21, M^+ +Na], 203 [20, M^+ - CN, - Me] and further fragments.

Preparation of **(46)**:

0.17 ml (2.7 mmol) of malononitrile was added to a solution of 0.8 g (2.7 mmol) of 2-bromo-1,3-diethyl-4,5-dimethylimidazoliumbromide in 30 ml of acetonitrile at -30°C. Then 7 ml of triethylamine was added slowly and the mixture was stirred for 24 hr at r.t., the solvent was removed in vacuo and the residue was dissolved in 15 ml of dichloromethane. The resulting solution was extracted with 30 ml of water. The organic layer was dried over Na_2SO_4 and evaporated in vacuo to dryness. The residue was recrystallised from dichloromethane/ diethylether to give 0.41 g (74 %), as colourless crystals. Elemental analysis for $C_{12}H_{16}N_4$ (216.29 g/mol): found (calc.) C 66.19 (66.64), H 7.28 (7.46), N 26.47 (25.90) %.

^{1}H-NMR (CD_2Cl_2):

δ = 1.30	[br s, 6 H, 1,3-CH*Me$_2$*]
δ = 2.08	[s, 6 H, 4,5-Me]

δ = 4.00	[br s, 4 H, C*H*$_2$Me]

^{13}C-NMR (CD_2Cl_2):

δ = 8.7	[4,5-Me]
δ = 14.5	[1,3-CH*Me*$_2$]
δ = 40.7	[1,3-*C*HMe$_2$]
δ = 123.5	[C4,5]
δ = 142.5	[C2]
δ = 18.8	[*C*(CN)$_2$]
δ = 121.7	[C(*C*N)$_2$]

MS (FAB): m/z (%) = 217 [100, M^+], 244 [21, M^+ +Na], 203 [10, M^+ - N] and further fragments.

4.5.14 Preparation of 2-amino-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium chloride **(47)**

0.3 g (7.7 mmol) of $NaNH_2$ was added to solution of 1.9 g (7.7 mmol) of 2-chloro-1,3-diisopropyl-4,5-dimethylimidazoliumchloride in 25 ml of THF at r.t., the solution was stirred for 72 hr at rt. The resulting solution was filtered off and solvent was removed in vacuo. Yield after recrystallisation from dichloromethane/diethylether 0.38 g (21 %), as colourless crystals. Elemental analysis for $C_{11}H_{22}N_3Cl$ (231.77 g/mol): found (calc.) C 57.43 (57.00), H 9.24 (9.57), N 18.38 (18.13) %.

^{1}H-NMR (CD_3CN):

δ = 1.65	[d, 12 H, 1,3-CH*Me*$_2$, ^{3}J=6.9 Hz]
δ = 2.18	[s, 6 H, 4,5-Me]
δ = 4.98	[sept, 2 H, C*H*Me$_2$]
Not observed	[2 H, NH_2]

^{13}C-NMR (CD_3CN):

δ = 10.1	[4,5-Me]
δ = 22.8	[1,3-CH*Me*$_2$]
δ = 51.1	[1,3-*C*HMe$_2$]

δ = 129.2 [C4,5]

δ = 120.1 [C2]

MS (FAB): m/z (%) = 196 [100, M^+], 180 [60, M^+ - NH_2] and further fragments.

4.5.15 Preparation of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium nitrate **(48)**

0.78 g (4.6 mmol) of silver nitrate was added to a solution of 0.80 g (3.1 mmol) of 1,3-diisopropyl-4,5-dimethylimidazoliumbromide in 30 ml of acetonitrile. The mixture was stirred for 24 hr at r.t., the solvent was removed in vacuo and 15 ml of dichloromethane was added. The resulting solution was filtered off. The solvent was removed in vacuo and the residue was recrystallised from dichloromethane/diethylether to give 0.69 g (92 %), as colourless crystals. Elemental analysis for $C_{11}H_{21}N_3O_3$ (243.31 g/mol): found (calc.) C 53.72 (54.30), H 8.98 (8.70), N 17.09 (17.27) %.

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

δ = 1.53 [d, 12 H, 1,3-CH*Me$_2$*, ^{3}J =7.0 Hz]

δ = 2.16 [s, 6 H, 4,5-Me]

δ = 4.43 [sept, 2 H, C*H*Me$_2$]

δ = 9.99 [s, 1 H, 2-H_{Im}]

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

δ = 9.3 [4,5-Me]

δ = 23.3 [1,3-CH*Me$_2$*]

δ = 51.4 [1,3-*C*HMe$_2$]

δ = 126.2 [C4,5]

δ = 133.2 [C2]

4.5.16 Preparation of 2-bromo-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-iumnitrate **(49)**

0.52 g (3.1 mmol) of silver nitrate was added to a solution of 0.70 g (2.1 mmol) of 2-bromo-1,3-diisopropyl-4,5-dimethylimidazoliumbromide in 30 ml of acetonitrile. The mixture was stirred for 24 hr at r.t., the solvent was removed in vacuo and 15 ml of dichloromethane was added. The

resulting solution was filtered off. The solvent was removed in vacuo and the residue was recrystallised from dichloromethane/diethylether to give 0.56 g (84 %), colourless crystals. Elemental analysis for $C_{11}H_{20}N_3O_3Br$ (322.20 g/mol): found (calc.) C 40.57 (41.01), H 6.41 (6.26), N 12.71 (13.04) %.

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

δ = 1.65	[d, 12 H, 1,3-CH*Me*$_2$, ^{3}J=6.9 Hz]
δ = 2.39	[s, 6 H, 4,5-Me]
δ = 4.98	[sept, 2 H, C*H*Me$_2$]

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

δ = 11.3	[4,5-Me]
δ = 21.4	[1,3-CH*Me*$_2$]
δ = 54.4	[1,3-*C*HMe$_2$]
δ = 129.1	[C4,5]
Not observed	[C2]

4.5.17 Preparation of 1,3-dimethyl-2,6-dioxo-5-(1-pyridiniumylmethyl)-1,2,3,6-tetrahydro-4-pyrimidinolate **(50)**

2.4 ml (32 mmol) of 37% aq. formaldehyde was added in one portion to 5.0 g (32 mmol) of 1, 3-dimethylbarbituric acid in 25 ml of pyridine at r.t., after the reaction mixture was stirred for 1 hr, the solvent was removed in vacuo at 50°C and the residue was dissolved in 40 ml of hexane for 1 hr. The precipitate was filtered off and dried in vacuo to give 7.2 g (91 %), yellow precipitate; m.p. 337°C. Elemental analysis for $C_{12}H_{13}N_3O_3$ (247.25 g/mol): found (calc.) C 58.43 (58.29), H 5.00 (5.30), N 16.98 (17.00) %.

^{1}H-NMR (CD_2Cl_2):

δ = 2.98	[s, 6 H, 1,3-Me$_B$]
δ = 5.24	[s, 2 H, CH_2]
δ = 7.26, 7.71, 8.53	[3br s, 5 H, Py]

^{13}C-NMR (CD_2Cl_2):

δ = 29.0	[1,3-Me$_B$]

δ = 46.3	[$C5_B$]
δ = 51.8	[CH_2]
δ = 150.2	[$C2_B$]
δ = 169.8	[$C4,6_B$]
δ = 124.4, 136.8, 149.7	[Py]

MS (FAB, NBA-Matrix): m/z (%) = 247[41, M^+], 79[100, $C_5H_5N^+$] and further fragments.

4.5.18 Preparation of 1,3-dimethyl-2,6-dioxo-5-[(triphenylphosphonio)methyl]-1,2,3,6-tetrahydro-4-pyrimidinolate **(51)**

A solution of 0.68 g (2.6 mmol) of PPh_3 in 5 ml CH_2Cl_2 was added to 0.65 g (2.6 mmol) of **50** in 15 ml of CH_2Cl_2 at r.t., after the reaction mixture was stirred over night, the solvent was removed in vacuo to dryness. The residue was recrystallised from dichloromethane/diethylether to give 1.01 g (90 %), as green crystals; m.p. 189°C (decomp.). Elemental analysis for $C_{25}H_{23}N_2O_3P$ (430.44 g/mol): found (calc.) C 69.43 (69.76), H 5.34 (5.39), N 6.63 (6.51) %.

^{1}H-NMR (CD_2Cl_2):

δ = 3.11	[s, 6 H, 1,3-Me_B]
δ = 7.55-8.15	[m, 15 H, Ph]
δ = 4.35	[s, 2 H, CH_2]

^{13}C-NMR (CD_2Cl_2):

δ = 27.4	[1,3-Me_B]
δ = 75.1	[$C5_B$]
δ = 153.6	[$C2_B$]
δ = 163.6	[$C4,6_B$]
δ = 25.7	[CH_2, ^1J = 43.8 Hz]
δ = 120.7, 129.6, 134.4, 134.6	[Ph]

^{31}P-NMR ($CDCl_3$):

δ = 23.9	[CH_2PPh_3]

MS (FAB): m/z (%) = 431 [41, M^+], 262 [100, PPh_3^+] and further fragments.

This compound can also be prepared by another reaction:

A solution of 0.33 g (1.3 mmol) of PPh_3 in 5 ml CH_2Cl_2 was added to 0.21 g (1.3 mmol) of **53** in 20 ml of CH_2Cl_2 at r.t., after the reaction mixture was stirred over night, the solvent was removed in vacuo to dryness. The residue was recrystallised from dichloromethane/diethylether to give 0.48 g (86 %). Elemental analysis for $C_{25}H_{23}N_2O_3P$ (430.44 g/mol): found (calc.) C 70.05 (69.76), H 5.75 (5.39), N 6.83 (6.51) %.

4.5.19 Preparation of 5-[(1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium-2-yl)methyl]-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate **(52)**

A solution of 0.43 g (2.4 mmol) of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene in 8 ml of THF was added to 0.60 g (2.4 mmol) of **50** in 15 ml of THF at -10°C. After the reaction mixture was stirred over night, the solvent was removed in vacuo to dryness. The residue was recrystallised from dichloromethane/diethylether to give 0.81 g (88 %), as colourless crystals; m.p. 138°C (decomp). Elemental analysis for $C_{18}H_{28}N_4O_3$ (348.44 g/mol): found (calc.) C 61.72 (62.05), H 8.14 (8.10), N 16.22 (16.08) %.

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

δ = 3.19	[s, 6 H, 1,3-Me_B]
δ = 3.10	[s, 2 H, CH_2]
δ = 1.61	[d, 12 H, 1,3-CH*Me*$_2$, ^{3}J= 7.0 Hz]
δ = 2.28	[s, 6 H, 4,5-Me]
δ = 4.50	[sept, 2 H, C*H*Me_2]

^{13}C-NMR (CD_2Cl_2):

δ = 26.3	[1,3-Me_B]
δ = 79.3	[$C5_B$]
δ = 153.6	[$C2_B$]
δ = 164.6	[$C4,6_B$]
δ = 21.4	[CH_2]
δ = 9.3	[4,5-Me]
δ = 19.9	[1,3-CH*Me*$_2$]

δ = 49.8	[1,3-*C*HMe$_2$]
δ = 124.7	[C4,5]
δ = 146.7	[C2]

MS (FAB): m/z (%) = 349 [20, M^+], 181 [100, ImH^+] and further fragments.

4.5.20 Preparation of 1,3-dimethyl-5-methylene-2,4,6(1H,3H,5H)-pyrimidinetrione **(53)**

0.15 ml (2.0 mmol) of CF_3COOH was added to a solution of 0.49 g (2.0 mmol) of **50** in 15 ml CH_2Cl_2 at r.t., after the reaction mixture was stirred 30 min. The solvent was removed in vacuo at r.t. to dryness. The residue was recrystallised from benzene to give 0.16 g (48 %). Yellow crystals were obtained by sublimation under vacium at 220°C. Elemental analysis for $C_7H_8N_2O_3$ (168.15 g/mol): found (calc.) C 50.43 (50.00), H 4.51 (4.80), N 16.98 (16.66) %.

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

δ = 3.00	[s, 6 H, 1,3-Me$_B$]
δ = 2.11	[s, 2 H, CH_2]

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

δ = 28.3	[1,3-Me$_B$]
δ = 51.0	[C5$_B$]
δ = 149.4	[C2$_B$]
δ = 169.0	[C4,6$_B$]
δ = 45.5	[CH_2]

MS (FD): m/z (%) = 168 [100], 156 [3, - CH_2].

4.5.21 Preparation of 5,5-dichloro-1,3-dimethyl-2,4,6(1H,3H,5H)-pyrimidinetrione **(54)**

A mixture of 1.45 ml (18.0 mmol) of sulfuryl chloride and 1.40 g (9.0 mmol) of 1,3-dimethylbarbituric acid in 25 ml of THF was refluxed for 6 hr. After cooling, solvent was removed in vacuo at r.t. to dryness. The residue was recrystallised from dichloromethane/

diethylether to give 1.72 g (85 %), as colourless crystals; m.p. 157°C. Elemental analysis for $C_6H_6Cl_2N_2O_3$ (225.03 g/mol): found (calc.) C 32.25 (32.02), H 2.51 (2.69), N 12.68 (12.45) %. IR (KBr): ν_{CO} = 1655 (sst, br) cm^{-1}

^{1}H-NMR (CD_2Cl_2):

δ = 3.30	[s, 6 H, 1,3-Me]

^{13}C-NMR (CD_2Cl_2):

δ = 30.7	[1,3-Me]
δ = 149.1	[C2]
δ = 72.2	[C5]
δ = 161.7	[C4,6]

MS (FAB): m/z (%) = 225 [93, M^+], 191 [100, M^+ - Cl] and further fragments.

4.5.22 Preparation of 5-chloro-1,3-dimethyl-5-nitro-2,4,6(1H,3H,5H)-pyrimidinetrione **(55)**

1.25 g (8.1 mmol) of silver nitrite was added to a solution of 0.80 g (3.6) of **54** in 30 ml of acetonitrile. The mixture was stirred for 24 hr at r.t., the solvent was removed in vacuo and 15 ml of dichloromethane was added. The resulting solution was filtered off and solvent was removed in vacuo. Yield after recrystallisation from dichloromethane/diethylether 0.62 g (73 %), as colourless crystals; m.p. 121°C. Elemental analysis for $C_6H_6ClN_3O_5$ (235.58 g/mol): found (calc.) C 30.78 (30.59), H 2.43 (2.57), N 17.62 (17.84) %.

IR (KBr): ν_{CO} = 1645 (sst, br) cm^{-1}

^{1}H-NMR (CD_2Cl_2):

δ = 3.34	[s, 6 H, 1,3-Me]

^{13}C-NMR (CD_2Cl_2):

δ = 30.6	[1,3-Me]
δ = 149.2	[C2]
δ = 89.2	[C5]
δ = 159.1	[C4,6]

MS (EI): m/z (%) = 189 [6, M^+ - NO_2], 161 [100, M^+ - NO_2 - 2Me] and further fragments.

4.5.23 Preparation of 2-chloro-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium 1,3-dimethyl-5- nitro-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate **(56)**

A solution of 0.80 g (4.4 mmol) of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene in 5 ml of diethylether was added to 1.04 g (4.4 mmol) of **55** in 25 ml of diethylether at -78 °C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from dichloromethane/diethylether 1.58 g (86 %), as colourless crystals; m.p. 141°C. Elemental analysis for $C_{17}H_{26}ClN_5O_5$ (415.87 g/mol): found (calc.) C 49.39 (49.10), H 6.02 (6.30), N 16.71 (16.84) %.

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

δ = 3.06	[s, 6 H, 1,3-Me_B]

δ = 1.43 [d, 12 H, 1,3-CH*Me*$_2$, ^{3}J= 6.8 Hz]

δ = 2.16 [s, 6 H, 4,5-Me]

δ = 4.78 [sept, 2 H, C*H*Me$_2$]

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

δ = 30.6 [1,3-Me$_B$]

δ = 151.6 [C2$_B$]

δ = 114.2 [C5$_B$]

δ = 158.2 [C4,6$_B$]

δ = 9.8 [4,5-Me]

δ = 20.2 [1,3-CH*Me*$_2$]

δ = 52.6 [1,3-*C*HMe$_2$]

δ = 126.3 [C4,5]

δ = 127.9 [C2]

MS (FAB): m/z (%) = 215 [100, M^+], 173 [14, M^+ - C_3H_7], 136 [23, M^+ - (C_3H_7), - Cl] and further fragments.

4.5.24 Preparation of 5,6-dihydro-1,3-dimethyl-5,6-bis-[1',3'-dimethyl]-2',4',6'-trioxopyrimid (5',5') yl[2,3-d] uracil **(57)**

A solution of 0.53 g (2.9 mmol) of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene in 5 ml of Et_2O was added to 0.65 g (2.9 mmol) of **54** in 15 ml of Et_2O at -20°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from dichloromethane/diethylether 1.11 g (83 %), as colourless crystals. Elemental analysis for $C_{18}H_{18}N_6O_9$ (462.37 g/mol): found (calc.) C 46.91 (46.76), H 3.60 (3.92), N 18.52 (18.18) %.

^{1}H-NMR (d_6-DMSO):

δ = 3.26-3.29-3.52 [s,s,s, 6,9,3 H,1,3-Me$_B$]

MS (FAB): m/z (%) = 215 [100, M^+], 173 [18, M^+ - C_3H_7], 136 [20, M^+ - (C_3H_7), - Cl] and further fragments.

4.5.25 Preparation of potassium 5-cyano-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate 1,4,7,10,13,16-hexaoxacyclooctadecane **(59)**

A mixture solution of 0.35 g (5.4 mmol) of KCN and 0.93 g (3.5 mmol) of 18-crown-6-ether in 15 ml CH_2Cl_2 was added to solution of 0.40 g (1.8 mmol) of **54** in 15 ml of CH_2Cl_2 at r.t., after the reaction mixture was stirred over night, solvent was removed in vacuo to dryness. The residue was recrystallised from dichloromethane/diethylether to give 0.48 g (55 %), as yellow crystals. Elemental analysis for $C_{19}H_{18}N_3O_3K$ (483.56 g/mol): found (calc.) C 47.43 (47.19), H 6.24 (6.25), N 8.98 (8.69) %.

^{1}H-NMR (CD_2Cl_2):

δ = 3.51	[s, 6 H, 1,3-Me_B]
δ = 3.65	[s, 12 H, crown ether]

^{13}C-NMR (CD_2Cl_2):

δ = 30.6	[1,3-Me_B]
δ = 41.4	[$C5_B$]
δ = 109.4	[CN]
δ = 154.3	[$C2_B$]
δ = 165.1	[$C4,6_B$]

4.5.26 Preparation of 5-[1-(1,3-dimethyl-2,4,6-trioxohexahydro-5-pyrimidinyl)-2-oxopropyl]-1,3-dimethyl-2,4,6(1H,3H,5H)-pyrimidinetrione **(60)**

A solution of 0.74 g (2.4 mmol) of 5,5-dibromo-1,3 dimethylbarbituric acid in 10 ml of acetone was added to 0.19 g (2.4 mmol) of Na_2S in 15 ml of H_2O at r.t., after the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from dichloromethane/diethylether 0.24 g (22 %), as colourless crystals; m.p. 115°C. Elemental analysis for $C_9H_{18}N_4O_7$ (366.33 g/mol): found (calc.) C 49.01 (49.18), H 4.54 (4.95), N 15.48 (15.29) %.

^{1}H-NMR (CD_2Cl_2):

δ = 3.21	[s, 12 H, 1,3-Me_B]
δ = 3.85	[s, 2 H, 5-H_B]
δ = 1.62	[s, 3 H, Ac]
δ = 4.01	[s, 1 H, *H*CAc]

^{13}C-NMR (CD_2Cl_2):

δ = 29.3	[1,3-Me_B]
δ = 49.1	[$C5_B$]
δ = 151.5	[$C2_B$]
δ = 166.7	[$C4,6_B$]
δ = 24.9	[CO*C*H_3]
δ = 57.7	[*H*CAc]
δ = 200.1	[*C*OCH_3]

MS (FAB): m/z (%) = 153 [100, $C_6H_6N_2O_3^+$], 309 [63, 2 $C_6H_7N_2O_3^+$], 168 [24, $C_7H_7N_2O_3^+$] and further fragments.

4.5.27 Preparation of [5,5'-Bipyrimidine]-2,2',4,4',6,6'(1H,1'H,3H,3'H,5H,5'H)-hexone, 5,5'-dihydroxy-1,1',3,3'-tetramethyl **(61)**

A solution of 1.66 g (5.3 mmol) of 5,5-dibromo-1,3 dimethylbarbituric acid in 10 ml THF was added to 0.41 g (5.3 mmol) of Na_2S in 8 ml of H_2O at r.t., after the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from (dimethylsulfoxide/dichloromethane)/diethylether 0.38 g (21 %), as colourless crystals. Elemental analysis for $C_{12}H_{14}N_4O_8$ (342.26 g/mol): found (calc.) C 42.51 (42.11), H 4.54 (4.12), N 16.18 (16.37) %.

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

δ = 3.31	[s, 6 H, 1,3-Me_B]
Not observed	[s, 2 H, OH]

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

δ = 28.5	[1,3-Me_B]
δ = 77.3	[$C5_B$]
δ = 151.0	[$C2_B$]
δ = 163.5	[$C4,6_B$]

MS (FAB): m/z (%) = 341 [9, M^+], 267 [13, M^+ - 4 Me, - O] and further fragments.

4.5.28 Preparation 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium 1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate **(62)**

A solution of 0.80 g (4.4 mmol) of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene in 5 ml of Et_2O was added to 0.69 g (4.4 mmol) of 1,3 dimethylbarbituric acid in 15 ml of THF at -10°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from dichloromethane/diethylether 1.38 g (93 %), as colourless crystals; m.p. 135°C. Elemental analysis for $C_{17}H_{27}N_4O_3$ (335.43 g/mol): found (calc.) C 60.43 (60.87), H 8.24 (8.11), N 16.98 (16.70) %.

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

δ = 3.21	[s, 6 H, 1,3-Me_B]
δ = 4.59	[s, 1 H,5-H_B]
δ = 1.54	[d, 12 H, 1,3-CH*Me*$_2$, ^{3}J =6.9 Hz]
δ = 2.22	[s, 6 H, 4,5-Me]
δ = 4.51	[sept, 2 H, C*H*Me_2]
δ = 9.80	[s, 1 H, 2-H_{Im}]

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

δ = 26.9	[1,3-Me_B]
δ = 76.8	[$C5_B$]
δ = 153.6	[$C2_B$]
δ = 164.6	[$C4,6_B$]
δ = 8.2	[4,5-Me]

δ = 22.2 [1,3-CH*Me*$_2$]

δ = 50.5 [1,3-*C*HMe$_2$]

δ = 125.8 [C4,5]

δ = 131.9 [C2]

4.5.29 Preparation of 1,3-dimethyl-1,3-dihydro-2H-imidazol-2-iminium 1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate **(64)**

A solution of 0.37 g (3.3 mmol) of 2-imino-1,3-dimethylimidazoline in 5 ml THF was added to 0.51 g (3.3 mmol) of 1,3 dimethylbarbituric acid in 15 ml of THF at -10°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from ethanol/diethylether 0.79 g (90 %), as colourless crystals; m.p. 149°C. Elemental analysis for $C_{11}H_{17}N_5O_3$ (267.29 g/mol): found (calc.) C 49.63 (49.38), H 6.44 (6.36), N 26.02 (26.19) %.

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

δ = 3.45 [s, 6 H, 1,3-Me$_B$]

δ = 4.04 [s, 1 H, 5-H$_B$]

δ = 3.01 [s, 6 H, 1,3- Me$_{Im}$]

δ = 7.00 [s, 2 H, 4,5-H$_{Im}$]

δ = 7.87 [br s, 2 H, NH$_2$]

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

δ = 26.6 [1,3-Me$_B$]

δ = 75.1 [C5$_B$]

δ = 153.6 [C2$_B$]

δ = 163.0 [C4,6$_B$]

δ = 32.5 [1,3-Me$_{Im}$]

δ = 146.1 [C2$_{Im}$]

δ = 116.4 [C4,5$_{Im}$]

4.5.30 Preparation of [5,5'-Bipyrimidine]-2,2',4,4',6,6'(1H,1'H,3H,3'H,5H,5'H)-hexone, 1,1',3,3'-tetramethyl **(65)**

A solution of 0.64 g (1.28 mmol) $BiPh_3CO_3$ and 0.13 g (0.83 mmol) of 1,3-dimethylbarbituric acid in 20 ml CH_2Cl_2 was refluxed for 20 hr. After cooling, solvent was removed in vacuo at r.t., to dryness. The residue was recrystallised from dichloromethane/diethylether to give 0.10 g (38 %), as green crystals; m.p. 157°C. Elemental analysis for $C_{12}H_{14}N_4O_6$ (310.26 g/mol): found (calc.) C 46.41 (46.45), H 4.03 (4.55), N 18.48 (18.06) %.

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

δ = 3.15	[s, 12 H, 1,3-Me_B]
δ = 3.81	[s, 2 H, 5-H_B]

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

δ = 28.2	[1,3-Me_B]
δ = 46.9	[$C5_B$]
δ = 151.2	[$C2_B$]
δ = 161.5	[$C4,6_B$]

MS (EI): m/z (%) = 310 [5, M^+], 280 [9, M^+ - 2Me] and further fragments.

4.5.31 Preparation of 2,6-hydroxy-5-[(6-hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydro-5-pyrimidinyl)sulfanyl]-1,3-dimethyl-2,4(1H,3H)-pyrimidinedione **(66)**

1 ml (13.7 mmol) of $SOCl_2$ was added to a solution of 2.1 g (13.7 mmol) of 1,3-dimethylbarbituric acid in 15 ml of THF at -15°C. After the reaction mixture was stirred 30 min., the solvent was removed in vacuo at r.t. to dryness. The residue was recrystallised from acetonitrile to give 1.91 g (82 %), as colourless crystals. Elemental analysis for $C_{12}H_{14}N_4O_6S$ (342.33 g/mol): found (calc.) C 42.43 (42.10), H 4.23 (4.12), N 16.68 (16.37) %.

^{13}C-NMR (MAS):

δ = 29.1	[1,3-Me_B]
δ = 81.7	[$C5_B$]
δ = 150.1	[$C2_B$]

δ = 156.5 [C4,6$_B$]

MS (FAB): m/z (%) = 343 [100, M^+], 309 [25, M^+, -S], 187 [32, M^+, - $C_6H_7N_2O_3$] and further fragments.

4.5.32 Preparation of 1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium 5-[(6-hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydro-5-pyrimidinyl)sulfanyl]-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate **(67)**

A solution of 0.27 g (1.5 mmol) of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene in 5 ml Et_2O was added to 0.50 g (1.5 mmol) of **66** in 15 ml of THF at -10°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from acetonitrile/diethylether 0.54 g (69 %), as colourless crystals. Elemental analysis for $C_{23}H_{34}N_6O_6S$ (522.62 g/mol): found (calc.) C 52.43 (52.86), H 6.24 (6.56), N 16.50 (16.08) %.
^{1}H-NMR ($CDCl_3$):

δ = 3.15 [s, 12 H, 1,3-Me$_B$]
δ = 1.49 [d, 12 H, 1,3-CH*Me$_2$*, ^{3}J= 7.8 Hz]
δ = 2.17 [s, 6 H, 4,5-Me]
δ = 4.59 [sept, 2 H, C*H*Me$_2$]
δ = 10.06 [s, 1 H, 2-H$_{Im}$]
Not observed [s, 1H, OH$_B$]

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

δ = 29.2 [1,3-Me$_B$]
δ = 66.2 [C5$_B$]
δ = 68.4 [C5$_{B\text{-}OH}$]
δ = 152.7 [C2$_B$]
δ = 165.9 [C4,6$_B$]
δ = 9. 4 [4,5-Me]
δ = 22.7 [1,3-CH*Me$_2$*]
δ = 51.9 [1,3-*C*HMe$_2$]
δ = 125.9 [C4,5]

δ = 135.8 [C2]

4.5.33 Preparation of di(1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium) 5-[(1,3-dimethyl-6-oxido-2,4-dioxo-1,2,3,4-tetrahydro-5-pyrimidinyl)sulfanyl]-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate **(68)**

A solution of 0.62 g (3.4 mmol) of 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene in 5 ml Et_2O was added to 0.58 g (1.7 mmol) of **66** in 15 ml of THF at -10°C. After the reaction mixture was stirred over night the precipitate was filtered off and dried in vacuo. Yield after recrystallisation from dichloromethane/diethylether 1.02 g (86 %), as colourless crystals; m.p. 164°C. Elemental analysis for $C_{34}H_{54}N_6O_6S$ (702.91 g/mol): found (calc.) C 58.43 (58.10), H 7.45 (7.74), N 15.98 (15.94) %.

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

δ = 3.12 [s, 12 H, 1,3-Me_B]

δ = 1.52 [d, 12 H, 1,3-CH*Me*$_2$, ^{3}J= 8.0 Hz]

δ = 2.18 [s, 6 H, 4,5-Me]

δ = 4.80 [sept, 2 H, C*H*Me_2]

δ = 10.64 [s, 1 H, 2-H_{Im}]

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

δ = 27.4 [1,3-Me_B]

δ = 81.0 [$C5_B$]

δ = 153.7 [$C2_B$]

δ = 163.1 [$C4,6_B$]

δ = 8.7 [4,5-Me]

δ = 27.4 [1,3-CH*Me*$_2$]

δ = 51.2 [1,3-*C*HMe_2]

δ = 124.4 [C4,5]

δ = 137.3 [C2]

4.5.34 Preparation of 1,3-dimethyl-2,6-dioxo-5-[(triphenylphosphonio)sulfanyl]-1,2,3,6-tetrahydro-4-pyrimidinolate **(69)**

A solution of 0.62 g (2.4 mmol) of PPh_3 in 15 ml CH_2Cl_2 was added to a suspension solution of 0.82 g (1.5 mmol) of **66** in 15 ml of CH_2Cl_2 at r.t., after the reaction mixture was stirred over night, solvent was removed in vacuo at r.t. to dryness. The residue was recrystallised from dichloromethane/diethylether to give 0.49 g (73 %), as green crystals; m.p. 157°C. Elemental analysis for $C_{24}H_{21}N_2O_3PS$ (448.48 g/mol): found (calc.) C 64.43 (64.27), H 4.44 (4.72), N 6.68 (6.25) %.

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

δ = 3.09	[s, 6 H, 1,3-Me_B]
δ = 7.50-7.85	[m, 15 H, Ph]

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

δ = 28.6	[1,3-Me_B]
δ = 60.8	[$C5_B$]
δ = 153.3	[$C2_B$]
δ = 165.0	[$C4,6_B$]
δ = 122.0	[d, $C1_{Ph,}$ ^1J = 75.4 Hz]
δ = 129.8	[$C3,5_{Ph}$, ^1J = 12.5 Hz]
δ = 134.8	[$C2,6_{Ph}$, ^1J = 6.3 Hz]
δ = not observed	[C4 Ph]

^{31}P-NMR ($CDCl_3$):

δ = 46.70	[$SPPh_3$]

5. Summary

Objectives of the present work were the synthesis of different types of imidazol-2-ylidene derivatives (scheme 1-2) and synthesis of different types of 1,3-dimethylbarbituric acid derivatives (scheme 3-4).

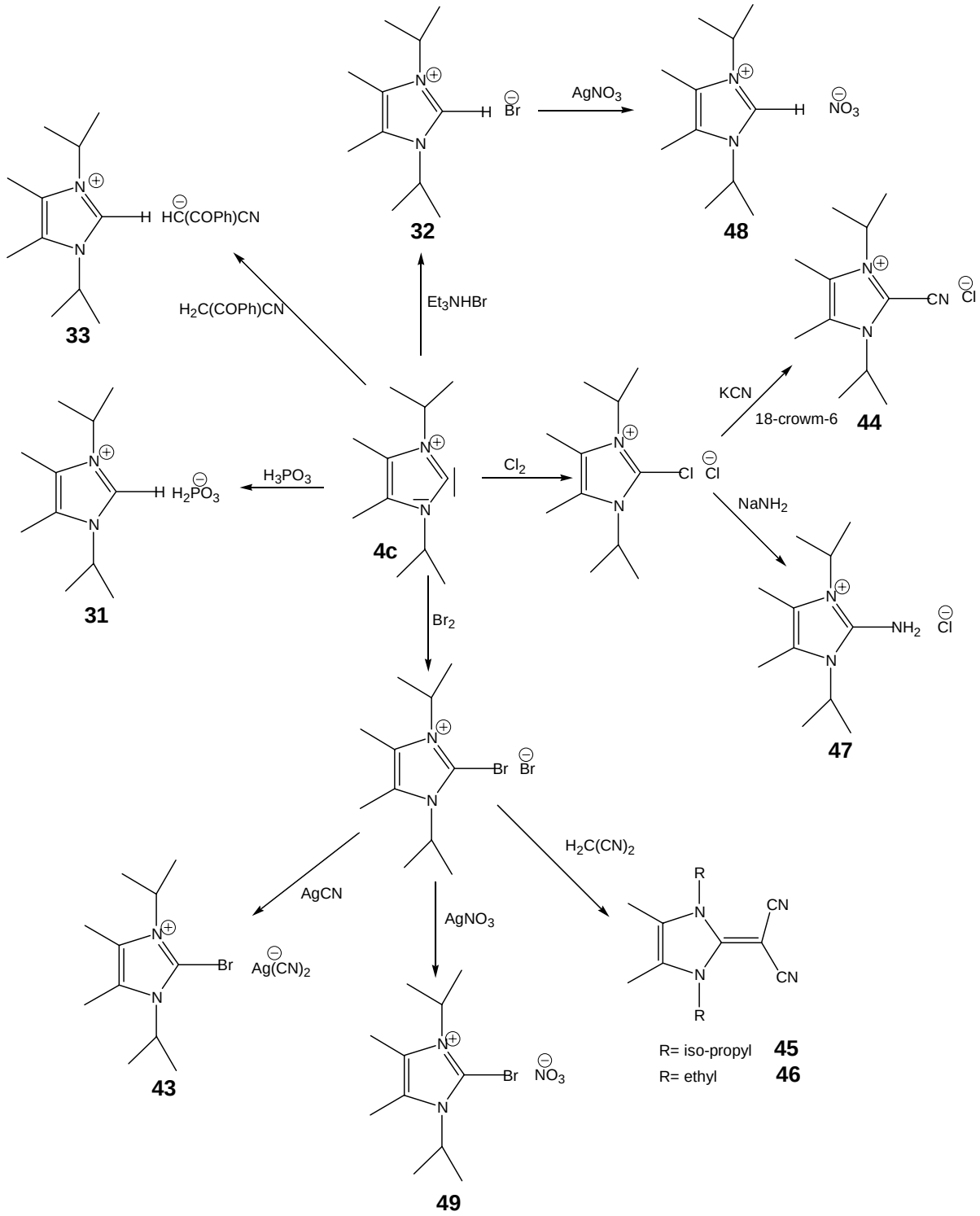
The resulting derivatives of imidazol-2-ylidene are classified into salts and neutral compounds. The salts **31, 32, 33, 35, 36, 37, 39, 42, 43, 44, 47, 48** and **49** were obtained by the nucleophilic reactions of the imidazol-2-ylidene **4c, 34 and 2** and substitution reactions of 2-halo-imidazolium salts.

The neutral compound **41** was obtained by the nucleophilic reaction of the 1,3-diadamantyl imidazol-2-ylidene **2** with sulfur, while compounds **45** and **46** were obtained by reaction of 2-bromoimidazolium bromide with malononitrile in the presence of base.

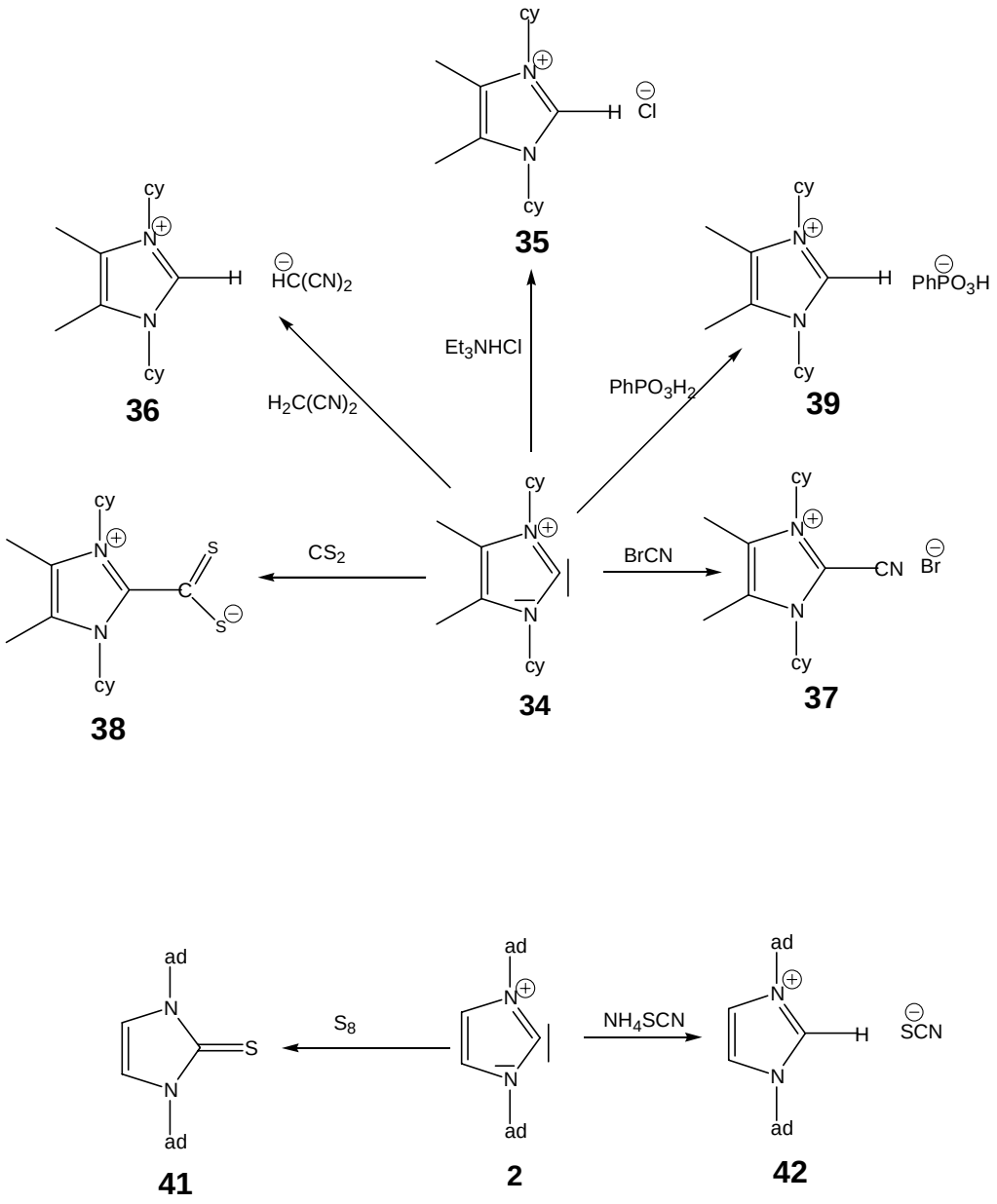
The resulting derivatives of 1,3-dimethylbarbituric acid are classified into salts and ylides and neutral compounds. The salts **56, 62, 64, 67** and **68** were obtained by reaction of 1,3-dimethylbarbituric acid **13** and derivatives of 1,3-dimethylbarbituric acid with strong ylide type bases.

The ylide compounds **51** and **52** were obtained by nucleophilic attack at the methylene 1,3-dimethylbarbituric acid **13**.

The neutral compounds **55, 57, 60, 61** and **65** were obtained by using 5,5-dihalo-1,3-dimethylbarbituric acid as starting material.

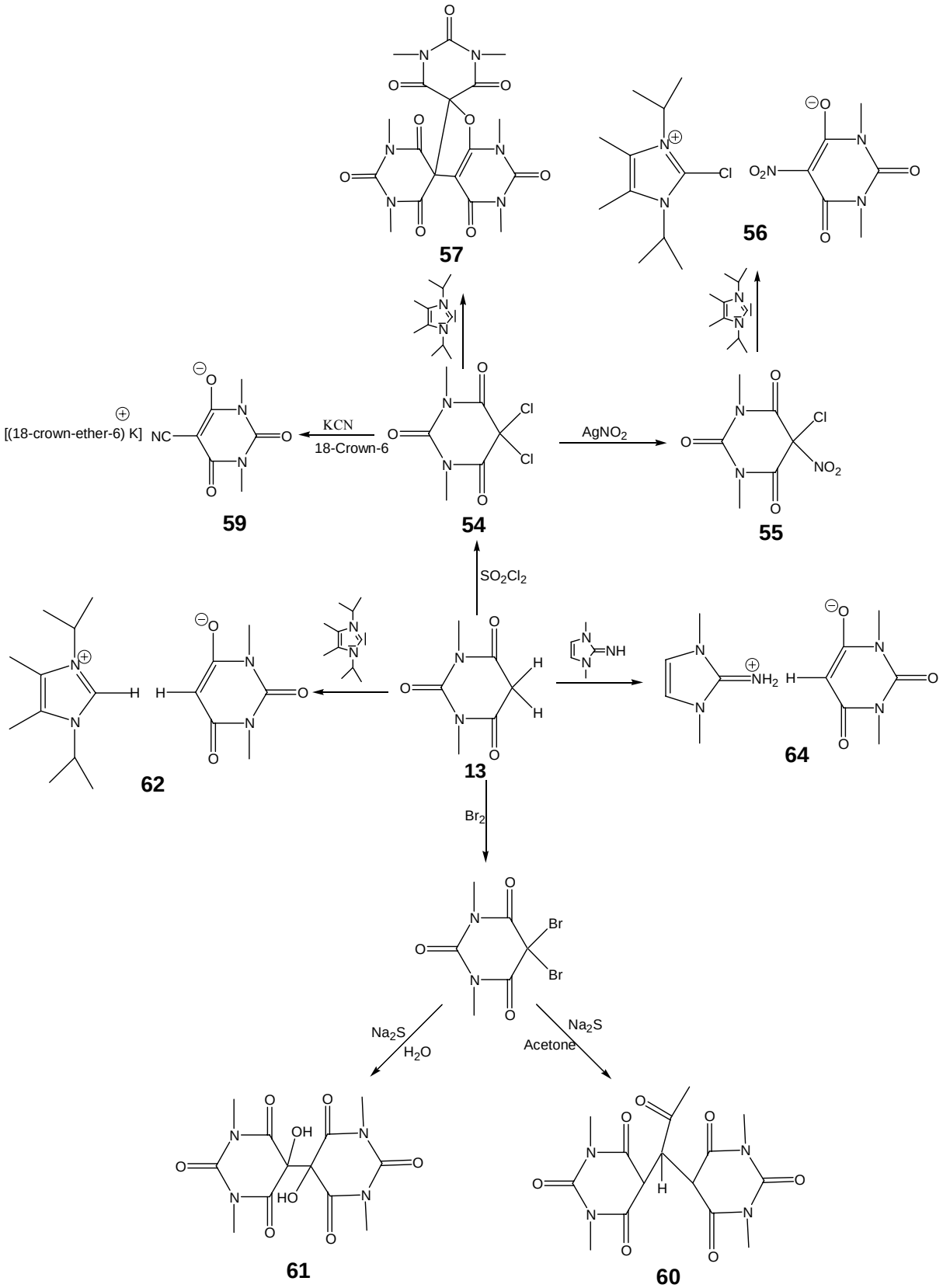

AgNO3
Br
NO3
32
48
HC(COPh)CN
33
H2C(COPh)CN
Et3NHBr
CN
Cl
KCN
18-crowm-6
44
H3PO3
H2PO3
31
4c
Cl2
Cl
NaNH2
NH2
47
Br2
Br
H2C(CN)2
AgCN
AgNO3
Ag(CN)2
43
49
R= iso-propyl
45
R= ethyl
46

Scheme 1

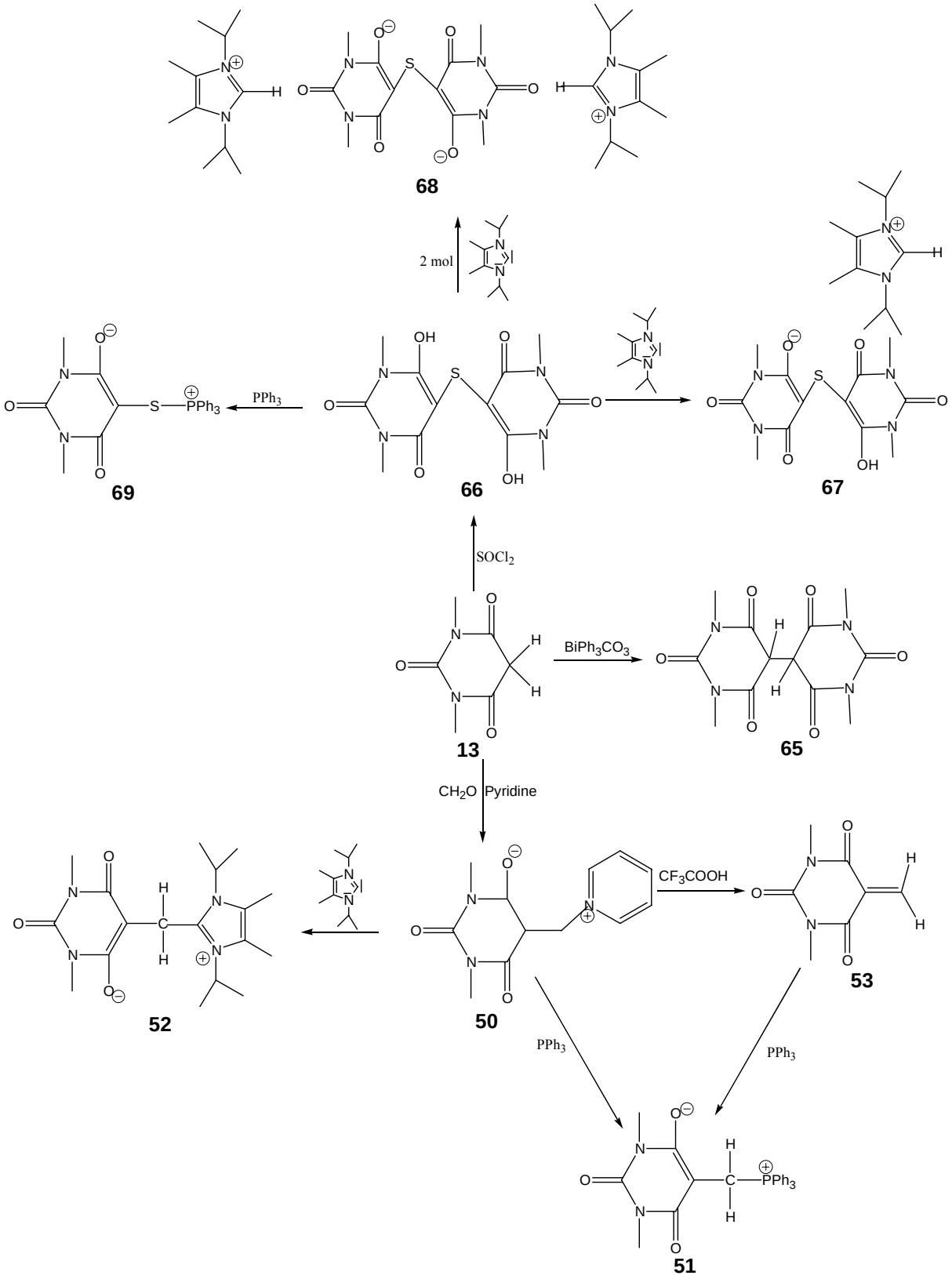

cy
N
H
Cl
N
cy
35
cy
N
H
HC(CN)2
N
cy
36
H2C(CN)2
Et3NHCl
PhPO3H2
cy
N
H
PhPO3H
N
cy
39
cy
N
S
C
S
N
cy
38
CS2
cy
N
N
cy
34
BrCN
cy
N
CN
Br
N
cy
37

ad
N
S
N
ad
41
S8
ad
N
N
ad
2
NH4SCN
ad
N
H
N
ad
SCN
42

Scheme 2


57
56
[(18-crown-ether-6) K]
KCN
18-Crown-6
AgNO2
59
54
55
SO2Cl2
62
13
64
Br2
Na2S
H2O
Na2S
Acetone
61
60

Scheme 3


68
2 mol
69
PPh3
66
67
SOCl2
13
BiPh3CO3
65
CH2O Pyridine
52
50
CF3COOH
53
PPh3
PPh3
51

Scheme 4

6. References

[1] A. J. Arduengo III, V. R. Dias, R. L. Harlow, M. Kleine, J. Am. Chem. Soc. 1992, 114, 5530.

[2] A. J. Arduengo III, R. L. Harlow, M. Kleine, J. Am. Chem. Soc. 1991, 113, 361.

[3] N. Kuhn, T. Kratz, Synthesis 1993, 561.

[4] N. Kuhn, A. Al-Sheikh, Coord. Chem. Rev. 2005, 24, 829.

[5] N. Kuhn, H. Bohnen, G. Henkel, Z. Naturforsch. 1994, 49b, 1473.

[6] N. Kuhn, G. Weyers, G. Henkel, Chem. Commun. 1997, 627.

[7] N. Kuhn, G. Weyers, S. Dümmling, B. Speiser, Phosphorus, Sulfur and Silicon 1997, 128, 45.

[8] N. Kuhn, K. Eichele, M. Walker, T. Berends, R. Minkwit, Z. Anorg. Allg. Chem. 2002, 2026.

[9] M. Tamm, S. Randoll, T. Bannenberg, E. Herdtweck, Chem. Sommun. 2004, 876.

[10] B. S. Jursic, D. E. Stevens, J. Hetero. C. 2003, 40, 701.

[11] H. Biltz, H. Wittek, Ber. 1921, 54B, 1035.

[12] J. W. Clark-Lewis, M. J. Thompson, J. Am. Chem. Soc. 1959, 1628.

[13] F. W. Smyth, T. Jenkins, J. Siekiera, A. Baydar, Analytica Chimica Acta 1975, 80, 233.

[14] M. V. Jovanovic, R. E. Biehl, J. Hetero. C. 1987, 24, 191.

[15] A. J. Barnes, L. Le Gall, J. Lauransan, Journal of Molecular Structure 1979, 56, 15.

[16] W. Bolton, Acta Cryst. 1963, 16, 950.

[17] B. S. Jursic, D. M. Neumann, K. L. Bowdy, E. D. Stevens, J. Hetero. C. 2004, 41, 233.

[18] A. V. Moskvin, N. R. Reznikova, B. A. Ivin, Russian Journal of Organic Chemistry (Translation of Zhurnal Organicheskoi Khimii) 2002, 38, 463.

[19] G. Alcerreca, R. Sanabria, R. Miranda, G. Arroyo, J. Tamariz, F. Delgado, Synthetic Communications 2000, 30, 1295.

[20] N. Shoji, Y. Kondo, T. Takemoto, Chemical & Pharmaceutical Bulletin 1973, 21, 2639.

[21] A. M. Arrett, M. A. Stockham, Journal of Endocrinology 1965, 33, 145.

[22] N. Kuhn, G. Bohnen, T. Kratz, Chem. Ber. 1994, 127, 849.

[23] N. Kuhn, G. Henkel, T. Kratz, J. Kreutzberg, R Boese, A. H. Maulitz, Chem. Ber. 1993, 126, 2041.

[24] N. Kuhn, H. Bohnen, J. Fahl, D. Bläser, Chem. Ber. 1996, 129, 1579.

[25] M. Johes Jr, W. Ando, M. E. Hndrick, A. Kulczycki jun., P. M. Howley, K. F. Hummel, D. S. Malament, J. Am. Chem. Soc. 1972, 94, 7469.

[26] N. Kuhn, K. Sweidan, Unpublished data.

[27] S. Furberg, P. Landamark, Acta Chem. Scand. II 1957, 9, 1505-1511.

[28] M. Steimann, Personal Communication.

[29] N. Kuhn, J. Fahl, R. Fawzi, C. Maichle-Mössmer, M. Steimann, Z. Naturforsh. 1998, 53b, 720.

[30] N. Kuhn, K. Sweidan, Unpublished data.

[31] N. Kuhn, K. Sweidan, Unpublished data.

[32] N. Kuhn, H. Bohnen, G. Hekel, Z. Kristall. 1996, 211, 57; N. Kuhn, R. Boese, D. Bläser, Z. Kristall. 1994, 209, 837.

[33] M. Steimann, Personal Communication.

[34] N. Kuhn, K. Sweidan, Unpublished data.

[35] N. Kuhn, K. Sweidan, Unpublished data.

[36] N. Kuhn, E. Niquet, M. Steimann, I. Walker, Z. Naturforsh. 1999, 54, 1181.

[37] N. Kuhn, K. Sweidan, Unpublished data.

[38] T. J. Weakley, Acta Cryst. 1976, B32, 2889.

[39] N. Kuhn, K. Sweidan, Unpublished data.

[40] G. B.Ansell, J. Chem. Soc. Perkin Trans. 2 1972, 7, 841.

[41] J. Huang, H-J. Schanz, E. D. Stevens, S. P. Nolan, K. B. Capps, A. Bauer, C. D. Hoff, Inorg. Chem. 2000, 39, 1042.

[42] N. Kuhn, K. Sweidan, Unpublished data.

[43] J. M. Ringle, J. Golding, C. M. Forsyth, G. B. Deacon, M. Forsyth, D. R. MacFarlane, J. Mater. Chem. 2002, 12, 3475.

[44] N. Kuhn, A. Abu-Rayyan, K. Eichele, S. Schwarz, M. Steimann, Inorg. Chim. Acta. 2004, 357, 1799.

[45] N. Kuhn, K. Sweidan, Unpublished data.

[46] T. Soma, T. Iwamoto, Inorg. Chem. 1996, 35, 1849.

[47] N. Kuhn, M. Walker, Unpublished data.

[48] N. Kuhn, A. Abu-Rayyan, M. Göhner, M. Steimann, Z. Anorg. Allg. Chem. 2002, 628, 1721.

[49] N. Kuhn, K. Sweidan, Unpublished data.

[50] M. Steimann, Personal Communication.

[51] N. Kuhn, K. Sweidan, Unpublished data.

[52] M. Steimann, Personal Communication.

[53] P. J. Langley, J. Hulliger, R. Thaimattam, G. R. Desiraju, New J. Chem. 1998, 1307.

[54] M. Z. Ebrahimi, G. W. Huffiman, Synthesis 1996, 215.

[55] M. Steimann, Personal Communication.

[56] N. Kuhn, A. Al-Sheikh, S. Schwarz, M. Steimann, Z. Naturforsh. 2005, 60, 398.

[57] V. Bertolasi, P. Gilli, V. Ferretti, New J. Chem. 2001, 25, 408.

[58] M. Steimann, Personal Communication.

[59] N. Kuhn, K. Sweidan, Unpublished data.

[60] N. Kuhn, K. Sweidan, Unpublished data.

[61] D. D. DesMarteau, W. T. Pennington, G. Resnati, Acta Cryst. 1994, C50, 1305.

[62] N. Kuhn, K. Sweidan, Unpublished data.

[63] N. Kuhn, K. Sweidan, Unpublished data.

[64] M. Poling, D. Van der Helm, Acta Cryst. 1976, B32, 3349.

[65] N. Kuhn, K. Sweidan, Unpublished data.

[66] H. R. Snyder, C. W. Kurse, J. Am. Chem. Soc. 1958, 80, 1942.

[67] N. Kuhn, K. Sweidan, Unpublished data.

[68] N. Kuhn, K. Sweidan, Unpublished data.

[69] N. Kuhn, R. Fawzi, M. Steimann, J. Wiethoff, D. Bläser, R. Boese, Z. Naturforsch. 1995, 50b, 1779.

[70] D. H. R. Barton, N. Y. Bhatnagar, Blazejewski, C. Jean, B. Charpiot, J. P. Finet, D. J. Lester, W. B. Motherwell, M. T. Papoula, S. P. Stanforth, J. Chem. Soc. Perkin Trans. 1 1985, 12, 2657.

[71] N. Kuhn, K. Sweidan, Unpublished data.

[72] N. Kuhn, K. Sweidan, Unpublished data.

[73] N. Kuhn, A. Al-Sheikh, C. Maichle-Moessmer, M. Steimann, M. Stroebelle, Z. Anorg. Allg. Chem. 2004, 630, 1659.

[74] N. Kuhn, K. Sweidan, Unpublished data.

[75] N. Kuhn, K. Sweidan, Unpublished data.

[76] N. Kuhn, K. Sweidan, Unpublished data.

7. Appendix

7.1 Abbreviations and definitions

Å	Angstrom (10^{-10})
Ad	Adamantly
δ	Chemical shift
°	Degree
B	1,3-Dimethylbarbituric acid
Cat.	Catalytic
Calc.	Calculated
CH_2Cl_2	Dichloromethane
Cy	Cyclohexyl
DMSO	Dimethylsulfoxide
EI	Electron ionization (mass spectroscopy)
Et	Ethyl
Et_2O	Diethyl ether
eV	Electron Volt
FAB	Fast atom bombardment (mass spectroscopy)
FD	Field desorption
Fig.	Figure
FT	Fourier transformation
g	Grams
h	Hour
Hz	Hertz
Im	Imidazole
i-Pr	Isopropyl
IR	Infrared spectroscopy
Me	Methyl
MeCN	Acetonitrile
Mes	Mesityl
m.p.	Melting point
ml	Milliliter
mmol	Millimol

MS	Mass spectroscopy
NMR	Nuclear magnetic resonance
PPh_3	Triphenylphosphine
Ph_3BiCO_3	Triphenylbismuth(III) carbonate
Ph	Phenyl
ppm	Parts per million
Py	Pyridine
r.t	Room temperature
Tab.	Table
THF	Tetrahydrofuran
TMS	Tetramethylsilane
tBu	Tert-butyl
ν	Frequency

7.2 Numbering of the compounds

1 1,3-Diadamantylimidazolium chloride

2 1,3-Diadamantylimidazol-2-ylidene

3a 1,3,4,5-Tetramethylimidazol-2-thione

3b 1,3-Diethyl-4,5-dimethylimidazol-2-thione

3c 1,3-Diisopropyl-4,5-dimethylimidazol-2-thione

4a 1,3,4,5-Tetramethylimidazol-2-ylidene

4b 1,3-Diethyl-4,5-dimethylimidazol-2-ylidene

4c 1,3-Diisopropyl-4,5-dimethylimidazol-2-ylidene

5a 1,3,4,5-tetramethyl-1H-imidazol-3-ium-2-carbodithioate

5b 1,3-Diethyl-4,5-dimethyl-1H-imidazol-3-ium-2-carbodithioate

5c 1,3-Diisopropyl-4,5-dimethyl-1H-imidazol-3-ium-2-carbodithioate

6a Dipotassium (1,3,4,5-tetramethyl-1,3-dihydro-2H-imidazol-2-ylidene)methanedithiolate

6b Dipotassium (1,3-diethyl-4,5-dimethyl-1,3-dihydro-2H-imidazol-2-ylidene)methanedithiolate

6c Dipotassium (1,3-diisopropyl-4,5-dimethyl-1,3-dihydro-2H-imidazol-2-ylidene)methanedithiolate

7a 2-[Bis(methylsulfanyl)methylene]-1,3,4,5-tetramethyl-2,3-dihydro-1H-imidazole

7b 2-[Bis(methylsulfanyl)methylene]-1,3-diethyl-4,5-dimethyl-2,3-dihydro-1H-imidazole

7c 2-[Bis(methylsulfanyl)methylene]-1,3-diisopropyl-4,5-dimethyl-2,3-dihydro-1H-imidazole

8 2-(Dichlorophosphoryl)-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium chloride

9 Chloro(1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium-2-yl)oxophosphoranolate

10 1,3-Diisopropyl-4,5-dimethyl-1H-imidazol-3-ium-2-ylphosphonate

11 1,3-Di-tert-butylimidazolin-2-ylidene

12 N-Silylated 2-iminoimidazoline

13 1,3-Dimethylbarbituric acid

14 1,3-Dimethyl-5-(1-methylethylidene)-2,4,6(1H,3H,5H)-pyrimidinetrione

15 1,3-Dimethyl-5-(1-phenylethylidene)-2,4,6(1H,3H,5H)-pyrimidinetrione

16 4-Methylene-2-oxetanone

17 6-Hydroxy-1,3-dimethyl-5-pyruvoyl-2,4(1H,3H)-pyrimidinedione

18 6-Hydroxy-1,3-dimethyl-5-(3-methyl-1H-pyrazol-5-yl)-2,4(1H,3H)-pyrimidinedione

19 6-Hydroxy-1,3-dimethyl-5-nitroso-2,4(1H,3H)-pyrimidinedione

20 6-Hydroxy-1,3-dimethyl-5-nitro-2,4(1H,3H)-pyrimidinedione

21 Imidazol-2-ylidene

22 Imidazol-2-ylidene adduct

23 2-Haloimidazolium salts

24 1,3-Dimethyl-2,4,6-(1H, 3H, 5H)-pyrimidinetrione-5-ylidene

25 1,3-Dimethylbarbituric's zwitterionic

26a 1,3-Dialkyl (dicyclic)-4,5-dimethylimidazolium salt

26b 1,3-Dialkyl (dicyclic)-4,5-dimethylimidazolium salt

26c 1,3-Diadamantylimidazolium salts

27 1,3-Dimethylbarbituric's zwitterionic

28 1,3-Dimethylbarbiturate e salts

29 5,5-Dihalo-1,3-Dimethyl-2,4,6-(1H, 3H, 5H)-pyrimidinetrione

30 Dimer of barbituric acid derivatives

31 1,3-Diisopropyl-4,5-dimethyl-1H-imidazol-3-ium phosphonate

32 1,3-Diisopropyl-4,5-dimethyl-1H-imidazol-3-ium bromide

33 1,3-Diisopropyl-4,5-dimethyl-1H-imidazol-3-ium(*Z*)-2-cyano-1-phenyl-1-ethenolate

35 1,3-Dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium chloride

34 1,3-Dicyclohexyl-4,5-dimethylimidazol-2-ylidene

36 1,3-Dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium dicyanomethylide

37 2-Cyano-1,3-dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium bromide

38 1,3-Dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium-2-carbodithioate

39 1,3-Dicyclohexyl-4,5-dimethyl-1H-imidazol-3-ium phenylphosphonate

40 Ammonium thiocyanat

41 1,3-Di(1-adamantyl)-1,3-dihydro-2H-imidazole-2-thione

42 1,3-Di(1-adamantyl)-1H-imidazol-3-ium thiocyanate

43 2-Bromo-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium dicyanoargentate

44 2-Cyano-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium chloride

45 2-(1,3-Diisopropyl-4,5-dimethyl-1,3-dihydro-2H-imidazol-2-ylidene) malononitrile

46 2-(1,3-Diethyl-4,5-dimethyl-1,3-dihydro-2H-imidazol-2-ylidene) malononitrile

47 2-Amino-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium chloride

48 1,3-Diisopropyl-4,5-dimethyl-1H-imidazol-3-ium nitrate

49 2-Bromo-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-iumnitrate

50 1,3-Dimethyl-2,6-dioxo-5-(1-pyridiniumylmethyl)-1,2,3,6-tetrahydro-4-pyrimidinolate.

51 1,3-Dimethyl-2,6-dioxo-5-[(triphenylphosphonio)methyl]-1,2,3,6-tetrahydro-4-pyrimidinolate

52 5-[(1,3-Diisopropyl-4,5-dimethyl-1H-imidazol-3-ium-2-yl)methyl]-1,3- dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate

53 1,3-Dimethyl-5-methylene-2,4,6(1H,3H,5H)-pyrimidinetrione

54 5,5-Dichloro-1,3-dimethyl-2,4,6(1H,3H,5H)-pyrimidinetrione pyrimidinolate

55 5-Chloro-1,3-dimethyl-5-nitro-2,4,6(1H,3H,5H)-pyrimidinetrione

56 2-Chloro-1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium 1,3-dimethyl-5-nitro-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate

57 5,6-Dihydro-1,3-dimethyl-5,6-bis-[1',3'-dimethyl]-2',4',6'-trioxopyrimid (5',5') yl[2,3- d] uracil

58 1,3-Dimethyl-2,4,6-trioxotetrahydro-5,5(2H)-pyrimidinedicarbonitrile

59 Potassium 5-cyano-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate 1,4,7,10,13,16-hexaoxacyclooctadecane

60 5-[1-(1,3-dimethyl-2,4,6-trioxohexahydro-5-pyrimidinyl)-2-oxopropyl]-1,3-dimethyl-2,4,6(1H,3H,5H)-pyrimidinetrione

61 [5,5'-Bipyrimidine]-2,2',4,4',6,6'(1H,1'H,3H,3'H,5H,5'H)-hexone, 5,5'-dihydroxy-1,1',3,3'-tetramethyl

62 1,3-Diisopropyl-4,5-dimethyl-1H-imidazol-3-ium 1,3-dimethyl-2,6- dioxo-tetrahydro-4-pyrimidinolate

63 2-Imino-1,3-dimethylimidazoline

64 1,3-Dimethyl-1,3-dihydro-2H-imidazol-2-iminium 1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate

65 [5,5'-Bipyrimidine]-2,2',4,4',6,6'(1H,1'H,3H,3'H,5H,5'H)-hexone, 1,1',3,3'-tetramethyl

66 2,6-Hydroxy-5-[(6-hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydro-5-pyrimidinyl)sulfanyl]-1,3-dimethyl-2,4(1H,3H)-pyrimidinedione

67 1,3-Diisopropyl-4,5-dimethyl-1H-imidazol-3-ium 5-[(6-hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydro-5-pyrimidinyl)sulfanyl]-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate

68 Di(1,3-diisopropyl-4,5-dimethyl-1H-imidazol-3-ium) 5-[(1,3-dimethyl-6-oxido-2,4-dioxo-1,2,3,4-tetrahydro-5-pyrimidinyl)sulfanyl]-1,3- dimethyl-2,6-dioxo-1,2,3,6-tetrahydro-4-pyrimidinolate

69 1,3-Dimethyl-2,6-dioxo-5-[(triphenylphosphonio)sulfanyl]-1,2,3,6-tetrahydro-4-pyrimidinolate

7.3 Crystal structure's data

7.3.1 Crystal data for $C_{11}H_{23}N_2O_3P$ **(31)**

Table 1: Crystal data and structure refinement for $C_{11}H_{23}N_2O_3P$ **(31)**

Empirical formula	$C_{11}H_{23}N_2O_3P$	
Formula weight	262.28	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 14.123(2) Å	α= 90°.
	b = 11.577(4) Å	β= 100.27(1)°.
	c = 18.192 (1) Å	γ = 90°.
Volume	2926.9(12) $Å^3$	
Z	8	
Density (calculated)	1.190 Mg/m^3	
Absorption coefficient	0.188 mm^{-1}	
F(000)	1136	
Crystal size	0.75 x 0.50 x 0.40 mm^3	
Theta range for data collection	5.19 to 23.26°.	
Index ranges	-1<=h<=15, -1<=k<=12, -20<=l<=20	
Reflections collected	5241	
Independent reflections	4162 [R(int) = 0.0376]	
Completeness to theta = 23.26°	98.8 %	
Max. and min. transmission	0.9286 and 0.8719	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4162 / 0 / 332	
Goodness-of-fit on F^2	1.026	
Final R indices [I>2sigma(I)]	R1 = 0.0609, wR2 = 0.1674	
R indices (all data)	R1 = 0.0811, wR2 = 0.1826	
Extinction coefficient	0.0013(12)	
Largest diff. peak and hole	0.466 and -0.389 $e.Å^{-3}$	

Table 2: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{11}H_{23}N_2O_3P$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
C(1)	1517(2)	3513(3)	869(2)	37(1)
C(2)	1239(2)	3474(3)	114(2)	38(1)
C(3)	233(3)	2393(3)	620(2)	37(1)
C(4)	2356(3)	4086(4)	1334(2)	54(1)
C(5)	1666(3)	4043(4)	-486(2)	58(1)
C(6)	841(3)	2687(3)	1977(2)	42(1)
C(7)	-13(3)	3350(4)	2147(2)	62(1)
C(8)	828(3)	1420(4)	2167(2)	57(1)
C(9)	-133(3)	2476(3)	-772(2)	44(1)
C(10)	-891(4)	3382(5)	-990(3)	83(2)
C(11)	-530(3)	1279(4)	-780(2)	58(1)
N(1)	439(2)	2759(2)	-27(1)	34(1)
N(2)	878(2)	2833(2)	1168(2)	36(1)
C(12)	3631(2)	6813(3)	6131(2)	38(1)
C(13)	3715(2)	7279(3)	6825(2)	36(1)
C(14)	3642(3)	5386(3)	6935(2)	42(1)
C(15)	3593(3)	7385(3)	5392(2)	52(1)
C(16)	3836(3)	8502(3)	7077(2)	50(1)
C(17)	3523(3)	4753(3)	5609(2)	44(1)
C(18)	4405(4)	3997(7)	5733(4)	119(3)
C(19)	2639(4)	4047(4)	5572(3)	75(1)
C(20)	3777(3)	6468(3)	8138(2)	43(1)
C(21)	4701(4)	5941(5)	8525(2)	80(2)
C(22)	2899(4)	5920(5)	8364(3)	80(2)
N(3)	3712(2)	6366(2)	7315(2)	38(1)
N(4)	3587(2)	5623(2)	6212(2)	38(1)
P(1)	6397(1)	7715(1)	1773(1)	47(1)
O(11)	6031(2)	8968(3)	1846(2)	56(1)
O(12)	7379(2)	7736(3)	1558(2)	66(1)
O(13)	6296(3)	7035(3)	2435(2)	74(1)
P(2)	7981(1)	657(1)	1365(1)	52(1)
O(21)	6907(2)	602(2)	1271(2)	49(1)
O(22)	8400(2)	1406(3)	853(2)	67(1)
O(23)	8418(3)	-571(4)	1370(3)	94(2)

Table 3: Bond lengths [Å] and angles [°] for $C_{11}H_{23}N_2O_3P$

C(1)-C(2)	1.360(5)	C(14)-N(3)	1.323(5)
C(1)-N(2)	1.380(4)	C(14)-N(4)	1.332(5)
C(1)-C(4)	1.484(5)	C(14)-H(14)	0.91(4)
C(2)-N(1)	1.386(4)	C(15)-H(15A)	0.9700
C(2)-C(5)	1.492(5)	C(15)-H(15B)	0.9700
C(3)-N(2)	1.325(4)	C(15)-H(15C)	0.9700
C(3)-N(1)	1.331(4)	C(16)-H(16A)	0.9700
C(3)-H(3)	0.97(4)	C(16)-H(16B)	0.9700
C(4)-H(4A)	0.9700	C(16)-H(16C)	0.9700
C(4)-H(4B)	0.9700	C(17)-N(4)	1.480(4)
C(4)-H(4C)	0.9700	C(17)-C(19)	1.484(6)
C(5)-H(5A)	0.9700	C(17)-C(18)	1.505(7)
C(5)-H(5B)	0.9700	C(17)-H(17)	0.9900
C(5)-H(5C)	0.9700	C(18)-H(18A)	0.9700
C(6)-N(2)	1.491(4)	C(18)-H(18B)	0.9700
C(6)-C(8)	1.508(5)	C(18)-H(18C)	0.9700
C(6)-C(7)	1.508(6)	C(19)-H(19A)	0.9700
C(6)-H(6)	0.9900	C(19)-H(19B)	0.9700
C(7)-H(7A)	0.9700	C(19)-H(19C)	0.9700
C(7)-H(7B)	0.9700	C(20)-N(3)	1.489(4)
C(7)-H(7C)	0.9700	C(20)-C(21)	1.498(6)
C(8)-H(8A)	0.9700	C(20)-C(22)	1.512(6)
C(8)-H(8B)	0.9700	C(20)-H(20)	0.9900
C(8)-H(8C)	0.9700	C(21)-H(21A)	0.9700
C(9)-N(1)	1.485(4)	C(21)-H(21B)	0.9700
C(9)-C(11)	1.495(5)	C(21)-H(21C)	0.9700
C(9)-C(10)	1.501(6)	C(22)-H(22A)	0.9700
C(9)-H(9)	0.9900	C(22)-H(22B)	0.9700
C(10)-H(10A)	0.9700	C(22)-H(22C)	0.9700
C(10)-H(10B)	0.9700	P(1)-O(13)	1.467(3)
C(10)-H(10C)	0.9700	P(1)-O(12)	1.506(3)
C(11)-H(11A)	0.9700	P(1)-O(11)	1.554(3)
C(11)-H(11B)	0.9700	P(1)-H(1)	1.27(4)
C(11)-H(11C)	0.9700	O(11)-H(11)	0.83(6)
C(12)-C(13)	1.358(5)	P(2)-O(22)	1.473(3)
C(12)-N(4)	1.388(4)	P(2)-O(21)	1.497(3)
C(12)-C(15)	1.491(5)	P(2)-O(23)	1.550(5)
C(13)-N(3)	1.383(4)	P(2)-H(2)	1.38(5)
C(13)-C(16)	1.489(5)	O(23)-H(23)	0.72(4)

C(2)-C(1)-N(2)	106.7(3)	N(1)-C(9)-C(10)	109.5(3)
C(2)-C(1)-C(4)	130.4(3)	C(11)-C(9)-C(10)	113.3(4)
N(2)-C(1)-C(4)	122.8(3)	N(1)-C(9)-H(9)	107.7
C(1)-C(2)-N(1)	106.6(3)	C(11)-C(9)-H(9)	107.7
C(1)-C(2)-C(5)	130.0(3)	C(10)-C(9)-H(9)	107.7
N(1)-C(2)-C(5)	123.4(3)	C(9)-C(10)-H(10A)	109.5
N(2)-C(3)-N(1)	108.3(3)	C(9)-C(10)-H(10B)	109.5
N(2)-C(3)-H(3)	128(2)	H(10A)-C(10)-H(10B)	109.5
N(1)-C(3)-H(3)	123(2)	C(9)-C(10)-H(10C)	109.5
C(1)-C(4)-H(4A)	109.5	H(10A)-C(10)-H(10C)	109.5
C(1)-C(4)-H(4B)	109.5	H(10B)-C(10)-H(10C)	109.5
H(4A)-C(4)-H(4B)	109.5	C(9)-C(11)-H(11A)	109.5
C(1)-C(4)-H(4C)	109.5	C(9)-C(11)-H(11B)	109.5
H(4A)-C(4)-H(4C)	109.5	H(11A)-C(11)-H(11B)	109.5
H(4B)-C(4)-H(4C)	109.5	C(9)-C(11)-H(11C)	109.5
C(2)-C(5)-H(5A)	109.5	H(11A)-C(11)-H(11C)	109.5
C(2)-C(5)-H(5B)	109.5	H(11B)-C(11)-H(11C)	109.5
H(5A)-C(5)-H(5B)	109.5	C(3)-N(1)-C(2)	109.0(3)
C(2)-C(5)-H(5C)	109.5	C(3)-N(1)-C(9)	124.6(3)
H(5A)-C(5)-H(5C)	109.5	C(2)-N(1)-C(9)	126.4(3)
H(5B)-C(5)-H(5C)	109.5	C(3)-N(2)-C(1)	109.4(3)
N(2)-C(6)-C(8)	109.9(3)	C(3)-N(2)-C(6)	123.9(3)
N(2)-C(6)-C(7)	108.4(3)	C(1)-N(2)-C(6)	126.4(3)
C(8)-C(6)-C(7)	113.9(3)	C(13)-C(12)-N(4)	107.0(3)
N(2)-C(6)-H(6)	108.1	C(13)-C(12)-C(15)	130.1(3)
C(8)-C(6)-H(6)	108.1	N(4)-C(12)-C(15)	122.8(3)
C(7)-C(6)-H(6)	108.1	C(12)-C(13)-N(3)	106.6(3)
C(6)-C(7)-H(7A)	109.5	C(12)-C(13)-C(16)	130.5(3)
C(6)-C(7)-H(7B)	109.5	N(3)-C(13)-C(16)	122.8(3)
H(7A)-C(7)-H(7B)	109.5	N(3)-C(14)-N(4)	109.0(3)
C(6)-C(7)-H(7C)	109.5	N(3)-C(14)-H(14)	128(2)
H(7A)-C(7)-H(7C)	109.5	N(4)-C(14)-H(14)	123(2)
H(7B)-C(7)-H(7C)	109.5	C(12)-C(15)-H(15A)	109.5
C(6)-C(8)-H(8A)	109.5	C(12)-C(15)-H(15B)	109.5
C(6)-C(8)-H(8B)	109.5	H(15A)-C(15)-H(15B)	109.5
H(8A)-C(8)-H(8B)	109.5	C(12)-C(15)-H(15C)	109.5
C(6)-C(8)-H(8C)	109.5	H(15A)-C(15)-H(15C)	109.5
H(8A)-C(8)-H(8C)	109.5	H(15B)-C(15)-H(15C)	109.5
H(8B)-C(8)-H(8C)	109.5	C(13)-C(16)-H(16A)	109.5
N(1)-C(9)-C(11)	110.8(3)	C(13)-C(16)-H(16B)	109.5

H(16A)-C(16)-H(16B)	109.5	H(21A)-C(21)-H(21B)	109.5
C(13)-C(16)-H(16C)	109.5	C(20)-C(21)-H(21C)	109.5
H(16A)-C(16)-H(16C)	109.5	H(21A)-C(21)-H(21C)	109.5
H(16B)-C(16)-H(16C)	109.5	H(21B)-C(21)-H(21C)	109.5
N(4)-C(17)-C(19)	110.3(3)	C(20)-C(22)-H(22A)	109.5
N(4)-C(17)-C(18)	110.2(3)	C(20)-C(22)-H(22B)	109.5
C(19)-C(17)-C(18)	110.7(5)	H(22A)-C(22)-H(22B)	109.5
N(4)-C(17)-H(17)	108.5	C(20)-C(22)-H(22C)	109.5
C(19)-C(17)-H(17)	108.5	H(22A)-C(22)-H(22C)	109.5
C(18)-C(17)-H(17)	108.5	H(22B)-C(22)-H(22C)	109.5
C(17)-C(18)-H(18A)	109.5	C(14)-N(3)-C(13)	109.1(3)
C(17)-C(18)-H(18B)	109.5	C(14)-N(3)-C(20)	125.4(3)
H(18A)-C(18)-H(18B)	109.5	C(13)-N(3)-C(20)	125.6(3)
C(17)-C(18)-H(18C)	109.5	C(14)-N(4)-C(12)	108.3(3)
H(18A)-C(18)-H(18C)	109.5	C(14)-N(4)-C(17)	125.2(3)
H(18B)-C(18)-H(18C)	109.5	C(12)-N(4)-C(17)	126.5(3)
C(17)-C(19)-H(19A)	109.5	O(13)-P(1)-O(12)	116.78(19)
C(17)-C(19)-H(19B)	109.5	O(13)-P(1)-O(11)	110.28(19)
H(19A)-C(19)-H(19B)	109.5	O(12)-P(1)-O(11)	110.10(17)
C(17)-C(19)-H(19C)	109.5	O(13)-P(1)-H(1)	108.1(19)
H(19A)-C(19)-H(19C)	109.5	O(12)-P(1)-H(1)	107.2(19)
H(19B)-C(19)-H(19C)	109.5	O(11)-P(1)-H(1)	103.4(19)
N(3)-C(20)-C(21)	109.4(3)	P(1)-O(11)-H(11)	116(4)
N(3)-C(20)-C(22)	109.5(3)	O(22)-P(2)-O(21)	117.72(17)
C(21)-C(20)-C(22)	112.9(4)	O(22)-P(2)-O(23)	109.8(2)
N(3)-C(20)-H(20)	108.3	O(21)-P(2)-O(23)	110.8(2)
C(21)-C(20)-H(20)	108.3	O(22)-P(2)-H(2)	107(2)
C(22)-C(20)-H(20)	108.3	O(21)-P(2)-H(2)	107(2)
C(20)-C(21)-H(21A)	109.5	O(23)-P(2)-H(2)	104(2)
C(20)-C(21)-H(21B)	109.5	P(2)-O(23)-H(23)	118(4)

Symmetry transformations used to generate equivalent atoms:

7.3.2 Crystal data for $C_{22}H_{42}Br_2N_4O0$ **(32)**

Table 4: Crystal data and structure refinement for $C_{22}H_{42}Br_2N_4O0$ **(32)**

Empirical formula	$C_{22}H_{42}Br_2N_4O0$	
Formula weight	522.42	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	C222(1)	
Unit cell dimensions	a = 10.894(5) Å	α= 90°.
	b = 11.559(4) Å	β= 90°.
	c = 20.579(5) Å	γ= 90°.
Volume	2591.3(15) $Å^3$	
Z	4	
Density (calculated)	1.339 Mg/m^3	
Absorption coefficient	3.142 mm^{-1}	
F(000)	1088	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	2.57 to 27.50°.	
Index ranges	-14<=h<=0, -14<=k<=14, -26<=l<=26	
Reflections collected	6116	
Independent reflections	2985 [R(int) = 0.0904]	
Completeness to theta = 27.50°	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2985 / 0 / 136	
Goodness-of-fit on F^2	1.054	
Final R indices [I>2sigma(I)]	R1 = 0.0793, wR2 = 0.1849	
R indices (all data)	R1 = 0.0879, wR2 = 0.1943	
Absolute structure parameter	-0.02(2)	
Extinction coefficient	0.0033(9)	
Largest diff. peak and hole	2.112 and -2.021 $e.Å^{-3}$	

Table 5: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{22}H_{42}Br_2N_4O0$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Br(1)	0	4791(1)	2500	46(1)
Br(2)	342(1)	10000	0	40(1)
C(8)	108(5)	4633(4)	267(2)	34(1)
N(2)	1343(4)	4428(4)	418(2)	30(1)
C(2)	408(5)	318(4)	7764(2)	30(1)
N(1)	628(5)	1467(3)	7909(2)	29(1)
C(12)	2746(5)	4278(5)	1351(3)	39(1)
C(1)	0	2132(5)	7500	34(2)
C(7)	2041(6)	5000	0	30(1)
C(10)	1804(6)	3662(5)	937(2)	34(1)
C(3)	958(7)	-657(5)	8126(3)	43(1)
C(4)	1466(6)	1916(5)	8415(3)	34(1)
C(11)	2321(10)	2559(6)	627(3)	53(2)
C(6)	773(7)	2741(5)	8863(2)	45(2)
C(9)	-935(6)	4152(7)	636(3)	49(2)
C(5)	2565(7)	2477(8)	8099(3)	47(1)

Table 6: Bond lengths [Å] and angles [°] for $C_{22}H_{42}Br_2N_4O0$

C(8)-C(8)#1	1.390(10)	N(1)-C(1)	1.329(5)
C(8)-N(2)	1.401(7)	N(1)-C(4)	1.479(6)
C(8)-C(9)	1.475(7)	C(12)-C(10)	1.511(8)
N(2)-C(7)	1.325(5)	C(1)-N(1)#2	1.329(5)
N(2)-C(10)	1.476(6)	C(7)-N(2)#1	1.325(5)
C(2)-N(1)	1.382(6)	C(10)-C(11)	1.533(9)
C(2)-C(2)#2	1.404(10)	C(4)-C(5)	1.509(9)
C(2)-C(3)	1.477(7)	C(4)-C(6)	1.526(8)
C(8)#1-C(8)-N(2)	106.2(3)	C(1)-N(1)-C(4)	124.1(4)
C(8)#1-C(8)-C(9)	129.6(4)	C(2)-N(1)-C(4)	126.6(4)
N(2)-C(8)-C(9)	124.2(5)	N(1)#2-C(1)-N(1)	109.3(6)
C(7)-N(2)-C(8)	108.8(4)	N(2)-C(7)-N(2)#1	110.0(6)
C(7)-N(2)-C(10)	125.1(5)	N(2)-C(10)-C(12)	110.8(5)
C(8)-N(2)-C(10)	126.0(4)	N(2)-C(10)-C(11)	108.8(4)
N(1)-C(2)-C(2)#2	106.1(3)	C(12)-C(10)-C(11)	112.1(6)
N(1)-C(2)-C(3)	123.6(5)	N(1)-C(4)-C(5)	109.6(4)
C(2)#2-C(2)-C(3)	130.3(3)	N(1)-C(4)-C(6)	109.8(5)
C(1)-N(1)-C(2)	109.3(4)	C(5)-C(4)-C(6)	112.6(5)

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1,-z #2 -x,y,-z+3/2

7.3.3 Crystal data for $C_{20}H_{27}N_3O$ **(33)**

Table 7. Crystal data and structure refinement for $C_{20}H_{27}N_3O$ **(33)**

Empirical formula	$C_{20}H_{27}N_3O$	
Formula weight	325.45	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 8.460 (2) Å	α= 90°.
	b = 12.032(2) Å	β= 90.45(3)°.
	c = 18.279(4) Å	γ = 90°.
Volume	1860.5(6) $Å^3$	
Z	4	
Density (calculated)	1.162 Mg/m^3	
Absorption coefficient	0.073 mm^{-1}	
F(000)	704	
Crystal size	0.45 x 0.30 x 0.20 mm^3	
Theta range for data collection	3.14 to 29.02°.	
Index ranges	-1<=h<=11, -1<=k<=16, -24<=l<=24	
Reflections collected	6397	
Independent reflections	4933 [R(int) = 0.0425]	
Completeness to theta = 29.02°	99.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4933 / 0 / 326	
Goodness-of-fit on F^2	1.010	
Final R indices [I>2sigma(I)]	R1 = 0.0558, wR2 = 0.1158	
R indices (all data)	R1 = 0.1511, wR2 = 0.1424	
Extinction coefficient	0.0042(14)	
Largest diff. peak and hole	0.219 and -0.190 $e.Å^{-3}$	

Table 8: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{20}H_{27}N_3O$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	1258(2)	2796(1)	820(1)	30(1)
N(2)	82(2)	3890(1)	1577(1)	30(1)
N(3)	3022(3)	7881(2)	4628(1)	59(1)
O(1)	1960(2)	5871(1)	3188(1)	44(1)
C(1)	249(2)	2839(2)	1372(1)	31(1)
C(2)	1736(2)	3873(2)	651(1)	33(1)
C(3)	992(2)	4559(2)	1126(1)	33(1)
C(4)	1754(2)	1773(2)	436(1)	35(1)
C(5)	1793(3)	796(2)	961(1)	45(1)
C(6)	714(3)	1563(2)	-224(1)	51(1)
C(7)	2927(3)	4120(2)	77(1)	48(1)
C(8)	1097(3)	5785(2)	1210(2)	46(1)
C(9)	-864(3)	4282(2)	2211(1)	37(1)
C(10)	-984(4)	3367(2)	2772(1)	51(1)
C(11)	-2455(3)	4699(3)	1949(2)	59(1)
C(12)	3355(3)	7012(2)	4410(1)	41(1)
C(13)	3795(3)	5960(2)	4158(1)	40(1)
C(14)	3080(2)	5456(2)	3560(1)	32(1)
C(15)	3713(2)	4339(2)	3323(1)	33(1)
C(16)	4484(3)	3606(2)	3797(1)	44(1)
C(17)	5037(3)	2591(2)	3553(2)	53(1)
C(18)	4842(3)	2294(2)	2830(2)	52(1)
C(19)	4095(3)	3011(2)	2355(2)	54(1)
C(20)	3524(3)	4017(2)	2602(1)	43(1)

Table 9: Bond lengths [Å] and angles [°] for $C_{20}H_{27}N_3O$

N(1)-C(1)	1.329(2)	C(4)-C(5)	1.517(3)
N(1)-C(2)	1.392(2)	C(9)-C(10)	1.508(3)
N(1)-C(4)	1.479(2)	C(9)-C(11)	1.511(3)
N(2)-C(1)	1.327(2)	C(12)-C(13)	1.399(3)
N(2)-C(3)	1.391(2)	C(13)-C(14)	1.384(3)
N(2)-C(9)	1.489(2)	C(14)-C(15)	1.511(3)
N(3)-C(12)	1.155(3)	C(15)-C(20)	1.382(3)
O(1)-C(14)	1.265(2)	C(15)-C(16)	1.394(3)
C(2)-C(3)	1.356(3)	C(16)-C(17)	1.383(3)
C(2)-C(7)	1.490(3)	C(17)-C(18)	1.378(4)
C(3)-C(8)	1.487(3)	C(18)-C(19)	1.375(4)
C(4)-C(6)	1.509(3)	C(19)-C(20)	1.381(3)
C(1)-N(1)-C(2)	108.76(15)	N(2)-C(9)-C(10)	109.70(17)
C(1)-N(1)-C(4)	125.37(16)	N(2)-C(9)-C(11)	109.98(18)
C(2)-N(1)-C(4)	125.85(15)	C(10)-C(9)-C(11)	113.1(2)
C(1)-N(2)-C(3)	108.84(16)	N(3)-C(12)-C(13)	178.5(2)
C(1)-N(2)-C(9)	125.50(16)	C(14)-C(13)-C(12)	122.8(2)
C(3)-N(2)-C(9)	125.59(15)	O(1)-C(14)-C(13)	124.92(18)
N(2)-C(1)-N(1)	108.81(16)	O(1)-C(14)-C(15)	117.56(17)
C(3)-C(2)-N(1)	106.75(16)	C(13)-C(14)-C(15)	117.51(17)
C(3)-C(2)-C(7)	130.40(18)	C(20)-C(15)-C(16)	117.74(19)
N(1)-C(2)-C(7)	122.74(18)	C(20)-C(15)-C(14)	118.91(17)
C(2)-C(3)-N(2)	106.82(16)	C(16)-C(15)-C(14)	123.35(18)
C(2)-C(3)-C(8)	130.08(19)	C(17)-C(16)-C(15)	121.1(2)
N(2)-C(3)-C(8)	123.06(19)	C(18)-C(17)-C(16)	120.0(2)
N(1)-C(4)-C(6)	110.65(17)	C(19)-C(18)-C(17)	119.6(2)
N(1)-C(4)-C(5)	110.46(17)	C(18)-C(19)-C(20)	120.2(2)
C(6)-C(4)-C(5)	112.7(2)	C(19)-C(20)-C(15)	121.3(2)

Symmetry transformations used to generate equivalent atoms:

7.3.4 Crystal data for $C_{18}H_{31}Cl_3N_2$ **(35)**

Table 10: Crystal data and structure refinement for $C_{18}H_{31}Cl_3N_2$ **(35)**

Empirical formula	$C_{18}H_{31}Cl_3N_2$	
Formula weight	381.80	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 10.6081(17) Å	α= 90°.
	b = 12.4809(17) Å	β= 98.488(16)°.
	c = 16.0833(19) Å	□= 90°.
Volume	2106.1(5) $Å^3$	
Z	4	
Density (calculated)	1.204 Mg/m^3	
Absorption coefficient	0.437 mm^{-1}	
F(000)	816	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	2.07 to 27.50°.	
Index ranges	-1<=h<=13, -16<=k<=1, -20<=l<=20	
Reflections collected	6109	
Independent reflections	4831 [R(int) = 0.0281]	
Completeness to theta = 27.50°	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4831 / 0 / 211	
Goodness-of-fit on F^2	0.987	
Final R indices [I>2sigma(I)]	R1 = 0.0852, wR2 = 0.2502	
R indices (all data)	R1 = 0.1069, wR2 = 0.2760	
Extinction coefficient	0.007(3)	
Largest diff. peak and hole	1.800 and -1.331 $e.Å^{-3}$	

Table 11: Atomic coordinates(x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{18}H_{31}Cl_3N_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Cl(1)	9249(1)	408(1)	6466(1)	38(1)
Cl(2)	2284(2)	6909(1)	9826(1)	94(1)
Cl(3)	4780(2)	7895(2)	10173(2)	124(1)
N(1)	10910(2)	2515(2)	8390(2)	26(1)
N(2)	9111(2)	3318(2)	8005(2)	25(1)
C(1)	9784(3)	2437(3)	7904(2)	29(1)
C(2)	10972(3)	3487(2)	8819(2)	23(1)
C(3)	9832(3)	3992(2)	8578(2)	22(1)
C(4)	12133(3)	3849(3)	9384(2)	30(1)
C(5)	9342(3)	5044(3)	8823(2)	28(1)
C(6)	7875(3)	3612(2)	7505(2)	26(1)
C(7)	6985(3)	2653(3)	7340(3)	41(1)
C(8)	5722(3)	3031(3)	6834(3)	42(1)
C(9)	5932(4)	3561(4)	6014(3)	48(1)
C(10)	6849(4)	4509(4)	6181(3)	48(1)
C(11)	8113(3)	4146(4)	6691(2)	41(1)
C(12)	11916(3)	1688(3)	8451(2)	29(1)
C(13)	12452(4)	1620(3)	7617(3)	40(1)
C(14)	13501(4)	764(3)	7681(3)	46(1)
C(15)	13024(4)	-315(3)	7945(3)	39(1)
C(16)	12470(4)	-232(3)	8757(2)	38(1)
C(17)	11403(3)	604(3)	8680(2)	32(1)
C(18)	3999(4)	6635(3)	9983(3)	44(1)

Table 12: Bond lengths [Å] and angles [°] for $C_{18}H_{31}Cl_3N_2$

Cl(2)-C(18)	1.832(5)	C(6)-C(11)	1.524(5)
Cl(3)-C(18)	1.782(5)	C(7)-C(8)	1.537(5)
N(1)-C(1)	1.332(4)	C(8)-C(9)	1.520(7)
N(1)-C(2)	1.392(4)	C(9)-C(10)	1.530(6)
N(1)-C(12)	1.477(4)	C(10)-C(11)	1.533(5)
N(2)-C(1)	1.334(4)	C(12)-C(17)	1.524(5)
N(2)-C(3)	1.390(4)	C(12)-C(13)	1.534(5)
N(2)-C(6)	1.480(4)	C(13)-C(14)	1.535(5)
C(2)-C(3)	1.368(4)	C(14)-C(15)	1.520(5)
C(2)-C(4)	1.489(4)	C(15)-C(16)	1.513(5)
C(3)-C(5)	1.487(4)	C(16)-C(17)	1.531(5)
C(6)-C(7)	1.523(5)		
C(1)-N(1)-C(2)	109.1(3)	C(2)-C(3)-C(5)	131.4(3)
C(1)-N(1)-C(12)	124.4(3)	N(2)-C(3)-C(5)	122.1(3)
C(2)-N(1)-C(12)	126.6(3)	N(2)-C(6)-C(7)	112.1(3)
C(1)-N(2)-C(3)	109.2(3)	N(2)-C(6)-C(11)	109.2(3)
C(1)-N(2)-C(6)	125.8(3)	C(7)-C(6)-C(11)	111.8(3)
C(3)-N(2)-C(6)	124.5(2)	C(6)-C(7)-C(8)	108.7(3)
N(2)-C(1)-N(1)	108.5(3)	C(9)-C(8)-C(7)	111.4(3)
C(3)-C(2)-N(1)	106.6(3)	C(10)-C(9)-C(8)	110.8(3)
C(3)-C(2)-C(4)	130.5(3)	C(11)-C(10)-C(9)	110.4(4)
N(1)-C(2)-C(4)	122.7(3)	C(6)-C(11)-C(10)	110.2(3)
C(2)-C(3)-N(2)	106.6(3)		
N(1)-C(12)-C(17)	110.9(3)	C(16)-C(15)-C(14)	111.6(3)
N(1)-C(12)-C(13)	109.8(3)	C(15)-C(16)-C(17)	110.7(3)
C(17)-C(12)-C(13)	110.7(3)	C(12)-C(17)-C(16)	109.8(3)
C(14)-C(13)-C(12)	109.7(3)	Cl(3)-C(18)-Cl(2)	106.6(2)
C(15)-C(14)-C(13)	111.6(3)		

Symmetry transformations used to generate equivalent atoms:

7.3.5 Crystal data for $C_{20}H_{30}N_4$ **(36)**

Table 13: Crystal data and structure refinement for $C_{20}H_{30}N_4$ **(36)**

Empirical formula	$C_{20}H_{30}N_4$	
Formula weight	326.48	
Temperature	273(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 10.616(4) Å	α= 90°.
	b = 12.581(5) Å	β= 101.84(6)°.
	c = 14.408(10) Å	γ= 90°.
Volume	1883.4(16) $Å^3$	
Z	4	
Density (calculated)	1.151 Mg/m^3	
Absorption coefficient	0.069 mm^{-1}	
F(000)	712	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	2.17 to 27.56°.	
Index ranges	-13<=h<=11, -16<=k<=16, -18<=l<=18	
Reflections collected	16541	
Independent reflections	4341 [R(int) = 0.0497]	
Completeness to theta = 27.56°	99.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4341 / 0 / 220	
Goodness-of-fit on F^2	1.881	
Final R indices [I>2sigma(I)]	R1 = 0.0430, wR2 = 0.1120	
R indices (all data)	R1 = 0.0471, wR2 = 0.1181	
Extinction coefficient	0.070(6)	
Largest diff. peak and hole	0.361 and -0.215 $e.Å^{-3}$	

Table 14: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{20}H_{30}N_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	6378(1)	7658(1)	1904(1)	21(1)
N(2)	8046(1)	6856(1)	1586(1)	20(1)
N(3)	2312(2)	6457(1)	1003(1)	55(1)
N(4)	2790(1)	9976(1)	1357(1)	40(1)
C(1)	7218(1)	6868(1)	2169(1)	22(1)
C(2)	6675(1)	8166(1)	1121(1)	21(1)
C(3)	7726(1)	7658(1)	917(1)	22(1)
C(4)	5915(1)	9079(1)	647(1)	29(1)
C(5)	8471(1)	7871(1)	166(1)	30(1)
C(6)	9126(1)	6095(1)	1647(1)	22(1)
C(7)	9831(1)	5954(1)	2673(1)	29(1)
C(8)	10931(1)	5153(1)	2727(1)	32(1)
C(9)	10444(1)	4098(1)	2276(1)	33(1)
C(10)	9738(1)	4253(1)	1255(1)	37(1)
C(11)	8624(1)	5038(1)	1200(1)	30(1)
C(12)	5398(1)	8024(1)	2432(1)	23(1)
C(13)	5983(1)	8889(1)	3137(1)	31(1)
C(14)	4981(1)	9299(1)	3678(1)	37(1)
C(15)	4416(1)	8388(1)	4164(1)	39(1)
C(16)	3852(1)	7533(1)	3452(1)	38(1)
C(17)	4859(1)	7105(1)	2920(1)	32(1)
C(18)	2001(1)	8346(1)	366(1)	39(1)
C(19)	2421(1)	9228(1)	915(1)	29(1)
C(20)	2181(1)	7322(1)	727(1)	36(1)

Table 15: Bond lengths [Å] and angles [°] for $C_{20}H_{30}N_4$

N(1)-C(1)	1.3370(11)	C(7)-C(8)	1.5316(13)
N(1)-C(2)	1.3889(13)	C(8)-C(9)	1.5213(16)
N(1)-C(12)	1.4830(13)	C(9)-C(10)	1.5196(19)
N(2)-C(1)	1.3345(14)	C(10)-C(11)	1.5302(14)
N(2)-C(3)	1.3884(12)	C(12)-C(17)	1.5236(14)
N(2)-C(6)	1.4826(11)	C(12)-C(13)	1.5302(15)
N(3)-C(20)	1.157(2)	C(13)-C(14)	1.5315(15)
N(4)-C(19)	1.1582(16)	C(14)-C(15)	1.5275(18)
C(2)-C(3)	1.3686(12)	C(15)-C(16)	1.521(2)
C(2)-C(4)	1.4862(13)	C(16)-C(17)	1.5362(16)
C(3)-C(5)	1.4899(15)	C(18)-C(19)	1.3830(17)
C(6)-C(7)	1.5241(17)	C(18)-C(20)	1.3874(19)
C(6)-C(11)	1.5251(13)		
C(1)-N(1)-C(2)	108.96(8)	C(2)-C(3)-N(2)	106.60(9)
C(1)-N(1)-C(12)	125.56(8)	C(2)-C(3)-C(5)	130.44(9)
C(2)-N(1)-C(12)	125.03(8)	N(2)-C(3)-C(5)	122.95(8)
C(1)-N(2)-C(3)	109.20(8)	N(2)-C(6)-C(7)	110.64(8)
C(1)-N(2)-C(6)	124.85(8)	N(2)-C(6)-C(11)	109.82(8)
C(3)-N(2)-C(6)	125.95(8)	C(7)-C(6)-C(11)	111.58(8)
N(2)-C(1)-N(1)	108.43(8)	C(6)-C(7)-C(8)	110.05(9)
C(3)-C(2)-N(1)	106.82(8)	C(9)-C(8)-C(7)	111.32(9)
C(3)-C(2)-C(4)	130.71(9)	C(10)-C(9)-C(8)	110.98(9)
N(1)-C(2)-C(4)	122.46(8)	C(9)-C(10)-C(11)	110.80(10)
C(6)-C(11)-C(10)	109.94(9)	C(15)-C(14)-C(13)	111.08(10)
N(1)-C(12)-C(17)	111.71(8)	C(16)-C(15)-C(14)	111.07(10)
N(1)-C(12)-C(13)	109.26(8)	C(15)-C(16)-C(17)	111.48(10)
C(17)-C(12)-C(13)	111.92(9)	C(12)-C(17)-C(16)	109.28(9)
C(12)-C(13)-C(14)	110.34(9)	C(19)-C(18)-C(20)	121.81(11)
N(4)-C(19)-C(18)	178.25(12)	N(3)-C(20)-C(18)	177.98(14)

Symmetry transformations used to generate equivalent atoms:

7.3.6 Crystal data for $C_{18}H_{28}BrN_3$ **(37)**

Table 16: Crystal data and structure refinement for $C_{18}H_{28}BrN_3$ **(37)**

Empirical formula	$C_{18}H_{28}BrN_3$	
Formula weight	366.34	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	orthorhombic	
Space group	Pbcn	
Unit cell dimensions	a = 23.806(5) Å	α= 90°.
	b = 12.051(3) Å	β= 90°.
	c = 13.170(2) Å	γ= 90°.
Volume	3778.4(13) $Å^3$	
Z	8	
Density (calculated)	1.288 Mg/m^3	
Absorption coefficient	2.177 mm^{-1}	
F(000)	1536	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.07 to 28.01°.	
Index ranges	-31<=h<=31, -15<=k<=15, 0<=l<=17	
Reflections collected	17423	
Independent reflections	4548 [R(int) = 0.4431]	
Completeness to theta = 28.01°	99.6 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4548 / 0 / 200	
Goodness-of-fit on F^2	1.699	
Final R indices [I>2sigma(I)]	R1 = 0.2514, wR2 = 0.5706	
R indices (all data)	R1 = 0.4556, wR2 = 0.6434	
Extinction coefficient	0.011(4)	
Largest diff. peak and hole	1.937 and -1.521 $e.Å^{-3}$	

Table 17: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{18}H_{28}BrN_3$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Br(1)	7453(1)	1106(2)	1248(3)	83(2)
N(1)	6636(7)	6790(14)	4314(18)	61(6)
N(3)	6619(7)	8209(15)	3295(14)	49(4)
N(21)	5239(7)	7479(19)	3825(17)	78(7)
C(2)	6310(7)	7475(16)	3839(16)	38(4)
C(4)	7203(9)	7967(16)	3480(20)	63(7)
C(5)	7162(9)	6990(30)	4210(20)	74(8)
C(11)	6431(8)	5950(20)	5036(17)	52(6)
C(12)	6172(11)	6429(17)	6200(20)	66(8)
C(13)	6025(10)	5180(70)	6840(20)	300(50)
C(14)	5649(11)	4653(19)	6050(20)	76(8)
C(15)	5882(11)	4110(20)	5180(20)	70(7)
C(16)	6068(13)	5090(20)	4590(20)	99(11)
C(21)	5737(8)	7483(15)	3925(15)	37(5)
C(31)	6432(10)	9080(30)	2560(40)	170(20)
C(32)	6160(20)	8530(40)	1700(20)	210(30)
C(33)	6050(11)	9120(40)	810(30)	160(20)
C(34)	5658(14)	10040(40)	1320(20)	136(18)
C(35)	5876(12)	10780(20)	2300(30)	111(13)
C(36)	6087(8)	9921(17)	3246(18)	55(6)
C(41)	7714(11)	8620(30)	3330(30)	115(15)
C(51)	7600(20)	6500(19)	4670(40)	260(40)

Table 18: Bond lengths [Å] and angles [°] for $C_{18}H_{28}BrN_3$

N(1)-C(5)	1.28(2)	C(11)-C(12)	1.75(4)
N(1)-C(2)	1.30(2)	C(12)-C(13)	1.76(8)
N(1)-C(11)	1.47(3)	C(13)-C(14)	1.51(4)
N(3)-C(2)	1.35(2)	C(14)-C(15)	1.43(3)
N(3)-C(4)	1.44(2)	C(15)-C(16)	1.48(3)
N(3)-C(31)	1.50(3)	C(31)-C(32)	1.45(6)
N(21)-C(21)	1.19(2)	C(31)-C(36)	1.59(4)
C(2)-C(21)	1.37(2)	C(32)-C(33)	1.40(4)
C(4)-C(41)	1.46(3)	C(33)-C(34)	1.59(6)
C(4)-C(5)	1.52(4)	C(34)-C(35)	1.66(4)
C(5)-C(51)	1.35(6)	C(35)-C(36)	1.70(4)
C(11)-C(16)	1.47(3)		

C(5)-N(1)-C(2)	114(2)	N(1)-C(11)-C(12)	117.2(18)
C(5)-N(1)-C(11)	122(2)	C(16)-C(11)-C(12)	112.0(17)
C(2)-N(1)-C(11)	123.5(18)	C(13)-C(12)-C(11)	102.0(19)
C(2)-N(3)-C(4)	107.7(17)	C(14)-C(13)-C(12)	99(3)
C(2)-N(3)-C(31)	129.7(17)	C(15)-C(14)-C(13)	121(2)
C(4)-N(3)-C(31)	122.5(18)	C(14)-C(15)-C(16)	100(2)
N(3)-C(2)-N(1)	110.2(16)	C(15)-C(16)-C(11)	122(3)
N(3)-C(2)-C(21)	125.5(17)	N(21)-C(21)-C(2)	169(2)
N(1)-C(2)-C(21)	124.2(19)	C(32)-C(31)-N(3)	109(3)
N(3)-C(4)-C(41)	132(2)	C(32)-C(31)-C(36)	120(3)
N(3)-C(4)-C(5)	101.5(18)	N(3)-C(31)-C(36)	103(3)
C(41)-C(4)-C(5)	124(2)	C(33)-C(32)-C(31)	121(4)
N(1)-C(5)-C(51)	129(3)	C(32)-C(33)-C(34)	96(4)
N(1)-C(5)-C(4)	106(2)	C(33)-C(34)-C(35)	121(3)
C(51)-C(5)-C(4)	125(2)	C(34)-C(35)-C(36)	109(2)
N(1)-C(11)-C(16)	115(2)	C(31)-C(36)-C(35)	97(2)

Symmetry transformations used to generate equivalent atoms:

7.3.7 Crystal data for $C_{19}H_{29}Cl_3N_2S_2$ **(38)**

Table 19: Crystal data and structure refinement for $C_{19}H_{29}Cl_3N_2S_2$ **(38)**

Empirical formula	$C_{19}H_{29}Cl_3N_2S_2$	
Formula weight	455.91	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P21/c	
Unit cell dimensions	a = 8.4800(14) Å	α= 90°.
	b = 16.2270(9) Å	β= 98.78(2)°.
	c = 17.263(3) Å	γ = 90°.
Volume	2347.6(6) $Å^3$	
Z	4	
Density (calculated)	1.290 Mg/m^3	
Absorption coefficient	0.575 mm^{-1}	
F(000)	960	
Crystal size	0.60 x 0.50 x 0.30 mm^3	
Theta range for data collection	3.13 to 27.98°.	
Index ranges	-11<=h<=11, 0<=k<=21, -1<=l<=22	
Reflections collected	6181	
Independent reflections	5644 [R(int) = 0.0424]	
Completeness to theta = 27.98°	96.4 %	
Absorption correction	Psi-Scans	
Max. and min. transmission	0.93897 and 0.77486	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5644 / 0 / 235	
Goodness-of-fit on F^2	1.048	
Final R indices [I>2sigma(I)]	R1 = 0.0714, wR2 = 0.1869	
R indices (all data)	R1 = 0.1325, wR2 = 0.2183	
Largest diff. peak and hole	0.893 and -0.613 $e.Å^{-3}$	

Table 20: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{19}H_{29}Cl_3N_2S_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
S(1)	3302(1)	1417(1)	2196(1)	51(1)
S(2)	855(1)	2719(1)	1662(1)	58(1)
N(1)	4175(3)	3407(2)	2935(2)	35(1)
N(2)	2666(3)	2784(2)	3656(2)	36(1)
C(1)	2392(4)	2318(2)	2253(2)	36(1)
C(2)	3061(4)	2835(2)	2935(2)	33(1)
C(3)	3592(5)	3347(2)	4139(2)	39(1)
C(4)	4542(4)	3734(2)	3690(2)	39(1)
C(5)	5765(6)	4380(3)	3922(2)	57(1)
C(6)	3518(6)	3462(3)	4990(2)	57(1)
C(11)	1355(4)	2240(2)	3829(2)	41(1)
C(12)	-98(5)	2729(3)	3955(3)	62(1)
C(13)	-1477(6)	2126(4)	4030(3)	76(2)
C(14)	-983(6)	1477(3)	4646(3)	71(1)
C(15)	491(7)	1035(3)	4523(3)	73(1)
C(16)	1875(5)	1627(3)	4465(3)	58(1)
C(21)	4777(4)	3653(2)	2202(2)	38(1)
C(22)	6537(5)	3535(3)	2230(3)	61(1)
C(23)	6957(6)	3719(3)	1411(3)	69(1)
C(24)	6454(6)	4583(3)	1153(3)	62(1)
C(25)	4720(5)	4717(3)	1154(2)	58(1)
C(26)	4226(5)	4511(2)	1943(2)	47(1)
Cl(1)	432(2)	4818(1)	2800(1)	83(1)
Cl(2)	2629(2)	6145(1)	3217(1)	93(1)
Cl(3)	894(3)	5432(2)	4361(1)	124(1)
C(30)	824(5)	5709(3)	3389(3)	55(1)

Table 21: Bond lengths [Å] and angles [°] for $C_{19}H_{29}Cl_3N_2S_2$

S(1)-C(1)	1.663(4)	C(11)-C(12)	1.509(6)
S(2)-C(1)	1.660(4)	C(12)-C(13)	1.545(7)
N(1)-C(2)	1.324(4)	C(13)-C(14)	1.510(7)
N(1)-C(4)	1.398(4)	C(14)-C(15)	1.484(7)
N(1)-C(21)	1.490(4)	C(15)-C(16)	1.532(6)
N(2)-C(2)	1.339(4)	C(21)-C(22)	1.499(5)
N(2)-C(3)	1.396(4)	C(21)-C(26)	1.514(5)
N(2)-C(11)	1.485(4)	C(22)-C(23)	1.540(6)
C(1)-C(2)	1.486(4)	C(23)-C(24)	1.512(7)
C(3)-C(4)	1.354(5)	C(24)-C(25)	1.487(6)
C(3)-C(6)	1.491(5)	C(25)-C(26)	1.522(5)
C(4)-C(5)	1.486(5)	Cl(1)-C(30)	1.770(5)
C(11)-C(16)	1.497(5)	Cl(2)-C(30)	1.752(5)
Cl(3)-C(30)	1.729(4)		
C(2)-N(1)-C(4)	108.9(3)	N(1)-C(2)-C(1)	125.6(3)
C(2)-N(1)-C(21)	121.7(3)	N(2)-C(2)-C(1)	125.6(3)
C(4)-N(1)-C(21)	129.3(3)	C(4)-C(3)-N(2)	106.9(3)
C(2)-N(2)-C(3)	108.5(3)	C(4)-C(3)-C(6)	128.6(3)
C(2)-N(2)-C(11)	121.7(3)	N(2)-C(3)-C(6)	124.5(3)
C(3)-N(2)-C(11)	129.7(3)	C(3)-C(4)-N(1)	106.9(3)
C(2)-C(1)-S(2)	115.8(3)	C(3)-C(4)-C(5)	128.7(3)
C(2)-C(1)-S(1)	114.7(2)	N(1)-C(4)-C(5)	124.5(3)
S(2)-C(1)-S(1)	129.5(2)	N(2)-C(11)-C(16)	113.4(3)
N(1)-C(2)-N(2)	108.8(3)		
N(2)-C(11)-C(12)	111.7(3)	C(14)-C(15)-C(16)	112.1(4)
C(16)-C(11)-C(12)	113.5(3)	C(11)-C(16)-C(15)	108.8(4)
C(11)-C(12)-C(13)	109.0(4)	N(1)-C(21)-C(22)	114.0(3)
C(14)-C(13)-C(12)	111.6(4)	N(1)-C(21)-C(26)	111.6(3)
C(15)-C(14)-C(13)	112.7(4)		
C(22)-C(21)-C(26)	112.8(3)	C(24)-C(23)-C(22)	110.9(4)
C(21)-C(22)-C(23)	108.3(4)	C(25)-C(24)-C(23)	111.7(4)
C(24)-C(25)-C(26)	112.1(3)		
C(21)-C(26)-C(25)	110.6(3)		

Cl(3)-C(30)-Cl(2)	111.6(3)
Cl(3)-C(30)-Cl(1)	108.7(3)
Cl(2)-C(30)-Cl(1)	108.9(2)

Symmetry transformations used to generate equivalent atoms:

7.3.8 Crystal data for $C_{23}H_{35}N_2O_3P$ **(39)**

Table 22: Crystal data and structure refinement for $C_{23}H_{35}N_2O_3P$ **(39)**

Empirical formula	$C_{23}H_{35}N_2O_3P$	
Formula weight	418.50	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 10.914(2) Å	α= 90°.
	b = 12.053(2) Å	β= 92.64(3)°.
	c = 17.561(4) Å	γ = 90°.
Volume	2307.7(8) $Å^3$	
Z	4	
Density (calculated)	1.205 Mg/m^3	
Absorption coefficient	0.144 mm^{-1}	
F(000)	904	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.37 to 28.00°.	
Index ranges	-1<=h<=14, 0<=k<=15, -23<=l<=23	
Reflections collected	6384	
Independent reflections	5541 [R(int) = 0.0410]	
Completeness to theta = 28.00°	99.7 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5541 / 0 / 269	
Goodness-of-fit on F^2	1.014	
Final R indices [I>2sigma(I)]	R1 = 0.0522, wR2 = 0.1208	
R indices (all data)	R1 = 0.1137, wR2 = 0.1410	
Extinction coefficient	0.0057(11)	
Largest diff. peak and hole	0.291 and -0.273 $e.Å^{-3}$	

Table 23: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{23}H_{35}N_2O_3P$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
P(1)	3809(1)	3902(1)	10568(1)	41(1)
N(1)	4023(2)	10573(2)	2745(1)	43(1)
N(2)	5376(2)	9824(1)	3522(1)	37(1)
O(1)	3404(1)	4844(1)	10046(1)	49(1)
O(2)	5170(2)	4109(2)	10870(1)	54(1)
O(3)	3030(1)	3651(1)	11220(1)	54(1)
C(1)	4205(2)	9787(2)	3273(1)	41(1)
C(2)	5111(2)	11147(2)	2661(1)	44(1)
C(3)	5960(2)	10680(2)	3146(1)	42(1)
C(4)	5218(2)	12103(2)	2133(2)	66(1)
C(5)	7275(2)	10972(2)	3300(2)	62(1)
C(6)	2819(2)	10802(2)	2348(1)	48(1)
C(7)	2289(2)	9743(2)	1998(2)	61(1)
C(8)	1036(2)	9966(3)	1602(2)	65(1)
C(9)	178(2)	10494(2)	2144(2)	60(1)
C(10)	704(2)	11535(2)	2480(2)	66(1)
C(11)	1953(2)	11324(2)	2892(1)	53(1)
C(12)	5954(2)	9075(2)	4102(1)	38(1)
C(13)	6700(2)	8175(2)	3729(1)	45(1)
C(14)	7302(2)	7422(2)	4335(2)	60(1)
C(15)	6394(2)	6951(2)	4876(2)	60(1)
C(16)	5638(3)	7860(2)	5219(1)	61(1)
C(17)	5002(2)	8563(2)	4598(1)	47(1)
C(18)	3866(2)	2660(2)	9995(1)	46(1)
C(19)	4726(2)	2515(2)	9447(2)	61(1)
C(20)	4704(4)	1575(3)	8996(2)	94(1)
C(21)	3840(5)	778(3)	9080(3)	143(2)
C(22)	2981(5)	913(3)	9617(3)	158(2)
C(23)	2998(4)	1848(2)	10070(2)	94(1)

Table 24: Bond lengths [Å] and angles [°] for $C_{23}H_{35}N_2O_3P$

P(1)-O(3)	1.4891(16)	C(8)-C(9)	1.507(3)
P(1)-O(1)	1.5124(16)	C(9)-C(10)	1.491(4)
P(1)-O(2)	1.5736(18)	C(10)-C(11)	1.535(3)
P(1)-C(18)	1.806(2)	C(12)-C(17)	1.518(3)
N(1)-C(1)	1.334(3)	C(12)-C(13)	1.523(3)
N(1)-C(2)	1.388(3)	C(13)-C(14)	1.524(3)
N(1)-C(6)	1.484(3)	C(14)-C(15)	1.514(3)
N(2)-C(1)	1.333(3)	C(15)-C(16)	1.514(3)
N(2)-C(3)	1.395(3)	C(16)-C(17)	1.523(3)
N(2)-C(12)	1.480(2)	C(18)-C(23)	1.374(4)
C(2)-C(3)	1.352(3)	C(18)-C(19)	1.385(3)
C(2)-C(4)	1.487(3)	C(19)-C(20)	1.382(4)
C(3)-C(5)	1.491(3)	C(20)-C(21)	1.359(5)
C(6)-C(11)	1.511(3)	C(21)-C(22)	1.370(5)
C(6)-C(7)	1.520(3)	C(22)-C(23)	1.379(4)
C(7)-C(8)	1.528(3)		

O(3)-P(1)-O(1)	117.21(9)	C(6)-C(7)-C(8)	110.6(2)
O(3)-P(1)-O(2)	109.71(10)	C(9)-C(8)-C(7)	110.9(2)
O(1)-P(1)-O(2)	109.25(10)	C(10)-C(9)-C(8)	111.5(2)
O(3)-P(1)-C(18)	107.20(10)	C(9)-C(10)-C(11)	111.4(2)
O(1)-P(1)-C(18)	107.52(9)	C(6)-C(11)-C(10)	109.8(2)
O(2)-P(1)-C(18)	105.26(11)	N(2)-C(12)-C(17)	111.23(17)
C(1)-N(1)-C(2)	109.07(17)	N(2)-C(12)-C(13)	110.98(16)
C(1)-N(1)-C(6)	123.96(18)	C(17)-C(12)-C(13)	110.49(17)
C(2)-N(1)-C(6)	126.91(18)	C(12)-C(13)-C(14)	110.28(19)
C(1)-N(2)-C(3)	108.74(17)	C(15)-C(14)-C(13)	112.8(2)
C(1)-N(2)-C(12)	125.28(17)	C(16)-C(15)-C(14)	111.3(2)
C(3)-N(2)-C(12)	125.98(17)	C(15)-C(16)-C(17)	110.9(2)
N(2)-C(1)-N(1)	108.33(18)	C(12)-C(17)-C(16)	109.64(19)
C(3)-C(2)-N(1)	106.89(18)	C(23)-C(18)-C(19)	118.0(2)
C(3)-C(2)-C(4)	130.1(2)	C(23)-C(18)-P(1)	119.70(19)
N(1)-C(2)-C(4)	123.0(2)	C(19)-C(18)-P(1)	122.23(19)
C(2)-C(3)-N(2)	106.95(18)	C(20)-C(19)-C(18)	120.5(3)
C(2)-C(3)-C(5)	129.9(2)	C(21)-C(20)-C(19)	120.8(3)
N(2)-C(3)-C(5)	123.09(19)	C(20)-C(21)-C(22)	119.4(3)
N(1)-C(6)-C(11)	110.29(18)	C(21)-C(22)-C(23)	120.2(4)
N(1)-C(6)-C(7)	110.22(18)	C(18)-C(23)-C(22)	121.2(3)
C(11)-C(6)-C(7)	111.58(19)		

Symmetry transformations used to generate equivalent atoms:

7.3.9 Crystal data for $C_{11.50}H_{16}NS_{0.50}$ **(41)**

Table 25: Crystal data and structure refinement for $C_{11.50}H_{16}NS_{0.50}$ **(41)**

Empirical formula	$C_{11.50}H_{16}NS_{0.50}$
Formula weight	184.28
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Trigonal, P3(2)21
Unit cell dimensions	a = 7.0791(8) A alpha = 90 deg.
	b = 7.0791(8) A beta = 90 deg.
	c = 34.273(3) A gamma = 120 deg.
Volume	1487.4(3) A^3
Z, Calculated density	6, 1.234 Mg/m^3
Absorption coefficient	0.173 mm^-1
F(000)	600
Crystal size	0.2 x 0.8 x 0.4 mm
Theta range for data collection	3.32 to 27.51 deg.
Limiting indices	-8<=h<=4, -9<=k<=9, -44<=l<=44
Reflections collected / unique	11269 / 2279 [R(int) = 0.0405]
Completeness to theta = 27.51	99.9 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2279 / 0 / 120
Goodness-of-fit on F^2	1.364
Final R indices [I>2sigma(I)]	R1 = 0.0989, wR2 = 0.3032
R indices (all data)	R1 = 0.0993, wR2 = 0.3033
Absolute structure parameter	0.6(4)
Extinction coefficient	0.035(7)
Largest diff. peak and hole	0.532 and -0.434 e.A^-3

Table 26: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for $C_{11.50}H_{16}NS_{0.50}$. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S(1)	2945(3)	0	1667	37(1)
N(1)	7099(8)	1197(8)	1428(1)	23(1)
C(1)	5358(11)	0	1667	24(2)
C(2)	8714(10)	704(11)	1522(2)	32(1)
C(3)	7265(10)	2674(10)	1100(2)	25(1)
C(4)	9533(10)	3707(13)	916(2)	38(2)
C(5)	5574(10)	1321(12)	781(2)	32(1)
C(6)	6925(11)	4527(11)	1249(2)	30(1)
C(7)	7044(14)	5983(12)	902(2)	42(2)
C(8)	9700(12)	5203(13)	577(2)	41(2)
C(9)	5701(12)	2796(13)	444(2)	35(2)
C(10)	5283(12)	4581(12)	594(2)	38(2)
C(11)	9311(14)	7028(13)	724(2)	47(2)
C(12)	8043(12)	3820(14)	262(2)	42(2)

Table 27: Bond lengths [A] and angles [deg] for $C_{11.50}H_{16}NS_{0.50}$

S(1)-C(1)	1.708(8)	C(4)-C(8)	1.536(9)
N(1)-C(1)	1.366(7)	C(5)-C(9)	1.527(9)
N(1)-C(2)	1.390(7)	C(6)-C(7)	1.547(8)
N(1)-C(3)	1.499(7)	C(7)-C(11)	1.520(12)
C(1)-N(1)#1	1.366(7)	C(7)-C(10)	1.554(10)
C(2)-C(2)#1	1.317(13)	C(8)-C(12)	1.532(10)
C(3)-C(4)	1.529(8)	C(8)-C(11)	1.535(12)
C(3)-C(6)	1.535(9)	C(9)-C(10)	1.523(11)
C(3)-C(5)	1.548(8)	C(9)-C(12)	1.569(10)

C(1)-N(1)-C(2)	107.8(5)	N(1)#1-C(1)-S(1)	126.3(3)
C(1)-N(1)-C(3)	127.9(5)	N(1)-C(1)-S(1)	126.3(3)
C(2)-N(1)-C(3)	124.2(5)	C(2)#1-C(2)-N(1)	108.5(3)
N(1)#1-C(1)-N(1)	107.4(7)	N(1)-C(3)-C(4)	109.8(5)
N(1)-C(3)-C(6)	110.8(4)	C(9)-C(5)-C(3)	110.3(5)
C(4)-C(3)-C(6)	107.7(6)	C(3)-C(6)-C(7)	109.5(5)
N(1)-C(3)-C(5)	109.3(5)	C(11)-C(7)-C(6)	109.0(6)
C(4)-C(3)-C(5)	107.9(5)	C(11)-C(7)-C(10)	110.6(6)
C(6)-C(3)-C(5)	111.1(5)	C(6)-C(7)-C(10)	109.7(6)
C(3)-C(4)-C(8)	110.0(5)	C(12)-C(8)-C(11)	111.5(7)
C(12)-C(8)-C(4)	108.7(6)	C(10)-C(9)-C(5)	110.1(5)
C(11)-C(8)-C(4)	110.4(6)	C(10)-C(9)-C(12)	110.4(6)
C(5)-C(9)-C(12)	107.6(6)	C(9)-C(10)-C(7)	109.4(6)
C(7)-C(11)-C(8)	108.2(6)	C(8)-C(12)-C(9)	108.1(5)

Symmetry transformations used to generate equivalent atoms:
#1 x-y,-y,-z+1/3

7.3.10 Crystal data for $C_{50}H_{66}CaN_8S_4$ **(42)**

Table 28: Crystal data and structure refinement for $C_{50}H_{66}CaN_8S_4$ **(42)**

Empirical formula	$C_{50}H_{66}CaN_8S_4$	
Formula weight	947.43	
Temperature	293(2) K	
Wavelength	71.073 pm	
Crystal system	triclinic	
Space group	P 1	
Unit cell dimensions	a = 661.44(10) pm	α= 100.55(2)°.
	b = 1128.27(16) pm	β= 90.09(2)°.
	c = 1754.5(4) pm	γ = 103.206(17)°.
Volume	1.2519(4) nm^3	
Z	1	
Density (calculated)	1.251 g/cm^3	
Absorption coefficient	0.286 mm^{-1}	
F(000)	503	
Crystal size	0.2 x 0.2 x 0.4 mm^3	
Theta range for data collection	3.29 to 25.78°.	
Index ranges	$-7 \leq h \leq 7$, $-13 \leq k \leq 13$, $0 \leq l \leq 20$	
Reflections collected	2111	
Independent reflections	2111 [R(int) = 0.0000]	
Completeness to theta = 25.78°	43.9 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2111 / 3 / 290	
Goodness-of-fit on F^2	0.819	
Final R indices [$I>2\sigma(I)$]	R1 = 0.0591, wR2 = 0.1304	
R indices (all data)	R1 = 0.1328, wR2 = 0.1551	
Absolute structure parameter	0.3(3)	
Largest diff. peak and hole	0.491 and -0.281 $e.Å^{-3}$	

Table 29: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (pm^2x 10^{-1}) for $C_{50}H_{66}CaN_8S_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
C(1)	4900(40)	6910(20)	1117(11)	34(6)
N(1)	5390(30)	7616(16)	1844(8)	24(4)
C(2)	5200(40)	6810(20)	2323(12)	41(6)
C(3)	4800(30)	5617(16)	1940(8)	12(4)
N(2)	4460(30)	5677(16)	1208(9)	28(4)
C(4)	5910(30)	9019(19)	2028(10)	21(5)
C(5)	4330(30)	9352(16)	2609(8)	19(4)
C(6)	8120(30)	9360(20)	2357(11)	28(5)
C(7)	5760(30)	9466(19)	1255(11)	30(5)
C(8)	4910(40)	800(20)	2809(13)	40(7)
C(9)	8660(40)	800(20)	2600(13)	42(7)
C(10)	6280(40)	870(20)	1479(13)	44(7)
C(11)	4770(50)	1340(30)	2084(14)	58(8)
C(12)	7180(30)	1280(20)	3183(11)	26(5)
C(13)	8540(40)	1370(20)	1856(13)	43(7)
C(14)	3800(40)	4550(30)	514(13)	45(6)
C(15)	1800(40)	3850(20)	690(11)	34(6)
C(16)	5470(30)	3820(20)	473(10)	24(5)
C(17)	3680(40)	5010(20)	9765(11)	35(6)
C(18)	1080(40)	2680(30)	64(13)	48(7)
C(19)	4740(40)	2670(20)	9822(11)	34(6)
C(20)	3020(30)	3975(19)	9102(10)	26(5)
C(21)	880(40)	3150(20)	9278(12)	37(6)
C(22)	2770(30)	1940(20)	-15(10)	25(5)
C(23)	4570(40)	3150(20)	9026(12)	45(7)
C(24)	4190(30)	5343(19)	6911(10)	22(5)
N(3)	4510(30)	6457(16)	6887(8)	26(4)
C(25)	4300(40)	6480(20)	6048(13)	50(7)
C(26)	3670(30)	5383(18)	5678(10)	22(5)
N(4)	3740(30)	4587(17)	6220(9)	31(5)
C(27)	5190(30)	7561(17)	7499(9)	12(4)

C(28)	3550(40)	8360(20)	7597(12)	39(6)
C(29)	7320(40)	8360(20)	7304(11)	34(6)
C(30)	5420(30)	7120(20)	8286(10)	27(5)
C(31)	4250(40)	9470(20)	8240(11)	34(6)
C(32)	7960(40)	9470(20)	7992(11)	28(5)
C(33)	6130(40)	8280(20)	8913(11)	32(6)
C(34)	4490(30)	9100(20)	8990(11)	28(5)
C(35)	6430(40)	310(20)	8053(13)	47(7)
C(36)	8220(40)	9030(20)	8742(12)	38(6)
C(37)	3150(40)	3250(20)	6028(11)	33(6)
C(38)	3310(40)	2620(20)	6736(11)	33(6)
C(39)	960(30)	2710(20)	5652(12)	36(6)
C(40)	4610(40)	2640(20)	5413(11)	43(6)
C(41)	2680(40)	1240(20)	6538(11)	27(5)
C(42)	370(40)	1350(20)	5474(12)	37(6)
C(43)	4110(40)	1330(20)	5213(13)	40(7)
C(44)	4260(40)	800(20)	5961(11)	33(6)
C(45)	540(40)	810(20)	6201(12)	41(7)
C(46)	2000(40)	900(30)	4866(14)	54(7)
Ca(1)	78(11)	5958(8)	3006(4)	148(3)
S(1)	2060(12)	3719(8)	3505(4)	71(3)
C(47)	4440(40)	4420(20)	3617(11)	41(7)
N(5)	6230(40)	4740(20)	3655(10)	50(6)
S(2)	6975(10)	8450(6)	4519(3)	49(2)
C(48)	4460(40)	7920(20)	4455(11)	35(6)
N(6)	2730(40)	7320(20)	4368(12)	69(7)
S(3)	-76(13)	7486(10)	195(4)	92(4)
C(49)	80(40)	7110(20)	998(12)	51(7)
N(7)	-100(30)	6426(17)	1532(9)	56(5)
S(4)	-893(12)	4665(10)	7818(4)	86(3)
C(50)	-850(30)	5277(16)	7030(9)	23(4)
N(8)	-1030(20)	5470(15)	6443(8)	39(4)

Table 30: Bond lengths [pm] and angles [°] for $C_{50}H_{66}CaN_8S_4$

C(1)-N(1)	137(2)	C(20)-C(23)	153(3)
C(1)-N(2)	139(3)	C(20)-C(21)	157(3)
N(1)-C(2)	133(3)	C(21)-C(18)#4	158(3)
N(1)-C(4)	151(3)	C(22)-C(19)#3	143(3)
C(2)-C(3)	136(3)	C(24)-N(3)	123(2)
C(3)-N(2)	131.9(19)	C(24)-N(4)	134(2)
N(2)-C(14)	157(3)	N(3)-C(27)	147(2)
C(4)-C(6)	150(3)	N(3)-C(25)	149(2)
C(4)-C(5)	152(3)	C(25)-C(26)	127(3)
C(4)-C(7)	154(2)	C(26)-N(4)	143(2)
C(5)-C(8)#1	156(3)	N(4)-C(37)	144(3)
C(6)-C(9)#1	156(3)	C(27)-C(28)	155(3)
C(7)-C(10)#1	152(3)	C(27)-C(29)	156(3)
C(8)-C(11)	152(3)	C(27)-C(30)	157(2)
C(8)-C(5)#2	156(3)	C(28)-C(31)	151(3)
C(8)-C(12)	157(3)	C(29)-C(32)	155(3)
C(9)-C(12)	152(3)	C(30)-C(33)	153(3)
C(9)-C(6)#2	156(3)	C(31)-C(34)	147(3)
C(9)-C(13)	157(3)	C(31)-C(35)#1	161(3)
C(10)-C(7)#2	152(3)	C(32)-C(36)	151(3)
C(10)-C(11)	156(3)	C(32)-C(35)#1	153(3)
C(10)-C(13)	157(3)	C(33)-C(36)	151(3)
C(14)-C(15)	144(3)	C(33)-C(34)	157(3)
C(14)-C(17)#3	151(3)	C(35)-C(32)#2	153(3)
C(14)-C(16)	152(3)	C(35)-C(31)#2	161(3)
C(15)-C(18)	153(3)	C(37)-C(39)	154(3)
C(16)-C(19)#3	154(3)	C(37)-C(38)	156(3)
C(17)-C(20)	148(3)	C(37)-C(40)	162(3)
C(17)-C(14)#4	151(3)	C(38)-C(41)	149(3)
C(18)-C(22)	153(3)	C(39)-C(42)	148(3)
C(18)-C(21)#3	158(3)	C(40)-C(43)	142(3)
C(19)-C(22)#4	143(3)	C(41)-C(45)	147(3)
C(19)-C(16)#4	154(3)	C(41)-C(44)	156(3)
C(19)-C(23)	160(3)	C(42)-C(45)	152(3)

C(42)-C(46)	161(4)
C(43)-C(46)	146(3)
C(43)-C(44)	155(3)
Ca(1)-N(7)	274.1(17)
Ca(1)-N(6)	293(2)
Ca(1)-N(5)#5	294(2)
Ca(1)-S(1)	334.4(11)
S(1)-C(47)	159(3)
C(47)-N(5)	116(3)
N(5)-Ca(1)#6	294(2)
S(2)-C(48)	163(2)
C(48)-N(6)	118(3)
S(3)-C(49)	155(2)
C(49)-N(7)	131(3)
S(4)-C(50)	165.1(16)
C(50)-N(8)	110.3(18)
N(1)-C(1)-N(2)	106.5(17)
C(2)-N(1)-C(1)	105(2)
C(2)-N(1)-C(4)	129.4(17)
C(1)-N(1)-C(4)	125.1(17)
N(1)-C(2)-C(3)	112.5(18)
N(2)-C(3)-C(2)	104.8(16)
C(3)-N(2)-C(1)	110.1(16)
C(3)-N(2)-C(14)	126.6(17)
C(1)-N(2)-C(14)	123.2(17)
C(6)-C(4)-N(1)	104.3(17)
C(6)-C(4)-C(5)	114.1(17)
N(1)-C(4)-C(5)	104.9(15)
C(6)-C(4)-C(7)	111.4(16)
N(1)-C(4)-C(7)	107.1(14)
C(5)-C(4)-C(7)	114.1(17)
C(4)-C(5)-C(8)#1	104.6(16)
C(4)-C(6)-C(9)#1	105.5(19)
C(10)#1-C(7)-C(4)	104.5(16)
C(11)-C(8)-C(5)#2	110.6(19)
C(11)-C(8)-C(12)	109(2)
C(5)#2-C(8)-C(12)	110(2)
C(12)-C(9)-C(6)#2	113.0(19)
C(12)-C(9)-C(13)	108(2)
C(6)#2-C(9)-C(13)	108.5(18)
C(7)#2-C(10)-C(11)	112(2)
C(7)#2-C(10)-C(13)	111(2)
C(11)-C(10)-C(13)	107(2)
C(8)-C(11)-C(10)	109(2)
C(9)-C(12)-C(8)	108(2)
C(9)-C(13)-C(10)	108(2)
C(15)-C(14)-C(17)#3	111(2)
C(15)-C(14)-C(16)	113(3)
C(17)#3-C(14)-C(16)	109.3(18)
C(15)-C(14)-N(2)	106.4(18)
C(17)#3-C(14)-N(2)	110(2)
C(16)-C(14)-N(2)	106.9(18)
C(14)-C(15)-C(18)	110(2)
C(14)-C(16)-C(19)#3	106.5(19)
C(20)-C(17)-C(14)#4	112(2)
C(22)-C(18)-C(15)	109(2)
C(22)-C(18)-C(21)#3	108.0(18)
C(15)-C(18)-C(21)#3	106(2)
C(22)#4-C(19)-C(16)#4	110.9(18)
C(22)#4-C(19)-C(23)	110.5(17)
C(16)#4-C(19)-C(23)	108.0(18)
C(17)-C(20)-C(23)	109.6(19)
C(17)-C(20)-C(21)	108.7(17)
C(23)-C(20)-C(21)	106(2)
C(20)-C(21)-C(18)#4	110.2(18)
C(19)#3-C(22)-C(18)	112(2)
C(20)-C(23)-C(19)	109.0(17)
N(3)-C(24)-N(4)	114.5(17)
C(24)-N(3)-C(27)	130.9(16)
C(24)-N(3)-C(25)	104.2(17)
C(27)-N(3)-C(25)	124.6(17)

C(26)-C(25)-N(3)	109(2)	C(38)-C(37)-C(40)	102.8(18)
C(25)-C(26)-N(4)	106.7(17)	C(41)-C(38)-C(37)	113.1(16)
C(24)-N(4)-C(26)	105.3(18)	C(42)-C(39)-C(37)	114(2)
C(24)-N(4)-C(37)	130.1(18)	C(43)-C(40)-C(37)	116(2)
C(26)-N(4)-C(37)	124.1(16)	C(45)-C(41)-C(38)	112(2)
N(3)-C(27)-C(28)	111.0(16)	C(45)-C(41)-C(44)	111(2)
N(3)-C(27)-C(29)	110.2(15)	C(38)-C(41)-C(44)	107.5(18)
C(28)-C(27)-C(29)	110(2)	C(39)-C(42)-C(45)	111.0(19)
N(3)-C(27)-C(30)	108.2(15)	C(39)-C(42)-C(46)	107.2(19)
C(28)-C(27)-C(30)	107.6(15)	C(45)-C(42)-C(46)	109(2)
C(29)-C(27)-C(30)	110.2(15)	C(40)-C(43)-C(46)	110(2)
C(31)-C(28)-C(27)	110.5(19)	C(40)-C(43)-C(44)	108.6(19)
C(32)-C(29)-C(27)	106.8(16)	C(46)-C(43)-C(44)	110(2)
C(33)-C(30)-C(27)	107.1(17)	C(43)-C(44)-C(41)	108.2(19)
C(34)-C(31)-C(28)	111(2)	C(41)-C(45)-C(42)	109.4(19)
C(34)-C(31)-C(35)#1	108.6(18)	C(43)-C(46)-C(42)	110(2)
C(28)-C(31)-C(35)#1	110.5(18)	N(7)-Ca(1)-N(6)	130.7(6)
C(36)-C(32)-C(35)#1	112.0(18)	N(7)-Ca(1)-N(5)#5	118.7(6)
C(36)-C(32)-C(29)	111.3(18)	N(6)-Ca(1)-N(5)#5	104.4(6)
C(35)#1-C(32)-C(29)	111.0(19)	N(7)-Ca(1)-S(1)	125.0(5)
C(36)-C(33)-C(30)	111.0(19)	N(6)-Ca(1)-S(1)	78.8(6)
C(36)-C(33)-C(34)	110(2)	N(5)#5-Ca(1)-S(1)	85.3(5)
C(30)-C(33)-C(34)	111.1(19)	C(47)-S(1)-Ca(1)	99.3(10)
C(31)-C(34)-C(33)	108.3(17)	N(5)-C(47)-S(1)	169(2)
C(32)#2-C(35)-C(31)#2	105(2)	C(47)-N(5)-Ca(1)#6	149.2(15)
C(33)-C(36)-C(32)	107.7(18)	N(6)-C(48)-S(2)	167(3)
N(4)-C(37)-C(39)	115(2)	C(48)-N(6)-Ca(1)	133.9(16)
N(4)-C(37)-C(38)	113.2(16)	N(7)-C(49)-S(3)	160(2)
C(39)-C(37)-C(38)	107.3(18)	C(49)-N(7)-Ca(1)	156.1(15)
N(4)-C(37)-C(40)	114.2(18)	N(8)-C(50)-S(4)	166.7(16)
C(39)-C(37)-C(40)	103.4(18)		

Symmetry transformations used to generate equivalent atoms:

#1 x,y+1,z #2 x,y-1,z #3 x,y,z-1 #4 x,y,z+1
#5 x-1,y,z #6 x+1,y,z

7.3.11 Crystal data for $C_{13}H_{20}AgBrN_4$ **(43)**

Table 31: Crystal data and structure refinement for $C_{13}H_{20}AgBrN_4$ **(43)**

Empirical formula	$C_{13}H_{20}AgBrN_4$	
Formula weight	420.11	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P212121	
Unit cell dimensions	a = 6.6986(15) Å	α= 90°.
	b = 10.6222(14) Å	β= 90°.
	c = 23.989(4) Å	γ = 90°.
Volume	1706.9(5) $Å^3$	
Z	4	
Density (calculated)	1.635 Mg/m^3	
Absorption coefficient	3.515 mm^{-1}	
F(000)	832	
Crystal size	0.4 x 0.2 x 0.2 mm^3	
Theta range for data collection	3.16 to 27.94°.	
Index ranges	-8<=h<=8, -1<=k<=14, -1<=l<=31	
Reflections collected	5034	
Independent reflections	4090 [R(int) = 0.0498]	
Completeness to theta = 27.94°	99.8 %	
Absorption correction	DIFABS	
Max. and min. transmission	1.177 and 0.517	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4090 / 0 / 205	
Goodness-of-fit on F^2	0.964	
Final R indices [I>2sigma(I)]	R1 = 0.0505, wR2 = 0.0713	
R indices (all data)	R1 = 0.1895, wR2 = 0.0940	
Absolute structure parameter	0.472(17)	
Largest diff. peak and hole	0.358 and -0.845 $e.Å^{-3}$	

Table 32: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{13}H_{20}AgBrN_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Ag(1)	2807(1)	5128(1)	1275(1)	73(1)
Br(1)	6227(1)	8826(1)	1326(1)	62(1)
N(2)	2837(9)	9891(6)	780(2)	44(2)
C(1)	3881(9)	9680(6)	1254(3)	44(2)
C(2)	1189(12)	10803(7)	1457(3)	49(2)
C(21)	-261(12)	11577(9)	1782(3)	84(3)
C(31)	-325(11)	11001(10)	474(3)	77(3)
N(1)	2892(10)	10249(7)	1669(2)	50(2)
C(3)	1183(13)	10589(7)	896(3)	51(2)
C(4)	3545(15)	9418(9)	229(3)	62(3)
C(5)	2180(20)	8514(13)	-23(5)	90(4)
C(6)	4165(14)	10549(10)	-140(4)	91(4)
C(7)	3491(14)	10181(10)	2260(3)	61(2)
C(8)	4090(30)	11498(14)	2459(6)	94(4)
C(9)	2055(16)	9463(9)	2605(3)	90(4)
N(22)	5315(11)	2657(8)	1046(3)	79(3)
C(11)	1084(15)	6693(9)	1400(4)	75(3)
N(11)	99(11)	7531(8)	1449(4)	90(3)
C(22)	4429(14)	3551(10)	1126(4)	76(3)

Table 33: Bond lengths [Å] and angles [°] for $C_{13}H_{20}AgBrN_4$

Ag(1)-C(22)	2.028(11)	C(2)-C(21)	1.492(10)
Ag(1)-C(11)	2.046(11)	C(31)-C(3)	1.496(10)
Br(1)-C(1)	1.823(7)	N(1)-C(7)	1.476(9)
N(2)-C(1)	1.354(8)	C(4)-C(5)	1.458(14)
N(2)-C(3)	1.362(8)	C(4)-C(6)	1.550(12)
N(2)-C(4)	1.490(9)	C(7)-C(9)	1.480(12)
C(1)-N(1)	1.340(8)	C(7)-C(8)	1.532(15)
C(2)-C(3)	1.367(9)	N(22)-C(22)	1.137(11)
C(2)-N(1)	1.381(8)	C(11)-N(11)	1.114(11)
C(22)-Ag(1)-C(11)	177.5(4)	C(2)-N(1)-C(7)	126.8(7)
C(1)-N(2)-C(3)	109.8(5)	N(2)-C(3)-C(2)	106.9(7)
C(1)-N(2)-C(4)	121.6(7)	N(2)-C(3)-C(31)	124.8(6)
C(3)-N(2)-C(4)	128.6(6)	C(2)-C(3)-C(31)	128.3(8)
N(1)-C(1)-N(2)	107.2(6)	C(5)-C(4)-N(2)	113.0(8)
N(1)-C(1)-Br(1)	125.4(5)	C(5)-C(4)-C(6)	116.2(9)
N(2)-C(1)-Br(1)	127.4(6)	N(2)-C(4)-C(6)	109.3(7)
C(3)-C(2)-N(1)	107.1(7)	N(1)-C(7)-C(9)	112.7(7)
C(3)-C(2)-C(21)	127.3(8)	N(1)-C(7)-C(8)	109.0(9)
N(1)-C(2)-C(21)	125.5(6)	C(9)-C(7)-C(8)	118.0(9)
C(1)-N(1)-C(2)	109.1(5)	N(11)-C(11)-Ag(1)	177.1(11)
C(1)-N(1)-C(7)	123.9(7)	N(22)-C(22)-Ag(1)	179.0(9)

Symmetry transformations used to generate equivalent atoms:

7.3.12 Crystal data for $C_{12}H_{22}ClN_3O$ **(44)**

Table 34: Crystal data and structure refinement for $C_{12}H_{22}ClN_3O$ **(44)**

Empirical formula	$C_{12}H_{22}ClN_3O$	
Formula weight	259.78	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pna2(1)	
Unit cell dimensions	a = 9.5298(19) Å	α= 90°.
	b = 12.648(3) Å	β= 90°.
	c = 12.511(3) Å	γ = 90°.
Volume	1508.0(5) $Å^3$	
Z	4	
Density (calculated)	1.144 Mg/m^3	
Absorption coefficient	0.244 mm^{-1}	
F(000)	560	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.13 to 30.52°.	
Index ranges	-13<=h<=13, -18<=k<=18, -17<=l<=17	
Reflections collected	24290	
Independent reflections	4552 [R(int) = 0.0840]	
Completeness to theta = 30.52°	98.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4552 / 1 / 169	
Goodness-of-fit on F^2	0.956	
Final R indices [I>2sigma(I)]	R1 = 0.0603, wR2 = 0.1445	
R indices (all data)	R1 = 0.1211, wR2 = 0.1624	
Absolute structure parameter	-0.08(12)	
Extinction coefficient	0.000(2)	
Largest diff. peak and hole	0.236 and -0.190 $e.Å^{-3}$	

Table 35: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{12}H_{22}ClN_3O$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Cl(1)	7830(1)	3256(1)	8037(2)	88(1)
N(1)	3647(3)	5359(2)	5821(2)	37(1)
N(2)	1901(3)	4791(2)	6777(2)	39(1)
N(3)	3295(4)	6999(2)	8078(3)	61(1)
O(1)	9659(4)	1170(2)	7694(3)	63(1)
C(1)	3134(4)	6341(3)	7457(3)	45(1)
C(2)	2899(3)	5517(2)	6699(2)	37(1)
C(3)	2024(4)	4137(2)	5914(3)	44(1)
C(4)	3115(4)	4497(2)	5311(3)	40(1)
C(5)	812(4)	4712(2)	7627(3)	49(1)
C(6)	-184(5)	5639(3)	7546(5)	76(1)
C(7)	1453(5)	4584(3)	8729(3)	58(1)
C(8)	1075(5)	3208(3)	5725(3)	63(1)
C(9)	3702(5)	4073(3)	4304(3)	57(1)
C(10)	4897(4)	5983(2)	5440(3)	43(1)
C(11)	6124(5)	5768(5)	6162(4)	79(1)
C(12)	4535(5)	7133(3)	5342(3)	63(1)

Table 36: Bond lengths [Å] and angles [°] for $C_{12}H_{22}ClN_3O$

N(1)-C(2)	1.324(4)	C(3)-C(4)	1.363(5)
N(1)-C(4)	1.361(4)	C(3)-C(8)	1.502(5)
N(1)-C(10)	1.507(4)	C(4)-C(9)	1.479(5)
N(2)-C(2)	1.326(4)	C(5)-C(6)	1.512(5)
N(2)-C(3)	1.365(4)	C(5)-C(7)	1.517(6)
N(2)-C(5)	1.489(4)	C(10)-C(12)	1.499(5)
N(3)-C(1)	1.149(4)	C(10)-C(11)	1.502(6)
C(1)-C(2)	1.427(5)		
C(2)-N(1)-C(4)	108.0(2)	C(4)-C(3)-C(8)	129.3(3)
C(2)-N(1)-C(10)	127.5(2)	N(2)-C(3)-C(8)	123.2(3)
C(4)-N(1)-C(10)	124.4(3)	N(1)-C(4)-C(3)	107.0(3)
C(2)-N(2)-C(3)	107.5(3)	N(1)-C(4)-C(9)	123.3(3)
C(2)-N(2)-C(5)	126.8(3)	C(3)-C(4)-C(9)	129.7(3)
C(3)-N(2)-C(5)	125.8(3)	N(2)-C(5)-C(6)	109.8(3)
N(3)-C(1)-C(2)	178.5(4)	N(2)-C(5)-C(7)	112.1(3)
N(1)-C(2)-N(2)	110.0(2)	C(6)-C(5)-C(7)	113.4(4)
N(1)-C(2)-C(1)	125.3(3)	C(12)-C(10)-C(11)	113.8(4)
N(2)-C(2)-C(1)	124.7(3)	C(12)-C(10)-N(1)	110.6(3)
C(4)-C(3)-N(2)	107.5(3)	C(11)-C(10)-N(1)	109.3(3)

Symmetry transformations used to generate equivalent atoms:

7.3.13 Crystal data for $C_{14}H_{20}N_4$ **(45)**

Table 37: Crystal data and structure refinement for $C_{14}H_{20}N_4$ **(45)**

Empirical formula	$C_{14}H_{20}N_4$	
Formula weight	244.34	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 9.3410(18) Å	α= 90°.
	b = 11.693(4) Å	β= 109.13(2)°.

	c = 13.203(6) Å γ = 90°.
Volume	1362.4(8) $Å^3$
Z	4
Density (calculated)	1.191 Mg/m^3
Absorption coefficient	0.074 mm^{-1}
F(000)	528
Crystal size	? x ? x ? mm^3
Theta range for data collection	2.31 to 29.53°.
Index ranges	-12<=h<=12, -16<=k<=16, -18<=l<=17
Reflections collected	14751
Independent reflections	3806 [R(int) = 0.0303]
Completeness to theta = 29.53°	99.9 %
Absorption correction	Empirical
Max. and min. transmission	0.4620 and 0.3712
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3806 / 0 / 244
Goodness-of-fit on F^2	1.891
Final R indices [I>2sigma(I)]	R1 = 0.0407, wR2 = 0.1024
R indices (all data)	R1 = 0.0434, wR2 = 0.1069
Extinction coefficient	0.089(9)
Largest diff. peak and hole	0.408 and -0.282 $e.Å^{-3}$

Table 38: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{14}H_{20}N_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	8627(1)	4190(1)	2058(1)	20(1)
N(2)	6528(1)	3359(1)	1090(1)	19(1)
N(3)	11150(1)	2714(1)	534(1)	40(1)
N(4)	6914(1)	4149(1)	-1689(1)	37(1)
C(1)	7914(1)	3708(1)	1094(1)	19(1)
C(2)	7674(1)	4137(1)	2681(1)	22(1)
C(3)	6354(1)	3638(1)	2071(1)	21(1)
C(4)	10112(1)	4780(1)	2314(1)	22(1)
C(5)	9978(1)	6039(1)	2539(1)	44(1)
C(6)	11349(1)	4165(1)	3178(1)	47(1)
C(7)	8085(1)	4528(1)	3815(1)	32(1)
C(8)	4973(1)	3390(1)	2362(1)	28(1)
C(9)	5456(1)	2721(1)	189(1)	21(1)
C(10)	4920(1)	1612(1)	561(1)	30(1)
C(11)	4147(1)	3478(1)	-458(1)	28(1)
C(12)	8516(1)	3584(1)	222(1)	22(1)
C(13)	9966(1)	3118(1)	391(1)	26(1)
C(14)	7642(1)	3896(1)	-826(1)	25(1)

Table 39: Bond lengths [Å] and angles [°] for $C_{14}H_{20}N_4$

N(1)-C(1)	1.3513(10)	C(2)-C(3)	1.3635(10)
N(1)-C(2)	1.3979(9)	C(2)-C(7)	1.4909(12)
N(1)-C(4)	1.4862(9)	C(3)-C(8)	1.4905(10)
N(2)-C(1)	1.3566(8)	C(4)-C(6)	1.5130(12)
N(2)-C(3)	1.3950(10)	C(4)-C(5)	1.5147(13)
N(2)-C(9)	1.4808(10)	C(9)-C(11)	1.5239(11)
N(3)-C(13)	1.1604(12)	C(9)-C(10)	1.5277(11)
N(4)-C(14)	1.1578(13)	C(12)-C(14)	1.4044(12)
C(1)-C(12)	1.4445(10)	C(12)-C(13)	1.4081(10)
C(1)-N(1)-C(2)	108.89(6)	C(2)-C(3)-C(8)	128.44(7)
C(1)-N(1)-C(4)	122.88(6)	N(2)-C(3)-C(8)	124.64(7)
C(2)-N(1)-C(4)	127.90(6)	N(1)-C(4)-C(6)	112.04(7)
C(1)-N(2)-C(3)	109.14(6)	N(1)-C(4)-C(5)	111.39(6)
C(1)-N(2)-C(9)	123.21(6)	C(6)-C(4)-C(5)	113.95(9)
C(3)-N(2)-C(9)	127.54(6)	N(2)-C(9)-C(11)	111.40(7)
N(1)-C(1)-N(2)	107.69(6)	N(2)-C(9)-C(10)	112.06(6)
N(1)-C(1)-C(12)	126.33(6)	C(11)-C(9)-C(10)	112.70(6)
N(2)-C(1)-C(12)	125.99(6)	C(14)-C(12)-C(13)	118.38(7)
C(3)-C(2)-N(1)	107.35(6)	C(14)-C(12)-C(1)	120.48(7)
C(3)-C(2)-C(7)	127.63(7)	C(13)-C(12)-C(1)	121.11(7)
N(1)-C(2)-C(7)	124.99(7)	N(3)-C(13)-C(12)	178.73(10)
C(2)-C(3)-N(2)	106.90(6)	N(4)-C(14)-C(12)	179.52(9)

Symmetry transformations used to generate equivalent atoms:

7.3.14 Crystal data for $C_{12}H_{16}N_4$ **(46)**

Table 40: Crystal data and structure refinement for $C_{12}H_{16}N_4$ **(46)**

Empirical formula	$C_{12}H_{16}N$
Formula weight	216.29
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic

Space group	C2/c	
Unit cell dimensions	a = 14.770(3) Å	α= 90°.
	b = 9.3490(19) Å	β= 105.27(3)°.
	c = 17.576(4) Å	γ = 90°.
Volume	2341.3(8) $Å^3$	
Z	8	
Density (calculated)	1.227 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	928	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.02 to 25.99°.	
Index ranges	-18<=h<=17, 0<=k<=11, -1<=l<=21	
Reflections collected	2529	
Independent reflections	2299 [R(int) = 0.0256]	
Completeness to theta = 25.99°	99.8 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2299 / 0 / 210	
Goodness-of-fit on F^2	1.086	
Final R indices [I>2sigma(I)]	R1 = 0.0406, wR2 = 0.0997	
R indices (all data)	R1 = 0.0599, wR2 = 0.1076	
Extinction coefficient	0.0065(8)	
Largest diff. peak and hole	0.230 and -0.146 $e.Å^{-3}$	

Table 41: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{12}H_{16}N_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	159(1)	9367(1)	3545(1)	31(1)
N(2)	1622(1)	9684(1)	3579(1)	32(1)
N(3)	-412(1)	13046(2)	4309(1)	52(1)
N(4)	1577(1)	13579(2)	2878(1)	59(1)
C(1)	819(1)	10379(2)	3572(1)	30(1)
C(2)	558(1)	8008(2)	3546(1)	34(1)
C(3)	1462(1)	8210(2)	3557(1)	34(1)
C(4)	-847(1)	9615(2)	3457(1)	35(1)
C(5)	-1126(1)	9432(2)	4220(1)	43(1)
C(6)	25(1)	6657(2)	3542(1)	45(1)
C(7)	2211(1)	7158(2)	3541(1)	48(1)
C(8)	2570(1)	10320(2)	3750(1)	37(1)
C(9)	2992(1)	10482(2)	4623(1)	52(1)
C(10)	690(1)	11900(2)	3580(1)	33(1)
C(11)	74(1)	12528(2)	3974(1)	36(1)
C(12)	1176(1)	12821(2)	3193(1)	39(1)

Table 42: Bond lengths [Å] and angles [°] for $C_{12}H_{16}N_4$

N(1)-C(1)	1.3503(17)	C(1)-C(10)	1.435(2)
N(1)-C(2)	1.4005(18)	C(2)-C(3)	1.344(2)
N(1)-C(4)	1.4709(17)	C(2)-C(6)	1.487(2)
N(2)-C(1)	1.3498(17)	C(3)-C(7)	1.486(2)
N(2)-C(3)	1.3975(18)	C(4)-C(5)	1.513(2)
N(2)-C(8)	1.4770(18)	C(8)-C(9)	1.504(3)
N(3)-C(11)	1.1486(19)	C(10)-C(12)	1.405(2)
N(4)-C(12)	1.154(2)	C(10)-C(11)	1.409(2)
C(1)-N(1)-C(2)	109.67(11)	C(2)-N(1)-C(4)	123.79(12)
C(1)-N(1)-C(4)	126.34(12)	C(1)-N(2)-C(3)	109.44(11)
C(1)-N(2)-C(8)	126.31(12)	N(2)-C(1)-C(10)	126.53(12)
C(3)-N(2)-C(8)	123.13(12)	N(1)-C(1)-C(10)	126.76(12)
N(2)-C(1)-N(1)	106.70(12)	C(3)-C(2)-N(1)	106.76(12)
C(3)-C(2)-C(6)	129.97(14)	C(2)-C(3)-C(7)	130.45(14)
N(1)-C(2)-C(6)	123.26(13)	N(2)-C(3)-C(7)	122.13(13)
C(2)-C(3)-N(2)	107.41(12)	N(1)-C(4)-C(5)	113.09(12)
N(2)-C(8)-C(9)	111.29(13)	C(12)-C(10)-C(11)	117.46(13)
C(12)-C(10)-C(1)	121.12(13)	C(11)-C(10)-C(1)	121.43(13)
N(3)-C(11)-C(10)	178.55(17)	N(4)-C(12)-C(10)	179.8(2)

Symmetry transformations used to generate equivalent atoms:

7.3.15 Crystal data for $C_{11}H_{22}ClN_3$ **(47)**

Table 43: Crystal data and structure refinement for $C_{11}H_{22}ClN_3$ **(47)**

Empirical formula	$C_{11}H_{22}ClN_3$	
Formula weight	231.77	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 14.530(3) Å	α= 90°.
	b = 10.1215(11) Å	β= 90°.
	c = 18.239(2) Å	γ = 90°.
Volume	2682.3(7) $Å^3$	
Z	8	
Density (calculated)	1.148 Mg/m^3	
Absorption coefficient	0.262 mm^{-1}	
F(000)	1008	
Crystal size	0.25 x 0.6 x 0.25 mm^3	
Theta range for data collection	2.23 to 27.50°.	
Index ranges	-18<=h<=18, -13<=k<=13, -23<=l<=1	
Reflections collected	12423	
Independent reflections	3077 [R(int) = 0.0469]	
Completeness to theta = 27.50°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3077 / 0 / 143	
Goodness-of-fit on F^2	1.006	
Final R indices [I>2sigma(I)]	R1 = 0.0733, wR2 = 0.2145	
R indices (all data)	R1 = 0.1038, wR2 = 0.2406	
Extinction coefficient	0.0029(18)	
Largest diff. peak and hole	0.865 and -0.486 $e.Å^{-3}$	

Table 44: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{11}H_{22}ClN_3$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Cl(1)	746(1)	9865(1)	1278(1)	59(1)
N(1)	1096(2)	4205(3)	4586(1)	53(1)
N(2)	1966(2)	3080(3)	3662(1)	44(1)
N(3)	2690(2)	3688(2)	4655(1)	41(1)
C(1)	1871(2)	3696(3)	4318(2)	40(1)
C(2)	2880(3)	2649(3)	3601(2)	50(1)
C(3)	3333(2)	3024(3)	4211(2)	48(1)
C(4)	1162(3)	2777(4)	3177(2)	59(1)
C(5)	885(4)	1348(5)	3228(2)	91(2)
C(6)	1331(4)	3229(5)	2396(2)	81(1)
C(7)	3260(3)	1951(5)	2946(2)	76(1)
C(8)	4312(3)	2813(5)	4409(2)	69(1)
C(9)	2851(2)	4392(3)	5357(2)	48(1)
C(10)	3226(5)	3538(5)	5943(2)	119(3)
C(11)	3370(5)	5620(4)	5239(2)	94(2)

Table 45: Bond lengths [Å] and angles [°] for $C_{11}H_{22}ClN$

N(1)-C(1)	1.331(4)	C(2)-C(3)	1.348(5)
N(2)-C(1)	1.356(4)	C(2)-C(7)	1.493(4)
N(2)-C(2)	1.403(4)	C(3)-C(8)	1.482(5)
N(2)-C(4)	1.497(4)	C(4)-C(5)	1.504(6)
N(3)-C(1)	1.339(4)	C(4)-C(6)	1.516(5)
N(3)-C(3)	1.408(4)	C(9)-C(11)	1.470(6)
N(3)-C(9)	1.483(4)	C(9)-C(10)	1.479(5)
C(1)-N(2)-C(2)	108.1(3)	C(1)-N(3)-C(9)	122.2(2)
C(1)-N(2)-C(4)	122.4(3)	C(3)-N(3)-C(9)	128.3(3)
C(2)-N(2)-C(4)	128.9(3)	N(1)-C(1)-N(3)	125.8(3)
C(1)-N(3)-C(3)	109.2(2)	N(1)-C(1)-N(2)	126.0(3)

N(3)-C(1)-N(2)	108.2(3)	N(2)-C(4)-C(5)	111.7(3)
C(3)-C(2)-N(2)	108.0(3)	N(2)-C(4)-C(6)	111.6(4)
C(3)-C(2)-C(7)	127.8(3)	C(5)-C(4)-C(6)	113.0(3)
N(2)-C(2)-C(7)	124.1(3)	C(11)-C(9)-C(10)	114.3(4)
C(2)-C(3)-N(3)	106.5(3)	C(11)-C(9)-N(3)	111.1(3)
C(2)-C(3)-C(8)	129.0(3)	C(10)-C(9)-N(3)	113.7(3)
N(3)-C(3)-C(8)	124.5(3)		

Symmetry transformations used to generate equivalent atoms:

7.3.16 Crystal data for $C_{11}H_{21}N_3O_3$ **(48)**

Table 46: Crystal data and structure refinement for $C_{11}H_{21}N_3O_3$ **(48)**

Empirical formula	$C_{11}H_{21}N_3O_3$	
Formula weight	243.31	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	P4(3)2(1)2	
Unit cell dimensions	a = 7.967(3) Å	α= 90°.
	b = 7.967(3) Å	β= 90°.
	c = 20.606(5) Å	γ = 90°.
Volume	1308.0(9) $Å^3$	
Z	4	
Density (calculated)	1.236 Mg/m^3	
Absorption coefficient	0.090 mm^{-1}	
F(000)	528	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	2.74 to 24.64°.	
Index ranges	-9<=h<=8, -9<=k<=9, -24<=l<=24	
Reflections collected	8772	
Independent reflections	1115 [R(int) = 0.0356]	
Completeness to theta = 24.64°	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1115 / 0 / 122	
Goodness-of-fit on F^2	1.049	

Final R indices [I>2sigma(I)]	R1 = 0.0264, wR2 = 0.0680
R indices (all data)	R1 = 0.0273, wR2 = 0.0693
Absolute structure parameter	0.8(16)
Extinction coefficient	0.060(5)
Largest diff. peak and hole	0.113 and -0.104 e.Å^{-3}

Table 47: Atomic coordinates(x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for $C_{11}H_{21}N_3O_3$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	8137(1)	9332(1)	410(1)	31(1)
N(2)	4797(1)	4797(1)	0	36(1)
O(1)	3709(1)	3709(1)	0	71(1)
O(2)	6102(1)	4606(2)	325(1)	57(1)
C(1)	8049(2)	8049(2)	0	35(1)
C(2)	9536(2)	10288(2)	258(1)	30(1)
C(3)	6892(2)	9649(2)	934(1)	34(1)
C(4)	6840(2)	8170(2)	1394(1)	47(1)
C(5)	5209(2)	10026(2)	633(1)	41(1)
C(6)	9989(2)	11828(2)	623(1)	43(1)

Table 48: Bond lengths [Å] and angles [°] for $C_{11}H_{21}N_3O_3$

C(4)-C(3)	1.513(2)
N(2)-O(1)	1.225(2)
N(2)-O(2)	1.2463(14)
N(2)-O(2)#1	1.2463(14)
N(1)-C(1)	1.3285(15)
N(1)-C(2)	1.3859(16)
N(1)-C(3)	1.4872(16)
C(2)-C(2)#1	1.359(2)
C(2)-C(6)	1.4835(18)
C(1)-N(1)#1	1.3285(15)
C(3)-C(5)	1.508(2)
O(1)-N(2)-O(2)	120.26(7)
O(1)-N(2)-O(2)#1	120.26(7)
O(2)-N(2)-O(2)#1	119.48(15)
C(1)-N(1)-C(2)	108.68(10)
C(1)-N(1)-C(3)	123.87(11)
C(2)-N(1)-C(3)	127.44(10)
C(2)#1-C(2)-N(1)	106.85(6)
C(2)#1-C(2)-C(6)	130.81(8)
N(1)-C(2)-C(6)	122.34(12)
N(1)#1-C(1)-N(1)	108.94(15)
N(1)-C(3)-C(5)	109.18(11)
N(1)-C(3)-C(4)	109.89(11)
C(5)-C(3)-C(4)	112.88(13)

Symmetry transformations used to generate equivalent atoms:
#1 y,x,-z

7.3.17 Crystal data for $C_{11}H_{20}BrN_3O_3$ **(49)**

Table 49: Crystal data and structure refinement for $C_{11}H_{20}BrN_3O_3$ **(49)**

Empirical formula	$C_{11}H_{20}BrN_3O_3$	
Formula weight	322.21	
Temperature	223(2) K	
Wavelength	1.54056 Å	
Crystal system	Orthorhombic	
Space group	Cmcm	
Unit cell dimensions	a = 6.7287(5) Å	α= 90°.
	b = 13.1473(13) Å	β= 90°.
	c = 16.047(3) Å	γ = 90°.
Volume	1419.6(3) $Å^3$	
Z	4	
Density (calculated)	1.508 Mg/m^3	
Absorption coefficient	4.010 mm^{-1}	
F(000)	664	
Crystal size	0.40 x 0.30 x 0.20 mm^3	
Theta range for data collection	5.51 to 64.93°.	
Index ranges	-7<=h<=7, -15<=k<=15, -18<=l<=18	
Reflections collected	4642	
Independent reflections	692 [R(int) = 0.1117]	
Completeness to theta = 64.93°	48.4 %	
Absorption correction	Psi-Scans	
Max. and min. transmission	0.9405 and 0.7162	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	692 / 0 / 79	
Goodness-of-fit on F^2	1.058	
Final R indices [I>2sigma(I)]	R1 = 0.0618, wR2 = 0.1231	
R indices (all data)	R1 = 0.0637, wR2 = 0.1243	
Extinction coefficient	0.00120(14)	
Largest diff. peak and hole	2.245 and -1.417 $e.Å^{-3}$	

Table 50: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{11}H_{20}BrN_3O_3$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Br(1)	5000	4796(1)	2500	47(1)
N(1)	5000	2800(4)	1822(3)	33(1)
C(1)	5000	3395(7)	2500	36(2)
C(2)	5000	1793(4)	2081(4)	31(1)
C(3)	5000	3101(5)	919(4)	42(2)
C(4)	5000	915(6)	1502(5)	46(2)
C(5)	3115(12)	3644(6)	689(4)	63(2)
N(2)	0	2408(7)	2500	69(3)
O(1)	0	1931(5)	1831(5)	88(2)
O(2)	0	3346(6)	2500	80(3)

Table 51: bond lengths [Å] and angles [°] for $C_{11}H_{20}BrN_3O_3$

Br(1)-C(1)	1.842(9)
N(1)-C(1)	1.340(7)
N(1)-C(2)	1.388(8)
N(1)-C(3)	1.502(8)
C(1)-N(1)#1	1.340(7)
C(2)-C(2)#1	1.345(12)
C(2)-C(4)	1.483(9)
C(3)-C(5)	1.502(7)
C(3)-C(5)#2	1.502(7)
N(2)-O(2)	1.233(11)
N(2)-O(1)	1.243(8)
N(2)-O(1)#1	1.243(8)
C(1)-N(1)-C(2)	108.3(5)
C(1)-N(1)-C(3)	129.0(5)
C(2)-N(1)-C(3)	122.7(5)
N(1)-C(1)-N(1)#1	108.6(7)
N(1)-C(1)-Br(1)	125.7(4)
N(1)#1-C(1)-Br(1)	125.7(4)
C(2)#1-C(2)-N(1)	107.4(3)
C(2)#1-C(2)-C(4)	128.8(4)
N(1)-C(2)-C(4)	123.7(6)
N(1)-C(3)-C(5)	111.3(4)
N(1)-C(3)-C(5)#2	111.3(4)
C(5)-C(3)-C(5)#2	115.2(8)
O(2)-N(2)-O(1)	120.3(5)
O(2)-N(2)-O(1)#1	120.3(5)
O(1)-N(2)-O(1)#1	119.4(10)

Symmetry transformations used to generate equivalent atoms:
#1 x,y,-z+1/2 #2 -x+1,y,z

7.3.18 Crystal data for $C_{25}H_{23}N_2O_3P$ **(51)**

Table 52: Crystal data and structure refinement for $C_{25}H_{23}N_2O_3P$ **(51)**

Empirical formula	$C_{25}H_{23}N_2O_3P$	
Formula weight	430.42	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 10.072(2) Å	α= 90°.
	b = 13.534(3) Å	β= 97.13(3)°.
	c = 16.224(3) Å	γ = 90°.
Volume	2194.4(8) $Å^3$	
Z	4	
Density (calculated)	1.303 Mg/m^3	
Absorption coefficient	0.155 mm^{-1}	
F(000)	904	
Crystal size	□ x ? x ? mm^3	
Theta range for data collection	3.01 to 30.08°.	
Index ranges	-1<=h<=14, -1<=k<=19, -22<=l<=22	
Reflections collected	7939	
Independent reflections	6388 [R(int) = 0.0966]	
Completeness to theta = 30.08°	98.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6388 / 0 / 373	
Goodness-of-fit on F^2	1.004	
Final R indices [I>2sigma(I)]	R1 = 0.0620, wR2 = 0.1369	
R indices (all data)	R1 = 0.1353, wR2 = 0.1653	
Extinction coefficient	0.0000(12)	
Largest diff. peak and hole	0.816 and -0.307 $e.Å^{-3}$	

Table 53: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{25}H_{23}N_2O_3P$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
P(1)	4515(1)	8780(1)	2449(1)	28(1)
N(1)	9029(2)	9887(2)	2984(1)	37(1)
N(2)	8548(2)	9713(2)	1546(1)	41(1)
O(1)	7743(2)	9136(1)	3857(1)	44(1)
O(2)	10152(2)	10778(2)	2109(2)	66(1)
O(3)	6945(2)	8631(2)	983(1)	51(1)
C(1)	7287(2)	8790(2)	2431(1)	29(1)
C(2)	7545(2)	9001(2)	1624(1)	34(1)
C(3)	9298(3)	10162(2)	2210(2)	43(1)
C(4)	7986(2)	9246(2)	3130(1)	32(1)
C(5)	8779(6)	10020(4)	708(2)	79(1)
C(6)	9822(4)	10339(3)	3707(2)	61(1)
C(7)	4766(2)	10030(2)	2138(1)	31(1)
C(8)	4345(3)	10322(2)	1327(2)	40(1)
C(9)	4691(3)	11253(2)	1054(2)	53(1)
C(10)	5447(3)	11879(2)	1593(2)	53(1)
C(11)	5874(3)	11589(2)	2401(2)	44(1)
C(12)	5552(2)	10663(2)	2676(2)	34(1)
C(13)	3851(2)	8721(2)	3428(1)	31(1)
C(14)	3536(3)	9552(2)	3861(2)	40(1)
C(15)	3067(3)	9441(3)	4631(2)	51(1)
C(16)	2901(3)	8508(3)	4942(2)	50(1)
C(17)	3193(3)	7677(2)	4505(2)	46(1)
C(18)	3672(2)	7779(2)	3745(2)	37(1)
C(19)	3348(2)	8193(2)	1678(1)	32(1)
C(20)	1995(3)	8146(2)	1756(2)	45(1)
C(21)	1116(3)	7712(3)	1143(2)	53(1)
C(22)	1589(3)	7299(2)	458(2)	51(1)
C(23)	2926(3)	7343(2)	374(2)	47(1)
C(24)	3809(3)	7795(2)	973(2)	40(1)
C(25)	6144(2)	8128(2)	2554(2)	32(1)

Table 54: Bond lengths [Å] and angles [°] for $C_{25}H_{23}N_2O_3P$

P(1)-C(7)	1.792(2)	C(7)-C(12)	1.397(3)
P(1)-C(19)	1.793(2)	C(8)-C(9)	1.394(4)
P(1)-C(13)	1.800(2)	C(9)-C(10)	1.377(4)
P(1)-C(25)	1.852(2)	C(10)-C(11)	1.384(4)
N(1)-C(3)	1.368(3)	C(11)-C(12)	1.382(3)
N(1)-C(4)	1.405(3)	C(13)-C(14)	1.384(3)
N(1)-C(6)	1.468(4)	C(13)-C(18)	1.395(3)
N(2)-C(3)	1.378(4)	C(14)-C(15)	1.397(4)
N(2)-C(2)	1.413(3)	C(15)-C(16)	1.379(4)
N(2)-C(5)	1.469(4)	C(16)-C(17)	1.381(4)
O(1)-C(4)	1.242(3)	C(17)-C(18)	1.384(3)
O(2)-C(3)	1.224(3)	C(19)-C(20)	1.386(3)
O(3)-C(2)	1.242(3)	C(19)-C(24)	1.394(3)
C(1)-C(2)	1.394(3)	C(20)-C(21)	1.378(4)
C(1)-C(4)	1.403(3)	C(21)-C(22)	1.380(4)
C(1)-C(25)	1.492(3)	C(22)-C(23)	1.372(4)
C(7)-C(8)	1.389(3)	C(23)-C(24)	1.376(4)

C(7)-P(1)-C(19)	108.96(11)	O(3)-C(2)-C(1)	125.1(2)
C(7)-P(1)-C(13)	111.79(11)	O(3)-C(2)-N(2)	118.6(2)
C(19)-P(1)-C(13)	108.25(11)	C(1)-C(2)-N(2)	116.2(2)
C(7)-P(1)-C(25)	108.75(11)	O(2)-C(3)-N(1)	122.1(3)
C(19)-P(1)-C(25)	110.43(11)	O(2)-C(3)-N(2)	121.5(3)
C(13)-P(1)-C(25)	108.66(11)	N(1)-C(3)-N(2)	116.5(2)
C(3)-N(1)-C(4)	124.1(2)	O(1)-C(4)-C(1)	125.3(2)
C(3)-N(1)-C(6)	118.0(3)	O(1)-C(4)-N(1)	118.3(2)
C(4)-N(1)-C(6)	117.7(2)	C(1)-C(4)-N(1)	116.4(2)
C(3)-N(2)-C(2)	124.0(2)	C(8)-C(7)-C(12)	120.0(2)
C(3)-N(2)-C(5)	117.7(3)	C(8)-C(7)-P(1)	119.88(19)
C(2)-N(2)-C(5)	118.2(3)	C(12)-C(7)-P(1)	119.51(18)
C(2)-C(1)-C(4)	122.4(2)	C(7)-C(8)-C(9)	119.9(3)
C(2)-C(1)-C(25)	119.1(2)	C(10)-C(9)-C(8)	119.7(3)
C(4)-C(1)-C(25)	118.27(19)	C(9)-C(10)-C(11)	120.6(3)

C(12)-C(11)-C(10)	120.4(3)	C(20)-C(19)-C(24)	119.2(2)
C(11)-C(12)-C(7)	119.5(2)	C(20)-C(19)-P(1)	121.56(18)
C(14)-C(13)-C(18)	120.5(2)	C(24)-C(19)-P(1)	119.20(19)
C(14)-C(13)-P(1)	123.06(19)	C(21)-C(20)-C(19)	120.3(3)
C(18)-C(13)-P(1)	116.44(18)	C(20)-C(21)-C(22)	119.9(3)
C(13)-C(14)-C(15)	119.3(3)	C(23)-C(22)-C(21)	120.2(3)
C(16)-C(15)-C(14)	119.8(3)	C(22)-C(23)-C(24)	120.3(3)
C(15)-C(16)-C(17)	120.9(2)	C(23)-C(24)-C(19)	120.0(3)
C(16)-C(17)-C(18)	119.8(3)	C(1)-C(25)-P(1)	112.99(17)
C(17)-C(18)-C(13)	119.6(3)		

Symmetry transformations used to generate equivalent atoms:

7.3.19 Crystal data for $C_{18}H_{30}N_4O_4$ **(52)**

Table 55: Crystal data and structure refinement for $C_{18}H_{30}N_4O_4$ **(52)**

Empirical formula	$C_{18}H_{30}N_4O_4$	
Formula weight	366.46	
Temperature	213(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 11.154(2) Å	α= 90°.
	b = 10.204(2) Å	β= 96.46(3)°
	c = 16.941(3) Å	γ = 90°.
Volume	1916.0(7) $Å^3$	
Z	4	
Density (calculated)	1.270 Mg/m^3	
Absorption coefficient	0.091 mm^{-1}	
F(000)	792	
Crystal size	0.70 x 0.35 x 0.25 mm^3	
Theta range for data collection	2.31 to 24.71°.	
Index ranges	-13<=h<=1, -11<=k<=1, -19<=l<=19	
Reflections collected	4250	
Independent reflections	3247 [R(int) = 0.0651]	
Completeness to theta = 24.71°	99.7 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3247 / 0 / 356	
Goodness-of-fit on F^2	1.045	
Final R indices [I>2sigma(I)]	R1 = 0.0448, wR2 = 0.1264	
R indices (all data)	R1 = 0.0486, wR2 = 0.1303	
Extinction coefficient	0.030(3)	
Largest diff. peak and hole	0.200 and -0.327 $e.Å^{-3}$	

Table 56: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{18}H_{30}N_4O_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	4906(1)	7655(1)	9(1)	34(1)
N(2)	6492(1)	6188(1)	326(1)	32(1)
N(3)	4946(1)	6675(1)	2910(1)	28(1)
N(4)	5221(1)	4569(1)	2959(1)	31(1)
O(1)	3274(1)	7699(1)	710(1)	40(1)
O(2)	6496(1)	7579(1)	-723(1)	44(1)
O(3)	6398(1)	4628(1)	1280(1)	42(1)
O(4)	2596(2)	118(2)	1407(1)	62(1)
C(1)	4775(1)	6107(2)	1060(1)	29(1)
C(2)	5892(1)	5574(2)	921(1)	31(1)
C(3)	5985(2)	7158(2)	-164(1)	33(1)
C(4)	4256(1)	7158(2)	616(1)	31(1)
C(5)	7650(2)	5639(2)	163(1)	41(1)
C(6)	4313(2)	8611(2)	-545(1)	49(1)
C(7)	4743(1)	5554(2)	2502(1)	27(1)
C(8)	5576(2)	6399(2)	3648(1)	35(1)
C(9)	5741(2)	5084(2)	3679(1)	37(1)
C(10)	4487(1)	7966(2)	2601(1)	30(1)
C(11)	5497(2)	8837(2)	2378(1)	41(1)
C(12)	3701(2)	8624(2)	3159(1)	44(1)
C(13)	5990(2)	7406(2)	4253(1)	51(1)
C(14)	6338(2)	4301(2)	4356(1)	57(1)
C(15)	5160(2)	3168(2)	2718(1)	35(1)
C(16)	6402(2)	2550(2)	2759(1)	46(1)
C(17)	4284(2)	2422(2)	3178(2)	55(1)
C(18)	4100(1)	5468(2)	1681(1)	29(1)

Table 57: Bond lengths [Å] and angles [°] for $C_{18}H_{30}N_4O_4$

N(1)-C(3)	1.368(2)	O(2)-C(3)	1.236(2)
N(1)-C(4)	1.418(2)	O(3)-C(2)	1.243(2)
N(1)-C(6)	1.459(2)	C(1)-C(4)	1.398(2)
N(2)-C(3)	1.372(2)	C(1)-C(2)	1.403(2)
N(2)-C(2)	1.417(2)	C(1)-C(18)	1.509(2)
N(2)-C(5)	1.462(2)	C(7)-C(18)	1.494(2)
N(3)-C(7)	1.342(2)	C(8)-C(9)	1.354(3)
N(3)-C(8)	1.392(2)	C(8)-C(13)	1.488(3)
N(3)-C(10)	1.487(2)	C(9)-C(14)	1.492(3)
N(4)-C(7)	1.343(2)	C(10)-C(12)	1.516(2)
N(4)-C(9)	1.393(2)	C(10)-C(11)	1.517(2)
N(4)-C(15)	1.487(2)	C(15)-C(16)	1.516(2)
O(1)-C(4)	1.252(2)	C(15)-C(17)	1.520(3)
C(3)-N(1)-C(4)	123.82(14)	N(1)-C(3)-N(2)	116.80(14)
C(3)-N(1)-C(6)	117.16(14)	O(1)-C(4)-C(1)	125.84(14)
C(4)-N(1)-C(6)	118.32(14)	O(1)-C(4)-N(1)	117.41(14)
C(3)-N(2)-C(2)	123.61(13)	C(1)-C(4)-N(1)	116.75(14)
C(3)-N(2)-C(5)	118.30(14)	N(3)-C(7)-N(4)	107.97(13)
C(2)-N(2)-C(5)	117.61(14)	N(3)-C(7)-C(18)	124.39(14)
C(7)-N(3)-C(8)	109.17(13)	N(4)-C(7)-C(18)	127.65(14)
C(7)-N(3)-C(10)	122.89(13)	C(9)-C(8)-N(3)	106.78(14)
C(8)-N(3)-C(10)	127.87(13)	C(9)-C(8)-C(13)	128.80(17)
C(7)-N(4)-C(9)	108.75(14)	N(3)-C(8)-C(13)	124.41(16)
C(7)-N(4)-C(15)	123.98(14)	C(8)-C(9)-N(4)	107.33(15)
C(9)-N(4)-C(15)	127.26(14)	C(8)-C(9)-C(14)	127.43(18)
C(4)-C(1)-C(2)	121.94(14)	N(4)-C(9)-C(14)	125.23(17)
C(4)-C(1)-C(18)	119.83(14)	N(3)-C(10)-C(12)	111.97(13)
C(2)-C(1)-C(18)	118.16(14)	N(3)-C(10)-C(11)	111.75(13)
O(3)-C(2)-C(1)	125.55(14)	C(12)-C(10)-C(11)	113.22(15)
O(3)-C(2)-N(2)	117.84(14)	N(4)-C(15)-C(16)	111.91(14)
C(1)-C(2)-N(2)	116.60(14)	N(4)-C(15)-C(17)	110.68(15)
O(2)-C(3)-N(1)	121.84(15)	C(16)-C(15)-C(17)	113.89(16)
O(2)-C(3)-N(2)	121.34(15)	C(7)-C(18)-C(1)	113.53(12)

7.3.20 Crystal data for $C_{14}H_{17}N_4O_6$ **(53)**

Table 58: Crystal data and structure refinement for $C_{14}H_{17}N_4O_6$ **(53)**

Empirical formula	$C_{14}H_{17}N_4O_6$	
Formula weight	337.32	
Temperature	293(2) K	
Wavelength	1.54184 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 14.701(3) Å	α= 90°.
	b = 13.296(3) Å	β= 101.50(3)°
	c = 8.0940(16) Å	γ = 90°.
Volume	1550.3(5) $Å^3$	
Z	4	
Density (calculated)	1.445 Mg/m^3	
Absorption coefficient	0.977 mm^{-1}	
F(000)	708	
Crystal size	0.28 x 0.20x 0.15 mm^3	
Theta range for data collection	6.14 to 60.00°.	
Index ranges	-16<=h<=16, -14<=k<=14, -1<=l<=9	
Reflections collected	5453	
Independent reflections	2302 [R(int) = 0.1773]	
Completeness to theta = 60.00°	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2302 / 0 / 222	
Goodness-of-fit on F^2	0.923	
Final R indices [I>2sigma(I)]	R1 = 0.0963, wR2 = 0.2567	
R indices (all data)	R1 = 0.2263, wR2 = 0.3138	
Extinction coefficient	0.0008(8)	
Largest diff. peak and hole	0.411 and -0.457 $e.Å^{-3}$	

Table 59: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{14}H_{17}N_4O_6$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	4564(5)	3594(5)	-129(10)	77(2)
N(2)	5661(4)	4075(5)	2320(8)	58(2)
N(3)	906(4)	8998(4)	706(7)	49(2)
N(4)	1619(4)	10433(4)	-245(7)	45(2)
O(1)	4117(4)	4066(5)	2335(9)	96(2)
O(2)	7187(4)	4105(5)	2234(7)	76(2)
O(3)	5147(4)	3201(5)	-2546(8)	82(2)
O(4)	1584(4)	8927(4)	-1598(7)	72(2)
O(5)	347(4)	9072(4)	3117(7)	75(2)
O(6)	1773(4)	11892(4)	1241(8)	69(2)
C(1)	4741(6)	3918(6)	1602(12)	62(2)
C(2)	5310(6)	3454(5)	-1060(11)	55(2)
C(3)	6186(5)	3659(5)	-182(8)	35(2)
C(4)	6395(6)	3937(6)	1462(11)	55(2)
C(5)	3661(6)	3322(7)	-919(14)	96(3)
C(6)	6953(7)	3557(8)	-1037(13)	93(3)
C(7)	5870(7)	4344(8)	4098(10)	86(3)
C(8)	1400(5)	9429(5)	-435(10)	54(2)
C(9)	743(5)	9481(5)	2125(9)	49(2)
C(10)	1058(5)	10526(5)	2342(8)	43(2)
C(11)	1506(5)	11006(5)	1109(9)	44(2)
C(12)	679(6)	7922(5)	505(11)	68(3)
C(13)	943(5)	11042(7)	3721(10)	65(2)
C(14)	2042(6)	10903(6)	-1577(10)	73(3)

Table 60: Bond lengths [Å] and angles [°] for $C_{14}H_{17}N_4O_6$

N(1)-C(5)	1.401(11)	O(1)-C(1)	1.204(9)
N(1)-C(1)	1.440(10)	O(2)-C(4)	1.227(9)
N(1)-C(2)	1.461(10)	O(3)-C(2)	1.226(9)
N(2)-C(1)	1.378(10)	O(4)-C(8)	1.228(8)
N(2)-C(4)	1.406(9)	O(5)-C(9)	1.211(8)
N(2)-C(7)	1.455(9)	O(6)-C(11)	1.239(8)
N(3)-C(9)	1.377(8)	C(2)-C(3)	1.369(10)
N(3)-C(8)	1.407(8)	C(3)-C(4)	1.357(10)
N(3)-C(12)	1.470(8)	C(3)-C(6)	1.441(9)
N(4)-C(11)	1.372(8)	C(9)-C(10)	1.464(10)
N(4)-C(8)	1.374(9)	C(10)-C(13)	1.349(9)
N(4)-C(14)	1.485(8)	C(10)-C(11)	1.449(9)
C(5)-N(1)-C(1)	119.5(8)	C(4)-C(3)-C(2)	124.7(7)
C(5)-N(1)-C(2)	118.2(8)	C(4)-C(3)-C(6)	116.8(8)
C(1)-N(1)-C(2)	122.1(7)	C(2)-C(3)-C(6)	118.5(7)
C(1)-N(2)-C(4)	123.9(7)	O(2)-C(4)-C(3)	123.7(7)
C(1)-N(2)-C(7)	116.7(7)	O(2)-C(4)-N(2)	117.9(8)
C(4)-N(2)-C(7)	119.3(7)	C(3)-C(4)-N(2)	118.3(8)
C(9)-N(3)-C(8)	123.7(6)	O(4)-C(8)-N(4)	122.1(6)
C(9)-N(3)-C(12)	118.3(6)	O(4)-C(8)-N(3)	120.2(6)
C(8)-N(3)-C(12)	117.3(6)	N(4)-C(8)-N(3)	117.6(6)
C(11)-N(4)-C(8)	124.4(6)	O(5)-C(9)-N(3)	122.0(7)
C(11)-N(4)-C(14)	118.8(6)	O(5)-C(9)-C(10)	121.9(7)
C(8)-N(4)-C(14)	116.7(6)	N(3)-C(9)-C(10)	116.1(6)
O(1)-C(1)-N(2)	123.3(9)	C(13)-C(10)-C(11)	119.6(7)
O(1)-C(1)-N(1)	121.4(9)	C(13)-C(10)-C(9)	119.6(7)
N(2)-C(1)-N(1)	115.3(7)	C(11)-C(10)-C(9)	120.8(6)
O(3)-C(2)-C(3)	122.9(7)	O(6)-C(11)-N(4)	120.6(6)
O(3)-C(2)-N(1)	121.4(8)	O(6)-C(11)-C(10)	122.6(7)
C(3)-C(2)-N(1)	115.7(7)	N(4)-C(11)-C(10)	116.7(6)

7.3.21 Crystal data for $C_6H_6Cl_2N_2O_3$ **(54)**

Table 61: Crystal data and structure refinement for $C_6H_6Cl_2N_2O_3$ **(54)**

Empirical formula	$C_6H_6Cl_2N_2O_3$	
Formula weight	225.03	
Temperature	220(2) K	
Wavelength	71.073 pm	
Crystal system	Monoclinic	
Space group	P 2_1/c	
Unit cell dimensions	a = 741.7(1) pm	α= 90°
	b = 1585.2(2) pm	β= 94.31(2)
	c = 1536.8(2) pm	□ = 90°
Volume	1.8017(4) nm^3	
Z	8	
Density (calculated)	1.659 g/cm^3	
Absorption coefficient	0.695 mm^{-1}	
F(000)	912	
Crystal size	0.80 x 0.60 x 0.34 mm^3	
Theta range for data collection	2.57 to 26.04°	
Index ranges	$-9 \le h \le 9$, $-19 \le k \le 19$, $-18 \le l \le 18$	
Reflections collected	19954	
Independent reflections	3538 [R_{int} = 0.0616]	
Completeness to theta = 26.04°	99.5 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3538 / 0 / 283	
Goodness-of-fit on F^2	0.953	
Final R indices [I>2sigma(I)]	R_1 = 0.0358, wR_2 = 0.0947	
R indices (all data)	R_1 = 0.0476, wR_2 = 0.0987	
Largest diff. peak and hole	0.305 and -0.288 e.$Å^{-3}$	

Table 62: Atomic coordinates and equivalent isotropic displacement parameters ($pm^2 x\ 10^{-1}$) for $C_6H_6Cl_2N_2O_3$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Cl(1)	6207(1)	6050(1)	5675(1)	56(1)
Cl(2)	8591(1)	6667(1)	7122(1)	57(1)
Cl(3)	1308(1)	5568(1)	1869(1)	67(1)
Cl(4)	3035(1)	5935(1)	287(1)	53(1)
O(1)	6650(3)	7986(1)	6108(1)	70(1)
O(2)	10176(2)	7724(1)	3869(1)	56(1)
O(3)	10442(2)	5639(1)	5863(1)	52(1)
O(4)	5417(3)	4984(1)	1521(1)	62(1)
O(5)	5901(2)	7016(1)	3580(1)	51(1)
O(6)	2107(3)	7471(1)	1192(1)	67(1)
N(1)	8407(2)	7860(1)	4990(1)	37(1)
N(2)	10156(2)	6623(1)	4801(1)	35(1)
N(3)	5552(2)	5954(1)	2601(1)	39(1)
N(4)	3884(2)	7235(1)	2426(1)	36(1)
C(1)	8149(3)	6674(1)	5988(1)	35(1)
C(2)	7661(3)	7573(1)	5705(1)	41(1)
C(3)	9608(3)	7422(1)	4510(1)	37(1)
C(4)	9723(3)	6261(1)	5560(1)	36(1)
C(5)	7887(5)	8714(2)	4674(2)	59(1)
C(6)	11625(4)	6235(2)	4347(2)	54(1)
C(7)	3192(3)	6069(1)	1418(1)	40(1)
C(8)	4859(3)	5619(1)	1829(1)	40(1)
C(9)	5161(3)	6751(1)	2913(1)	35(1)
C(10)	3028(3)	7002(1)	1652(2)	42(1)
C(11)	7012(4)	5488(2)	3091(2)	57(1)
C(12)	3654(4)	8110(2)	2729(2)	49(1)

Table 63: Bond lengths [pm] and angles [°] for $C_6H_6Cl_2N_2O_3$

Cl(1)-C(1)	178.4(2)	N(2)-C(4)	136.1(3)
Cl(2)-C(1)	174.8(2)	N(2)-C(3)	139.2(3)
Cl(3)-C(7)	179.1(2)	N(2)-C(6)	147.2(3)
Cl(4)-C(7)	174.6(2)	N(3)-C(8)	136.5(3)
O(1)-C(2)	120.2(3)	N(3)-C(9)	138.9(3)
O(2)-C(3)	120.0(3)	N(3)-C(11)	147.1(3)
O(3)-C(4)	119.8(2)	N(4)-C(10)	135.6(3)
O(4)-C(8)	119.9(3)	N(4)-C(9)	139.4(3)
O(5)-C(9)	120.2(2)	N(4)-C(12)	147.7(3)
O(6)-C(10)	120.3(3)	C(1)-C(2)	152.7(3)
N(1)-C(2)	134.5(3)	C(1)-C(4)	152.9(3)
N(1)-C(3)	138.5(3)	C(7)-C(8)	152.2(3)
N(1)-C(5)	147.9(3)	C(7)-C(10)	152.9(3)
C(2)-N(1)-C(3)	125.8(2)	O(2)-C(3)-N(1)	121.1(2)
C(2)-N(1)-C(5)	117.7(2)	O(2)-C(3)-N(2)	121.0(2)
C(3)-N(1)-C(5)	116.5(2)	N(1)-C(3)-N(2)	118.0(2)
C(4)-N(2)-C(3)	125.2(2)	O(3)-C(4)-N(2)	123.5(2)
C(4)-N(2)-C(6)	117.4(2)	O(3)-C(4)-C(1)	121.2(2)
C(3)-N(2)-C(6)	115.9(2)	N(2)-C(4)-C(1)	115.2(2)
C(8)-N(3)-C(9)	125.4(2)	C(8)-C(7)-C(10)	115.6(2)
C(8)-N(3)-C(11)	117.6(2)	C(8)-C(7)-Cl(4)	110.3(2)
C(9)-N(3)-C(11)	116.4(2)	C(10)-C(7)-Cl(4)	110.5(2)
C(10)-N(4)-C(9)	125.1(2)	C(8)-C(7)-Cl(3)	105.4(1)
C(10)-N(4)-C(12)	118.4(2)	C(10)-C(7)-Cl(3)	105.0(2)
C(9)-N(4)-C(12)	116.0(2)	Cl(4)-C(7)-Cl(3)	109.6(1)
C(2)-C(1)-C(4)	116.7(2)	O(4)-C(8)-N(3)	123.4(2)
C(2)-C(1)-Cl(2)	108.2(1)	O(4)-C(8)-C(7)	121.6(2)
C(4)-C(1)-Cl(2)	109.5(1)	N(3)-C(8)-C(7)	114.8(2)
C(2)-C(1)-Cl(1)	105.8(1)	O(5)-C(9)-N(3)	121.3(2)
C(4)-C(1)-Cl(1)	105.9(1)	O(5)-C(9)-N(4)	120.9(2)
Cl(2)-C(1)-Cl(1)	110.7(1)	N(3)-C(9)-N(4)	117.8(2)
O(1)-C(2)-N(1)	123.2(2)	O(6)-C(10)-N(4)	123.6(2)
O(1)-C(2)-C(1)	120.3(2)	O(6)-C(10)-C(7)	120.8(2)
N(1)-C(2)-C(1)	116.5(2)	N(4)-C(10)-C(7)	115.5(2

7.3.22 Crystal data for $C_{12}H_{12}Cl_2N_6O_{10}$ **(55)**

Table 64: Crystal data and structure refinement for $C_{12}H_{12}Cl_2N_6O_{10}$ **(55)**

Empirical formula	$C_{12}H_{12}Cl_2N_6O_{10}$	
Formula weight	471.18	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Cc	
Unit cell dimensions	a = 9.6708(19) Å	α= 90°.
	b = 12.486(3) Å	β= 118.48(3)°
	c = 8.5834(17) Å	γ = 90°.
Volume	911.0(3) $Å^3$	
Z	2	
Density (calculated)	1.718 Mg/m^3	
Absorption coefficient	0.427 mm^{-1}	
F(000)	480	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.09 to 30.95°.	
Index ranges	-13<=h<=13, -1<=k<=18, -12<=l<=12	
Reflections collected	3126	
Independent reflections	2884 [R(int) = 0.0301]	
Completeness to theta = 30.95°	99.7 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2884 / 2 / 161	
Goodness-of-fit on F^2	1.026	
Final R indices [I>2sigma(I)]	R1 = 0.0504, wR2 = 0.1096	
R indices (all data)	R1 = 0.0993, wR2 = 0.1252	
Absolute structure parameter	0.14(8)	
Extinction coefficient	0.000(2)	
Largest diff. peak and hole	0.249 and -0.205 $e.Å^{-3}$	

Table 65: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{12}H_{12}Cl_2N_6O_{10}$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Cl(1)	1391(1)	1906(1)	663(1)	39(1)
N(1)	-35(3)	2290(2)	3641(3)	30(1)
N(2)	-726(3)	515(2)	2463(3)	30(1)
N(3)	3321(3)	973(2)	3794(4)	34(1)
O(1)	2409(3)	2939(2)	4419(3)	41(1)
O(2)	-2289(3)	1500(2)	3212(3)	43(1)
O(3)	881(3)	-424(2)	1720(4)	46(1)
O(4)	4255(3)	1037(2)	3238(4)	48(1)
O(5)	3548(3)	617(2)	5224(3)	47(1)
C(1)	1641(3)	1366(2)	2665(4)	29(1)
C(2)	1393(4)	2267(2)	3706(4)	28(1)
C(3)	-1100(3)	1441(3)	3099(4)	29(1)
C(4)	572(4)	383(3)	2255(4)	30(1)
C(5)	-447(5)	3250(3)	4348(6)	44(1)
C(6)	-1853(5)	-379(4)	1937(6)	42(1)

Table 66: Bond lengths [Å] and angles [°] for $C_{12}H_{12}Cl_2N_6O_{10}$

Cl(1)-C(1)	1.752(3)	N(3)-O(5)	1.223(4)
N(1)-C(2)	1.356(4)	N(3)-C(1)	1.523(4)
N(1)-C(3)	1.395(4)	O(1)-C(2)	1.212(4)
N(1)-C(5)	1.482(4)	O(2)-C(3)	1.201(4)
N(2)-C(4)	1.359(4)	O(3)-C(4)	1.202(4)
N(2)-C(3)	1.397(4)	C(1)-C(2)	1.525(4)
N(2)-C(6)	1.473(5)	C(1)-C(4)	1.534(4)
N(3)-O(4)	1.209(4)		
C(2)-N(1)-C(3)	125.1(3)	N(3)-C(1)-Cl(1)	110.7(2)
C(2)-N(1)-C(5)	117.9(3)	C(2)-C(1)-Cl(1)	107.7(2)
C(3)-N(1)-C(5)	116.8(3)	C(4)-C(1)-Cl(1)	108.7(2)
C(4)-N(2)-C(3)	125.4(3)	O(1)-C(2)-N(1)	124.8(3)
C(4)-N(2)-C(6)	117.5(3)	O(1)-C(2)-C(1)	119.4(3)
C(3)-N(2)-C(6)	117.0(3)	N(1)-C(2)-C(1)	115.6(3)
O(4)-N(3)-O(5)	127.6(3)	O(2)-C(3)-N(1)	120.6(3)
O(4)-N(3)-C(1)	119.9(3)	O(2)-C(3)-N(2)	121.2(3)
O(5)-N(3)-C(1)	112.4(3)	N(1)-C(3)-N(2)	118.1(3)
N(3)-C(1)-C(2)	106.4(2)	O(3)-C(4)-N(2)	124.4(3)
N(3)-C(1)-C(4)	106.6(3)	O(3)-C(4)-C(1)	119.7(3)
C(2)-C(1)-C(4)	116.6(3)	N(2)-C(4)-C(1)	115.8(3)

7.3.23 Crystal data for $C_{17}H_{26}ClN_5O_5$ **(56)**

Table 67: Crystal data and structure refinement for $C_{17}H_{26}ClN_5O_5$ **(56)**

Empirical formula	$C_{17}H_{26}ClN_5O_5$	
Formula weight	415.88	
Temperature	213(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/m	
Unit cell dimensions	a = 9.476(2) Å	α= 90°.
	b = 6.983(1) Å	β= 93.32(3)°.
	c = 14.848(3) Å	γ = 90°.
Volume	980.8(3) $Å^3$	
Z	2	
Density (calculated)	1.408 Mg/m^3	
Absorption coefficient	0.235 mm^{-1}	
F(000)	440	
Crystal size	1.4 x 0.15 x 0.10 mm^3	
Theta range for data collection	3.22 to 31.08°.	
Index ranges	-1<=h<=13, -1<=k<=10, -21<=l<=21	
Reflections collected	4418	
Independent reflections	3360 [R(int) = 0.0457]	
Completeness to theta = 31.08°	99.5 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3360 / 0 / 202	
Goodness-of-fit on F^2	1.028	
Final R indices [I>2sigma(I)]	R1 = 0.0599, wR2 = 0.1488	
R indices (all data)	R1 = 0.1175, wR2 = 0.1739	
Extinction coefficient	0.000(2)	
Largest diff. peak and hole	0.663 and -0.401 $e.Å^{-3}$	

Table 68: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{17}H_{26}ClN_5O_5$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Cl(1)	9996(1)	7500	4995(1)	26(1)
N(1)	7505(2)	7500	4025(2)	23(1)
N(2)	9356(2)	7500	3211(2)	23(1)
N(3)	4792(3)	2500	1057(2)	35(1)
N(4)	7093(3)	2500	587(2)	39(1)
N(5)	7240(4)	2500	3075(2)	37(1)
O(1)	9003(3)	2500	1556(2)	50(1)
O(2)	5211(3)	2500	-427(2)	62(1)
O(3)	4311(3)	2500	2524(2)	52(1)
O(4)	6452(4)	2500	3675(2)	106(2)
O(5)	8476(4)	2500	3270(2)	123(2)
C(1)	8912(3)	7500	4053(2)	21(1)
C(2)	7022(3)	7500	3121(2)	23(1)
C(3)	8161(3)	7500	2617(2)	26(1)
C(4)	6587(3)	7500	4809(2)	25(1)
C(5)	6781(3)	5692(4)	5368(2)	39(1)
C(6)	5481(3)	7500	2828(2)	32(1)
C(7)	8204(4)	7500	1613(2)	33(1)
C(8)	10878(3)	7500	2998(2)	29(1)
C(9)	11252(3)	5673(4)	2510(2)	43(1)
C(10)	7720(4)	2500	1474(2)	33(1)
C(11)	6720(4)	2500	2168(2)	31(1)
C(12)	5220(4)	2500	1982(2)	32(1)
C(13)	5671(4)	2500	354(2)	40(1)
C(14)	3265(5)	2500	818(3)	54(1)
C(15)	8081(6)	2500	-140(3)	58(1)

Table 69: Bond lengths [Å] and angles [°] for $C_{17}H_{26}ClN_5O_5$

Cl(1)-C(1)	1.687(3)	N(5)-O(4)	1.195(5)
N(1)-C(1)	1.332(3)	N(5)-C(11)	1.408(4)
N(1)-C(2)	1.393(3)	O(1)-C(10)	1.215(4)
N(1)-C(4)	1.494(4)	O(2)-C(13)	1.215(4)
N(2)-C(1)	1.341(3)	O(3)-C(12)	1.211(4)
N(2)-C(3)	1.394(4)	C(2)-C(3)	1.348(4)
N(2)-C(8)	1.494(4)	C(2)-C(6)	1.500(4)
N(3)-C(13)	1.372(5)	C(3)-C(7)	1.493(4)
N(3)-C(12)	1.409(4)	C(4)-C(5)#1	1.516(3)
N(3)-C(14)	1.470(5)	C(4)-C(5)	1.516(3)
N(4)-C(13)	1.372(5)	C(8)-C(9)	1.519(3)
N(4)-C(10)	1.413(4)	C(8)-C(9)#1	1.519(3)
N(4)-C(15)	1.469(5)	C(10)-C(11)	1.440(5)
N(5)-O(5)	1.190(5)	C(11)-C(12)	1.433(5)

C(1)-N(1)-C(2)	107.6(2)	C(3)-C(2)-N(1)	107.8(2)
C(1)-N(1)-C(4)	127.1(2)	C(3)-C(2)-C(6)	129.5(3)
C(2)-N(1)-C(4)	125.3(2)	N(1)-C(2)-C(6)	122.6(3)
C(1)-N(2)-C(3)	107.6(2)	C(2)-C(3)-N(2)	107.2(2)
C(1)-N(2)-C(8)	123.7(2)	C(2)-C(3)-C(7)	128.5(3)
C(3)-N(2)-C(8)	128.6(2)	N(2)-C(3)-C(7)	124.3(3)
C(13)-N(3)-C(12)	126.1(3)	N(1)-C(4)-C(5)#1	111.67(16)
C(13)-N(3)-C(14)	116.6(3)	N(1)-C(4)-C(5)	111.67(16)
C(12)-N(3)-C(14)	117.3(3)	C(5)#1-C(4)-C(5)	112.7(3)
C(13)-N(4)-C(10)	126.1(3)	N(2)-C(8)-C(9)	110.84(16)
C(13)-N(4)-C(15)	118.2(3)	N(2)-C(8)-C(9)#1	110.84(16)
C(10)-N(4)-C(15)	115.7(3)	C(9)-C(8)-C(9)#1	114.2(3)
O(5)-N(5)-O(4)	117.9(3)	O(1)-C(10)-N(4)	117.3(3)
O(5)-N(5)-C(11)	121.2(3)	O(1)-C(10)-C(11)	128.7(3)
O(4)-N(5)-C(11)	120.9(3)	N(4)-C(10)-C(11)	114.1(3)
N(1)-C(1)-N(2)	109.8(2)	N(5)-C(11)-C(12)	118.2(3)
N(1)-C(1)-Cl(1)	125.9(2)	N(5)-C(11)-C(10)	118.5(3)
N(2)-C(1)-Cl(1)	124.4(2)	C(12)-C(11)-C(10)	123.3(3)

O(3)-C(12)-N(3)	118.2(3)	O(2)-C(13)-N(4)	122.2(4)
O(3)-C(12)-C(11)	127.4(3)	O(2)-C(13)-N(3)	121.8(4)
N(3)-C(12)-C(11)	114.4(3)	N(4)-C(13)-N(3)	116.0(3)

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+3/2, z

7.3.24 Crystal data for $C_{18}H_{18}N_6O_9$ **(57)**

Table 70: Crystal data and structure refinement for $C_{18}H_{18}N_6O_9$ **(57)**

Empirical formula	$C_{18}H_{18}N_6O_9$	
Formula weight	462.38	
Temperature	223(2) K	
Wavelength	1.54184 Å	
Crystal system	orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 13.1526(10) Å	α= 90°.
	b = 15.873(3) Å	β= 90°.
	c = 19.571(2) Å	γ = 90°.
Volume	4086.0(9) $Å^3$	
Z	8	
Density (calculated)	1.503 Mg/m^3	
Absorption coefficient	1.058 mm^{-1}	
F(000)	1920	
Crystal size	0.65 x 0.10 x 0.10 mm^3	
Theta range for data collection	9.05 to 58.93°.	
Index ranges	-4<=h<=13, -4<=k<=17, -4<=l<=21	
Reflections collected	6481	
Independent reflections	2851 [R(int) = 0.0467]	
Completeness to theta = 58.93°	97.2 %	
Max. and min. transmission	0.9016 and 0.5463	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2851 / 0 / 299	
Goodness-of-fit on F^2	1.050	
Final R indices [I>2sigma(I)]	R1 = 0.0603, wR2 = 0.1598	
R indices (all data)	R1 = 0.0860, wR2 = 0.1779	
Extinction coefficient	0.0014(3)	
Largest diff. peak and hole	0.468 and -0.373 $e.Å^{-3}$	

Table 71: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{18}H_{18}N_6O_9$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
O(116)	5448(2)	9170(2)	3064(1)	38(1)
O(112)	4058(3)	11008(2)	1486(1)	51(1)
O(114)	5162(3)	11946(2)	3537(2)	63(1)
N(13)	4546(3)	11493(2)	2522(2)	38(1)
N(15)	5257(3)	10548(2)	3333(2)	32(1)
C(11)	4333(3)	9969(2)	2353(2)	29(1)
C(12)	4313(3)	10863(2)	2069(2)	34(1)
C(14)	4998(4)	11366(2)	3161(2)	40(1)
C(16)	5090(3)	9847(2)	2934(2)	30(1)
C(113)	4389(4)	12375(2)	2314(3)	62(2)
C(115)	5882(4)	10441(3)	3953(2)	46(1)
O(102)	2087(2)	10877(2)	2458(1)	41(1)
O(104)	3230(3)	10313(2)	4592(1)	55(1)
O(106)	3249(2)	8276(2)	2979(1)	42(1)
N(105)	3395(3)	9300(2)	3788(2)	34(1)
N(03)	2758(2)	10660(2)	3511(2)	32(1)
C(01)	3204(3)	9704(2)	2593(2)	28(1)
C(02)	2598(3)	10457(2)	2842(2)	30(1)
C(04)	3142(3)	10105(2)	4003(2)	35(1)
C(06)	3261(3)	9011(2)	3130(2)	30(1)
C(103)	2317(4)	11452(3)	3766(2)	54(1)
C(105)	3709(4)	8685(3)	4312(2)	50(1)
N(23)	3291(2)	8584(2)	1061(2)	32(1)
N(25)	5031(3)	8695(2)	768(2)	38(1)
C(21)	4437(3)	9343(2)	1778(2)	31(1)
C(22)	3509(3)	9072(2)	1602(2)	29(1)
C(24)	4088(3)	8361(2)	621(2)	35(1)
C(26)	5279(3)	9183(2)	1345(2)	36(1)
C(123)	2282(3)	8235(3)	924(2)	45(1)
C(125)	5855(4)	8464(4)	296(2)	63(2)
O(122)	2734(2)	9348(2)	2003(1)	31(1)
O(124)	3926(2)	7915(2)	129(1)	44(1)
O(126)	6146(2)	9443(2)	1432(2)	50(1)

Table 72: Bond lengths [Å] and angles [°] for $C_{18}H_{18}N_6O_9$

O(116)-C(16)	1.201(4)	N(105)-C(105)	1.476(5)
O(112)-C(12)	1.212(4)	N(03)-C(02)	1.364(5)
O(114)-C(14)	1.198(5)	N(03)-C(04)	1.399(5)
N(13)-C(12)	1.369(5)	N(03)-C(103)	1.472(5)
N(13)-C(14)	1.400(5)	C(01)-O(122)	1.426(4)
N(13)-C(113)	1.472(5)	C(01)-C(02)	1.517(5)
N(15)-C(16)	1.377(4)	C(01)-C(06)	1.524(5)
N(15)-C(14)	1.384(5)	N(23)-C(22)	1.343(4)
N(15)-C(115)	1.475(5)	N(23)-C(24)	1.401(5)
C(11)-C(21)	1.507(5)	N(23)-C(123)	1.463(5)
C(11)-C(16)	1.524(5)	N(25)-C(24)	1.379(5)
C(11)-C(12)	1.525(5)	N(25)-C(26)	1.407(5)
C(11)-C(01)	1.613(5)	N(25)-C(125)	1.471(5)
O(102)-C(02)	1.209(4)	C(21)-C(22)	1.339(5)
O(104)-C(04)	1.204(4)	C(21)-C(26)	1.417(6)
O(106)-C(06)	1.204(4)	C(22)-O(122)	1.360(4)
N(105)-C(06)	1.377(5)	C(24)-O(124)	1.214(4)
N(105)-C(04)	1.387(5)	C(26)-O(126)	1.225(5)

C(12)-N(13)-C(14)	124.6(3)	N(13)-C(12)-C(11)	116.1(3)
C(12)-N(13)-C(113)	119.0(3)	O(114)-C(14)-N(15)	121.8(4)
C(14)-N(13)-C(113)	116.3(3)	O(114)-C(14)-N(13)	121.0(4)
C(16)-N(15)-C(14)	125.5(3)	N(15)-C(14)-N(13)	117.2(3)
C(16)-N(15)-C(115)	117.6(3)	O(116)-C(16)-N(15)	122.7(3)
C(14)-N(15)-C(115)	116.4(3)	O(116)-C(16)-C(11)	121.9(3)
C(21)-C(11)-C(16)	114.5(3)	N(15)-C(16)-C(11)	115.2(3)
C(21)-C(11)-C(12)	110.1(3)	C(06)-N(105)-C(04)	124.1(3)
C(16)-C(11)-C(12)	113.6(3)	C(06)-N(105)-C(105)	117.8(3)
C(21)-C(11)-C(01)	97.4(3)	C(04)-N(105)-C(105)	117.7(3)
C(16)-C(11)-C(01)	110.6(3)	C(02)-N(03)-C(04)	124.6(3)
C(12)-C(11)-C(01)	109.4(3)	C(02)-N(03)-C(103)	117.8(3)
O(112)-C(12)-N(13)	122.1(3)	C(04)-N(03)-C(103)	116.5(3)
O(112)-C(12)-C(11)	121.6(3)	O(122)-C(01)-C(02)	110.2(3)

O(122)-C(01)-C(06)	107.0(3)	O(104)-C(04)-N(105)	121.4(4)
C(02)-C(01)-C(06)	111.9(3)	O(104)-C(04)-N(03)	121.5(3)
O(122)-C(01)-C(11)	105.5(3)	N(105)-C(04)-N(03)	117.2(3)
C(02)-C(01)-C(11)	111.9(3)	O(106)-C(06)-N(105)	123.7(3)
C(06)-C(01)-C(11)	110.1(3)	O(106)-C(06)-C(01)	122.0(3)
O(102)-C(02)-N(03)	123.5(3)	N(105)-C(06)-C(01)	114.2(3)
O(102)-C(02)-C(01)	121.8(3)	C(22)-N(23)-C(24)	118.0(3)
N(03)-C(02)-C(01)	114.4(3)	C(22)-N(23)-C(123)	123.8(3)
C(24)-N(23)-C(123)	118.1(3)	N(23)-C(22)-O(122)	118.8(3)
C(24)-N(25)-C(26)	126.0(3)	O(124)-C(24)-N(25)	123.2(4)
C(24)-N(25)-C(125)	115.8(3)	O(124)-C(24)-N(23)	120.2(4)
C(26)-N(25)-C(125)	118.1(4)	N(25)-C(24)-N(23)	116.6(3)
C(22)-C(21)-C(26)	120.0(3)	O(126)-C(26)-N(25)	120.9(4)
C(22)-C(21)-C(11)	108.8(3)	O(126)-C(26)-C(21)	125.7(3)
C(26)-C(21)-C(11)	129.5(3)	N(25)-C(26)-C(21)	113.4(4)
C(21)-C(22)-N(23)	125.7(4)	C(22)-O(122)-C(01)	105.7(3)
C(21)-C(22)-O(122)	115.5(3)		

7.3.25 Crystal data for $C_{19}H_{30}KN_3O_9$ **(59)**

Table 73: Crystal data and structure refinement for $C_{19}H_{30}KN_3O$ **(59)**

Empirical formula	$C_{19}H_{30}KN_3O$	
Formula weight	483.56	
Temperature	213(2) K	
Wavelength	1.54184 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 8.137 (2) Å	α= 90°.
	b = 15.175(3) Å	β= 101.88(3)°
	c = 9.941(2) Å	γ = 90°.
Volume	1201.2(4) $Å^3$	
Z	2	
Density (calculated)	1.337 Mg/m^3	
Absorption coefficient	2.395 mm^{-1}	
F(000)	512	
Crystal size	0.30 x 0.25 x 0.20 mm^3	
Theta range for data collection	5.40 to 64.99°.	
Index ranges	-1<=h<=9, -17<=k<=15, -11<=l<=11	
Reflections collected	4911	
Independent reflections	4015 [R(int) = 0.0285]	
Completeness to theta = 64.99°	99.8 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4015 / 1 / 322	
Goodness-of-fit on F^2	1.049	
Final R indices [I>2sigma(I)]	R1 = 0.0531, wR2 = 0.1377	
R indices (all data)	R1 = 0.0565, wR2 = 0.1405	
Absolute structure parameter	0.004(14)	
Extinction coefficient	0.0010(5)	
Largest diff. peak and hole	0.329 and -0.331 $e.Å^{-3}$	

Table 74: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{19}H_{30}KN_3O$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
K(1)	1168(1)	1435(1)	9396(1)	46(1)
O(1)	-1477(6)	1131(3)	10729(7)	122(2)
O(2)	1450(7)	1989(4)	12103(4)	110(2)
O(3)	3354(6)	2830(3)	10467(4)	87(1)
O(4)	3598(4)	1963(3)	7992(4)	78(1)
O(5)	852(6)	950(2)	6630(3)	85(1)
O(6)	-1438(5)	284(3)	8158(6)	106(2)
C(1)	-809(12)	1145(9)	12265(10)	150(5)
C(2)	111(16)	1941(8)	12682(9)	149(5)
C(3)	2511(15)	2746(5)	12548(6)	136(4)
C(4)	3929(12)	2765(5)	11859(8)	114(3)
C(5)	4710(8)	2907(5)	9708(11)	114(3)
C(6)	4070(9)	2836(5)	8272(9)	108(2)
C(7)	3162(9)	1806(7)	6546(7)	109(3)
C(8)	2426(12)	936(6)	6252(7)	123(4)
C(9)	-165(18)	167(4)	6309(6)	150(5)
C(10)	-1814(14)	299(5)	6802(13)	174(7)
C(11)	-2976(8)	453(5)	8813(17)	161(6)
C(12)	-2428(14)	452(9)	10340(20)	228(10)
N(1)	-2357(4)	3467(2)	6233(3)	46(1)
N(2)	-1850(4)	3928(2)	8533(3)	47(1)
N(3)	-5805(6)	6010(3)	6132(4)	69(1)
O(7)	-516(5)	2743(2)	7888(4)	75(1)
O(8)	-3137(4)	5144(2)	9182(3)	53(1)
O(9)	-4282(4)	4159(2)	4595(3)	55(1)
C(13)	-1516(5)	3351(3)	7559(4)	49(1)
C(14)	-3550(5)	4134(2)	5800(4)	41(1)
C(15)	-3773(4)	4729(2)	6844(4)	40(1)
C(16)	-2951(4)	4653(2)	8241(4)	42(1)
C(17)	-1988(7)	2853(3)	5187(5)	70(1)
C(18)	-4912(5)	5434(3)	6457(4)	48(1)
C(19)	-1020(6)	3762(4)	9961(4)	66(1)

Table 75: Bond lengths [Å] and angles [°] for $C_{19}H_{30}KN_3O$

K(1)-O(7)	2.687(3)	C(1)-C(2)	1.436(16)
K(1)-O(8)#1	2.733(3)	C(3)-C(4)	1.459(13)
K(1)-O(4)	2.764(3)	C(5)-C(6)	1.420(11)
K(1)-O(2)	2.784(4)	C(7)-C(8)	1.454(11)
K(1)-O(1)	2.789(4)	C(9)-C(10)	1.534(15)
K(1)-O(5)	2.807(3)	C(11)-C(12)	1.50(2)
K(1)-O(6)	2.826(4)	N(1)-C(13)	1.366(5)
K(1)-O(3)	2.829(4)	N(1)-C(14)	1.406(5)
O(1)-C(12)	1.297(16)	N(1)-C(17)	1.472(5)
O(1)-C(1)	1.511(11)	N(2)-C(13)	1.373(5)
O(2)-C(2)	1.336(11)	N(2)-C(16)	1.411(5)
O(2)-C(3)	1.450(11)	N(2)-C(19)	1.463(5)
O(3)-C(4)	1.370(8)	N(3)-C(18)	1.140(6)
O(3)-C(5)	1.463(9)	O(7)-C(13)	1.229(5)
O(4)-C(6)	1.391(8)	O(8)-C(16)	1.229(5)
O(4)-C(7)	1.428(8)	O(8)-K(1)#2	2.733(3)
O(5)-C(8)	1.407(10)	O(9)-C(14)	1.224(5)
O(5)-C(9)	1.445(10)	C(14)-C(15)	1.415(5)
O(6)-C(10)	1.320(13)	C(15)-C(18)	1.416(5)
O(6)-C(11)	1.547(13)	C(15)-C(16)	1.417(5)

O(7)-K(1)-O(8)#1	174.48(11)	O(4)-K(1)-O(6)	120.67(17)
O(7)-K(1)-O(4)	80.75(12)	O(2)-K(1)-O(6)	120.87(19)
O(8)#1-K(1)-O(4)	93.73(10)	O(1)-K(1)-O(6)	61.4(2)
O(7)-K(1)-O(2)	104.32(12)	O(5)-K(1)-O(6)	60.13(17)
O(8)#1-K(1)-O(2)	78.06(11)	O(7)-K(1)-O(3)	82.28(12)
O(4)-K(1)-O(2)	118.38(16)	O(8)#1-K(1)-O(3)	94.89(10)
O(7)-K(1)-O(1)	91.88(13)	O(4)-K(1)-O(3)	60.85(14)
O(8)#1-K(1)-O(1)	93.62(12)	O(2)-K(1)-O(3)	59.31(17)
O(4)-K(1)-O(1)	172.10(13)	O(1)-K(1)-O(3)	115.60(18)
O(2)-K(1)-O(1)	60.6(2)	O(5)-K(1)-O(3)	119.26(14)
O(7)-K(1)-O(5)	73.37(11)	O(6)-K(1)-O(3)	169.68(11)
O(8)#1-K(1)-O(5)	104.17(10)	C(12)-O(1)-C(1)	112.2(10)
O(4)-K(1)-O(5)	60.80(13)	C(12)-O(1)-K(1)	117.6(7)
O(2)-K(1)-O(5)	177.58(14)	C(1)-O(1)-K(1)	109.0(4)
O(1)-K(1)-O(5)	119.89(18)	C(2)-O(2)-C(3)	113.5(8)
O(7)-K(1)-O(6)	87.88(12)	C(2)-O(2)-K(1)	119.2(7)
O(8)#1-K(1)-O(6)	95.17(10)	C(3)-O(2)-K(1)	117.3(4)

C(4)-O(3)-C(5)	112.9(7)	O(5)-C(9)-C(10)	108.8(6)
C(4)-O(3)-K(1)	112.6(4)	O(6)-C(10)-C(9)	106.8(6)
C(5)-O(3)-K(1)	110.4(4)	C(12)-C(11)-O(6)	109.4(6)
C(6)-O(4)-C(7)	111.2(6)	O(1)-C(12)-C(11)	109.5(10)
C(6)-O(4)-K(1)	111.9(4)	C(13)-N(1)-C(14)	124.4(3)
C(7)-O(4)-K(1)	113.9(4)	C(13)-N(1)-C(17)	117.8(3)
C(8)-O(5)-C(9)	116.1(7)	C(14)-N(1)-C(17)	117.9(3)
C(8)-O(5)-K(1)	111.1(4)	C(13)-N(2)-C(16)	124.2(3)
C(9)-O(5)-K(1)	111.4(4)	C(13)-N(2)-C(19)	117.1(4)
C(10)-O(6)-C(11)	112.9(8)	C(16)-N(2)-C(19)	118.7(3)
C(10)-O(6)-K(1)	115.5(5)	C(13)-O(7)-K(1)	159.5(3)
C(11)-O(6)-K(1)	108.3(4)	C(16)-O(8)-K(1)#2	132.6(3)
C(2)-C(1)-O(1)	111.2(7)	O(7)-C(13)-N(1)	121.7(4)
O(2)-C(2)-C(1)	110.1(8)	O(7)-C(13)-N(2)	120.5(4)
O(2)-C(3)-C(4)	110.6(6)	N(1)-C(13)-N(2)	117.8(3)
O(3)-C(4)-C(3)	109.7(6)	O(9)-C(14)-N(1)	119.2(3)
C(6)-C(5)-O(3)	110.7(5)	O(9)-C(14)-C(15)	126.0(3)
O(4)-C(6)-C(5)	107.6(6)	N(1)-C(14)-C(15)	114.8(3)
O(4)-C(7)-C(8)	111.1(6)	C(14)-C(15)-C(18)	117.3(3)
O(5)-C(8)-C(7)	107.0(5)	C(14)-C(15)-C(16)	124.2(3)
C(18)-C(15)-C(16)	118.5(3)	N(2)-C(16)-C(15)	114.6(3)
O(8)-C(16)-N(2)	119.3(3)	N(3)-C(18)-C(15)	178.5(5)
O(8)-C(16)-C(15)	126.1(4)		

7.3.26 Crystal data for $C_{15}H_{18}N_4O_7$ **(60)**

Table 76: Crystal data and structure refinement for $C_{15}H_{18}N_4O_7$ **(60)**

Empirical formula	$C_{15}H_{18}N_4O_7$	
Formula weight	366.33	
Temperature	231(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 9.253 (2) Å	α= 90°.
	b = 13.179(3) Å	β= 90°.
	c = 13.360(3) Å	γ = 90°.
Volume	1629.2(6) $Å^3$	
Z	4	
Density (calculated)	1.494 Mg/m^3	
Absorption coefficient	0.120 mm^{-1}	
F(000)	768	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.05 to 30.91°.	
Index ranges	-1<=h<=13, -1<=k<=19, -19<=l<=19	
Reflections collected	6410	
Independent reflections	5133 [R(int) = 0.0402]	
Completeness to theta = 30.91°	99.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5133 / 0 / 241	
Goodness-of-fit on F^2	0.989	
Final R indices [I>2sigma(I)]	R1 = 0.0583, wR2 = 0.0941	
R indices (all data)	R1 = 0.1862, wR2 = 0.1232	
Absolute structure parameter	-1.1(16)	
Extinction coefficient	0.0043(13)	
Largest diff. peak and hole	0.185 and -0.176 $e.Å^{-3}$	

Table 77: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{15}H_{18}N_4O_7$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	1812(3)	5024(2)	922(2)	38(1)
N(2)	2637(3)	3806(2)	-260(2)	40(1)
N(3)	6558(3)	6948(2)	2315(2)	43(1)
N(4)	5209(3)	8212(2)	1473(2)	42(1)
O(1)	3231(3)	6292(2)	1459(2)	57(1)
O(2)	384(3)	3736(2)	383(2)	67(1)
O(3)	4835(3)	3945(2)	-937(2)	59(1)
O(4)	7064(2)	5451(2)	1563(2)	49(1)
O(5)	5993(3)	8428(2)	3058(2)	60(1)
O(6)	4263(2)	7906(2)	-52(2)	52(1)
O(7)	3342(3)	6095(2)	-1231(2)	54(1)
C(1)	3086(4)	5526(2)	948(2)	40(1)
C(2)	4326(3)	5086(2)	392(2)	37(1)
C(3)	3974(4)	4250(2)	-330(2)	41(1)
C(4)	1551(4)	4152(2)	347(2)	41(1)
C(5)	634(4)	5371(2)	1582(2)	54(1)
C(6)	2332(4)	2915(3)	-893(3)	58(1)
C(7)	6559(3)	6295(2)	1510(2)	39(1)
C(8)	5953(3)	6701(2)	545(2)	36(1)
C(9)	5030(3)	7635(2)	631(2)	39(1)
C(10)	5931(4)	7901(2)	2324(2)	45(1)
C(11)	7253(4)	6633(3)	3252(2)	60(1)
C(12)	4579(4)	9232(2)	1505(3)	60(1)
C(13)	5319(3)	5859(2)	-127(2)	38(1)
C(14)	4605(4)	6271(2)	-1071(2)	43(1)
C(15)	5559(4)	6769(2)	-1827(2)	58(1)

Table 78 Bond lengths [Å] and angles [°] for $C_{15}H_{18}N_4O_7$

N(1)-C(1)	1.352(4)	N(2)-C(4)	1.369(4)
N(1)-C(4)	1.404(4)	N(2)-C(3)	1.371(4)
N(1)-C(5)	1.474(4)	N(2)-C(6)	1.475(4)

N(3)-C(7)	1.376(4)	O(6)-C(9)	1.210(3)
N(3)-C(10)	1.383(4)	O(7)-C(14)	1.210(4)
N(3)-C(11)	1.467(4)	C(1)-C(2)	1.485(4)
N(4)-C(9)	1.368(4)	C(2)-C(3)	1.500(4)
N(4)-C(10)	1.381(4)	C(2)-C(13)	1.537(4)
N(4)-C(12)	1.466(4)	C(7)-C(8)	1.504(4)
O(1)-C(1)	1.226(3)	C(8)-C(9)	1.503(4)
O(2)-C(4)	1.211(3)	C(8)-C(13)	1.543(4)
O(3)-C(3)	1.206(3)	C(13)-C(14)	1.524(4)
O(4)-C(7)	1.209(3)	C(14)-C(15)	1.493(4)
O(5)-C(10)	1.203(3)		

C(1)-N(1)-C(4)	124.4(3)	O(2)-C(4)-N(2)	121.8(3)
C(1)-N(1)-C(5)	118.5(3)	O(2)-C(4)-N(1)	120.1(3)
C(4)-N(1)-C(5)	117.0(3)	N(2)-C(4)-N(1)	118.1(3)
C(4)-N(2)-C(3)	124.1(3)	O(4)-C(7)-N(3)	122.0(3)
C(4)-N(2)-C(6)	117.6(3)	O(4)-C(7)-C(8)	121.4(3)
C(3)-N(2)-C(6)	118.2(3)	N(3)-C(7)-C(8)	116.6(3)
C(7)-N(3)-C(10)	125.0(3)	C(9)-C(8)-C(7)	115.9(2)
C(7)-N(3)-C(11)	119.3(3)	C(9)-C(8)-C(13)	114.6(2)
C(10)-N(3)-C(11)	115.7(3)	C(7)-C(8)-C(13)	112.7(2)
C(9)-N(4)-C(10)	124.8(3)	O(6)-C(9)-N(4)	121.8(3)
C(9)-N(4)-C(12)	119.0(3)	O(6)-C(9)-C(8)	121.2(3)
C(10)-N(4)-C(12)	116.2(3)	N(4)-C(9)-C(8)	116.7(3)
O(1)-C(1)-N(1)	120.9(3)	O(5)-C(10)-N(4)	121.5(3)
O(1)-C(1)-C(2)	121.0(3)	O(5)-C(10)-N(3)	120.8(3)
N(1)-C(1)-C(2)	118.0(3)	N(4)-C(10)-N(3)	117.7(3)
C(1)-C(2)-C(3)	116.2(3)	C(14)-C(13)-C(2)	110.5(2)
C(1)-C(2)-C(13)	115.4(2)	C(14)-C(13)-C(8)	113.0(2)
C(3)-C(2)-C(13)	109.1(2)	C(2)-C(13)-C(8)	116.2(2)
O(3)-C(3)-N(2)	120.0(3)	O(7)-C(14)-C(15)	122.4(3)
O(3)-C(3)-C(2)	122.3(3)	O(7)-C(14)-C(13)	119.7(3)
N(2)-C(3)-C(2)	117.7(3)	C(15)-C(14)-C(13)	117.4(3)

7.3.27 Crystal data for $C_{16}H_{26}N_4O_{10}S_2$ **(61)**

Table 79: Crystal data and structure refinement for $C_{16}H_{26}N_4O_{10}S_2$ **(61)**

Empirical formula	$C_{16}H_{26}N_4O_{10}S_2$	
Formula weight	498.53	
Temperature	208(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 8.3150(17) Å	α= 90°.
	b = 15.281(3) Å	β= 90°.
	c = 17.383(4) Å	γ = 90°.
Volume	2208.7(8) $Å^3$	
Z	4	
Density (calculated)	1.499 Mg/m^3	
Absorption coefficient	0.302 mm^{-1}	
F(000)	1048	
Crystal size	0.25 x0.25 x 0.15 mm^3	
Theta range for data collection	3.03 to 31.00°.	
Index ranges	-1<=h<=5, -1<=k<=22, -25<=l<=25	
Reflections collected	5926	
Independent reflections	4163 [R(int) = 0.0379]	
Completeness to theta = 31.00°	61.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4163 / 0 / 391	
Goodness-of-fit on F^2	0.956	
Final R indices [I>2sigma(I)]	R1 = 0.0450, wR2 = 0.0950	
R indices (all data)	R1 = 0.1058, wR2 = 0.1058	
Absolute structure parameter	-0.06(10)	
Extinction coefficient	0.0000(6)	
Largest diff. peak and hole	0.615 and -0.348 $e.Å^{-3}$	

Table 80: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{16}H_{26}N_4O_{10}S_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
S(1)	993(2)	6534(1)	777(1)	38(1)
S(2)	5191(2)	5681(1)	1183(1)	46(1)
N(1)	8629(5)	8359(2)	2591(1)	25(1)
N(2)	11399(4)	8351(2)	2254(2)	24(1)
N(3)	10557(5)	11062(2)	725(2)	22(1)
N(4)	7800(5)	11052(2)	1029(2)	24(1)
O(1)	9068(4)	9291(2)	729(1)	31(1)
O(2)	6809(5)	8834(2)	1723(2)	39(1)
O(3)	10511(4)	7757(2)	3362(2)	53(1)
O(4)	12135(4)	8866(2)	1091(2)	46(1)
O(5)	10023(4)	10114(2)	2624(1)	30(1)
O(6)	7058(4)	10707(2)	2245(1)	37(1)
O(7)	8731(4)	11529(2)	-121(1)	49(1)
O(8)	12357(5)	10733(2)	1667(2)	41(1)
O(9)	6121(5)	5653(2)	453(1)	57(1)
O(10)	147(4)	6576(2)	1542(1)	37(1)
C(1)	9515(5)	9254(2)	1498(2)	20(1)
C(2)	8170(7)	8790(2)	1935(2)	25(1)
C(3)	10215(7)	8150(2)	2771(2)	28(1)
C(4)	11120(6)	8811(2)	1583(2)	27(1)
C(5)	7415(9)	8066(3)	3129(3)	39(2)
C(6)	12963(7)	7936(3)	2359(3)	40(1)
C(7)	9654(5)	10226(2)	1850(2)	18(1)
C(8)	10997(7)	10701(2)	1426(2)	26(1)
C(9)	9014(6)	11214(2)	504(2)	22(1)
C(10)	8061(6)	10683(2)	1742(2)	22(1)
C(11)	11881(7)	11367(3)	232(2)	35(1)
C(12)	6212(8)	11375(3)	857(3)	41(2)
C(13)	-352(11)	7011(4)	112(3)	64(2)
C(14)	750(12)	5432(3)	466(4)	60(2)
C(15)	5889(9)	4792(3)	1740(2)	39(2)
C(16)	6069(10)	6534(3)	1731(2)	45(2)

Table 81: Bond lengths [Å] and angles [°] for $C_{16}H_{26}N_4O_{10}S_2$

S(1)-O(10)	1.507(3)	N(4)-C(9)	1.382(5)
S(1)-C(13)	1.766(6)	N(4)-C(12)	1.441(7)
S(1)-C(14)	1.780(5)	O(1)-C(1)	1.389(4)
S(2)-O(9)	1.487(3)	O(2)-C(2)	1.192(5)
S(2)-C(15)	1.766(4)	O(3)-C(3)	1.214(4)
S(2)-C(16)	1.773(5)	O(4)-C(4)	1.205(5)
N(1)-C(2)	1.372(5)	O(5)-C(7)	1.390(4)
N(1)-C(3)	1.393(5)	O(6)-C(10)	1.208(5)
N(1)-C(5)	1.446(6)	O(7)-C(9)	1.211(4)
N(2)-C(3)	1.369(5)	O(8)-C(8)	1.207(5)
N(2)-C(4)	1.380(4)	C(1)-C(4)	1.504(6)
N(2)-C(6)	1.459(6)	C(1)-C(2)	1.527(6)
N(3)-C(9)	1.359(5)	C(1)-C(7)	1.610(4)
N(3)-C(8)	1.386(5)	C(7)-C(10)	1.510(6)
N(3)-C(11)	1.471(6)	C(7)-C(8)	1.523(6)
N(4)-C(10)	1.380(4)		

O(10)-S(1)-C(13)	105.3(3)	C(10)-N(4)-C(12)	118.0(4)
O(10)-S(1)-C(14)	104.8(2)	C(9)-N(4)-C(12)	118.2(4)
C(13)-S(1)-C(14)	96.9(4)	O(1)-C(1)-C(4)	110.6(3)
O(9)-S(2)-C(15)	105.9(2)	O(1)-C(1)-C(2)	107.6(3)
O(9)-S(2)-C(16)	105.4(2)	C(4)-C(1)-C(2)	113.1(3)
C(15)-S(2)-C(16)	97.8(2)	O(1)-C(1)-C(7)	110.4(2)
C(2)-N(1)-C(3)	124.1(4)	C(4)-C(1)-C(7)	108.3(3)
C(2)-N(1)-C(5)	119.5(5)	C(2)-C(1)-C(7)	106.9(3)
C(3)-N(1)-C(5)	116.4(4)	O(2)-C(2)-N(1)	123.3(4)
C(3)-N(2)-C(4)	123.3(4)	O(2)-C(2)-C(1)	121.0(3)
C(3)-N(2)-C(6)	117.4(4)	N(1)-C(2)-C(1)	115.7(4)
C(4)-N(2)-C(6)	118.5(4)	O(3)-C(3)-N(2)	121.4(5)
C(9)-N(3)-C(8)	124.4(4)	O(3)-C(3)-N(1)	119.7(4)
C(9)-N(3)-C(11)	119.2(3)	N(2)-C(3)-N(1)	118.8(3)
C(8)-N(3)-C(11)	116.2(4)	O(4)-C(4)-N(2)	121.2(4)
C(10)-N(4)-C(9)	123.4(4)	O(4)-C(4)-C(1)	121.4(3)

N(2)-C(4)-C(1)	117.5(3)	O(8)-C(8)-C(7)	122.5(3)
O(5)-C(7)-C(10)	111.8(3)	N(3)-C(8)-C(7)	115.0(4)
O(5)-C(7)-C(8)	111.4(3)	O(7)-C(9)-N(3)	120.3(4)
C(10)-C(7)-C(8)	111.2(3)	O(7)-C(9)-N(4)	121.4(5)
O(5)-C(7)-C(1)	105.7(2)	N(3)-C(9)-N(4)	118.2(3)
C(10)-C(7)-C(1)	108.5(3)	O(6)-C(10)-N(4)	121.9(4)
C(8)-C(7)-C(1)	107.9(3)	O(6)-C(10)-C(7)	122.0(3)
O(8)-C(8)-N(3)	122.4(4)	N(4)-C(10)-C(7)	116.1(3)

7.3.28 Crystal data for $C_{17}H_{28}N_4O_3$ **(62)**

Table 82: Crystal data and structure refinement for $C_{17}H_{28}N_4O_3$ **(62)**

Empirical formula	$C_{17}H_{28}N_4O_3$	
Formula weight	336.43	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 12.625(3) Å	α= 90°.
	b = 10.046(2) Å	β= 111.81(3)°
	c = 15.605(3) Å	γ = 90°.
Volume	1837.5(6) $Å^3$	
Z	4	
Density (calculated)	1.216 Mg/m^3	
Absorption coefficient	0.085 mm^{-1}	
F(000)	728	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.31 to 31.05°.	
Index ranges	-1<=h<=18, 0<=k<=14, -22<=l<=21	
Reflections collected	6548	
Independent reflections	5845 [R(int) = 0.0552]	
Completeness to theta = 31.05°	99.2 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5845 / 0 / 330	
Goodness-of-fit on F^2	1.016	
Final R indices [I>2sigma(I)]	R1 = 0.0542, wR2 = 0.1306	
R indices (all data)	R1 = 0.1096, wR2 = 0.1536	
Extinction coefficient	0.0034(14)	
Largest diff. peak and hole	0.299 and -0.216 $e.Å^{-3}$	

Table 83: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{17}H_{28}N_4O_3$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	3501(1)	69(1)	1637(1)	38(1)
N(2)	1714(1)	370(1)	1735(1)	32(1)
N(3)	2297(1)	4379(1)	697(1)	31(1)
N(4)	1750(1)	5050(1)	1781(1)	31(1)
O(1)	5068(1)	1164(2)	2608(1)	55(1)
O(2)	1917(1)	-956(1)	625(1)	51(1)
O(3)	1500(1)	1552(1)	2904(1)	42(1)
C(1)	4027(1)	945(2)	2387(1)	38(1)
C(2)	3328(1)	1468(2)	2815(1)	38(1)
C(3)	2169(1)	1178(2)	2534(1)	32(1)
C(4)	2354(1)	-218(2)	1290(1)	34(1)
C(5)	494(2)	87(2)	1379(1)	41(1)
C(6)	4212(2)	-606(3)	1214(2)	56(1)
C(7)	2571(1)	5144(2)	1443(1)	33(1)
C(8)	1262(1)	3757(2)	551(1)	30(1)
C(9)	918(1)	4181(1)	1232(1)	30(1)
C(10)	3008(1)	4203(2)	130(1)	35(1)
C(11)	3890(2)	3139(2)	555(2)	57(1)
C(12)	3521(2)	5522(2)	17(1)	45(1)
C(13)	731(2)	2794(2)	-209(1)	38(1)
C(14)	-126(1)	3835(2)	1413(1)	38(1)
C(15)	1754(1)	5729(2)	2626(1)	35(1)
C(16)	2267(2)	4831(2)	3457(1)	46(1)
C(17)	2361(2)	7062(2)	2741(2)	54(1)

Table 84: Bond lengths [Å] and angles [°] for $C_{17}H_{28}N_4O_3$

N(1)-C(4)	1.374(2)	O(1)-C(1)	1.247(2)
N(1)-C(1)	1.417(2)	O(2)-C(4)	1.2269(19)
N(1)-C(6)	1.464(2)	O(3)-C(3)	1.2447(19)
N(2)-C(4)	1.378(2)	C(1)-C(2)	1.393(2)
N(2)-C(3)	1.419(2)	C(2)-C(3)	1.394(2)
N(2)-C(5)	1.459(2)	C(8)-C(9)	1.358(2)
N(3)-C(7)	1.3296(19)	C(8)-C(13)	1.483(2)
N(3)-C(8)	1.3879(18)	C(9)-C(14)	1.488(2)
N(3)-C(10)	1.4873(19)	C(10)-C(11)	1.508(3)
N(4)-C(7)	1.3285(19)	C(10)-C(12)	1.513(2)
N(4)-C(9)	1.3903(19)	C(15)-C(16)	1.513(3)
N(4)-C(15)	1.4828(19)	C(15)-C(17)	1.520(3)
C(4)-N(1)-C(1)	123.91(13)	C(2)-C(3)-N(2)	115.50(14)
C(4)-N(1)-C(6)	117.26(16)	O(2)-C(4)-N(1)	122.46(15)
C(1)-N(1)-C(6)	118.81(15)	O(2)-C(4)-N(2)	121.41(14)
C(4)-N(2)-C(3)	124.53(13)	N(1)-C(4)-N(2)	116.13(14)
C(4)-N(2)-C(5)	117.31(13)	N(4)-C(7)-N(3)	108.64(13)
C(3)-N(2)-C(5)	118.10(13)	C(9)-C(8)-N(3)	106.58(13)
C(7)-N(3)-C(8)	109.07(12)	C(9)-C(8)-C(13)	130.44(14)
C(7)-N(3)-C(10)	124.90(12)	N(3)-C(8)-C(13)	122.94(13)
C(8)-N(3)-C(10)	126.01(12)	C(8)-C(9)-N(4)	106.92(12)
C(7)-N(4)-C(9)	108.78(12)	C(8)-C(9)-C(14)	129.74(14)
C(7)-N(4)-C(15)	125.12(13)	N(4)-C(9)-C(14)	123.34(13)
C(9)-N(4)-C(15)	126.07(12)	N(3)-C(10)-C(11)	109.42(13)
O(1)-C(1)-C(2)	126.25(17)	N(3)-C(10)-C(12)	110.17(14)
O(1)-C(1)-N(1)	117.38(16)	C(11)-C(10)-C(12)	113.16(16)
C(2)-C(1)-N(1)	116.37(14)	N(4)-C(15)-C(16)	110.30(14)
C(1)-C(2)-C(3)	123.27(15)	N(4)-C(15)-C(17)	110.15(14)
O(3)-C(3)-C(2)	127.23(15)	C(16)-C(15)-C(17)	112.79(16)
O(3)-C(3)-N(2)	117.26(14)		

7.3.29 Crystal data for $C_{11}H_{17}N_5O_3$ **(64)**

Table 85: Crystal data and structure refinement for $C_{11}H_{17}N_5O_3$ **(64)**

Empirical formula	$C_{11}H_{17}N_5O_3$	
Formula weight	267.30	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 14.026(3) Å	α= 90°.
	b = 7.9080(16) Å	β= 90°.
	c = 23.341(5) Å	γ = 90°.
Volume	2588.9(9) $Å^3$	
Z	8	
Density (calculated)	1.372 Mg/m^3	
Absorption coefficient	0.103 mm^{-1}	
F(000)	1136	
Crystal size	0.6 x 0.3 x 0.3 mm^3	
Theta range for data collection	3.08 to 30.98°.	
Index ranges	-1<=h<=20, -1<=k<=11, -33<=l<=1	
Reflections collected	5167	
Independent reflections	4112 [R(int) = 0.0379]	
Completeness to theta = 30.98°	99.7 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4112 / 0 / 241	
Goodness-of-fit on F^2	1.008	
Final R indices [I>2sigma(I)]	R1 = 0.0515, wR2 = 0.1131	
R indices (all data)	R1 = 0.1268, wR2 = 0.1370	
Extinction coefficient	0.0051(8)	
Largest diff. peak and hole	0.230 and -0.196 $e.Å^{-3}$	

Table 86: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{11}H_{17}N_5O_3$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	5786(1)	5056(2)	3176(1)	31(1)
N(2)	6335(1)	2435(2)	3510(1)	30(1)
N(3)	7605(1)	4707(2)	6211(1)	32(1)
N(4)	8285(1)	3716(2)	5446(1)	34(1)
N(5)	6597(1)	3459(2)	5508(1)	34(1)
O(1)	6237(1)	1734(2)	4455(1)	37(1)
O(2)	6512(1)	3200(2)	2575(1)	41(1)
O(3)	5056(1)	6931(2)	3779(1)	39(1)
C(1)	6059(1)	2815(2)	4078(1)	28(1)
C(2)	5615(1)	4365(2)	4163(1)	32(1)
C(3)	5463(1)	5526(2)	3727(1)	30(1)
C(4)	6227(1)	3540(2)	3059(1)	30(1)
C(5)	5682(2)	6248(3)	2701(1)	50(1)
C(6)	6767(2)	777(3)	3404(1)	47(1)
C(7)	7443(1)	3909(2)	5709(1)	29(1)
C(8)	8580(1)	5024(3)	6259(1)	40(1)
C(9)	8993(1)	4420(3)	5789(1)	41(1)
C(10)	6875(2)	5229(3)	6618(1)	41(1)
C(11)	8426(2)	2922(3)	4891(1)	46(1)

Table 87: Bond lengths [Å] and angles [°] for $C_{11}H_{17}N_5O_3$

N(1)-C(4)	1.376(2)	N(4)-C(9)	1.393(2)
N(1)-C(3)	1.413(2)	N(4)-C(11)	1.453(3)
N(1)-C(5)	1.463(2)	N(5)-C(7)	1.325(2)
N(2)-C(4)	1.377(2)	O(1)-C(1)	1.252(2)
N(2)-C(1)	1.412(2)	O(2)-C(4)	1.229(2)
N(2)-C(6)	1.466(2)	O(3)-C(3)	1.255(2)
N(3)-C(7)	1.351(2)	C(1)-C(2)	1.388(2)
N(3)-C(8)	1.395(2)	C(2)-C(3)	1.387(2)
N(3)-C(10)	1.457(2)	C(8)-C(9)	1.328(3)
N(4)-C(7)	1.339(2)		
C(4)-N(1)-C(3)	123.65(14)	C(2)-C(1)-N(2)	116.42(15)
C(4)-N(1)-C(5)	117.10(15)	C(3)-C(2)-C(1)	123.28(15)
C(3)-N(1)-C(5)	119.22(16)	O(3)-C(3)-C(2)	125.83(15)
C(4)-N(2)-C(1)	123.48(15)	O(3)-C(3)-N(1)	117.84(16)
C(4)-N(2)-C(6)	118.89(15)	C(2)-C(3)-N(1)	116.32(15)
C(1)-N(2)-C(6)	117.62(15)	O(2)-C(4)-N(1)	121.35(16)
C(7)-N(3)-C(8)	108.52(15)	O(2)-C(4)-N(2)	121.87(16)
C(7)-N(3)-C(10)	125.49(15)	N(1)-C(4)-N(2)	116.78(14)
C(8)-N(3)-C(10)	125.91(16)	N(5)-C(7)-N(4)	126.62(16)
C(7)-N(4)-C(9)	108.61(15)	N(5)-C(7)-N(3)	125.68(16)
C(7)-N(4)-C(11)	125.33(16)	N(4)-C(7)-N(3)	107.66(14)
C(9)-N(4)-C(11)	126.05(17)	C(9)-C(8)-N(3)	107.31(17)
O(1)-C(1)-C(2)	126.29(15)	C(8)-C(9)-N(4)	107.90(17)
O(1)-C(1)-N(2)	117.29(15)		

7.3.30 Crystal data for $C_{24}H_{28}N_8O_{12}$ **(65)**

Table 88: Crystal data and structure refinement for $C_{24}H_{28}N_8O_{12}$ **(65)**

Empirical formula	$C_{24}H_{28}N_8O_{12}$	
Formula weight	620.54	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	I4(1)/a	
Unit cell dimensions	a = 14.112(2) Å	α= 90°.
	b = 14.112(2) Å	β= 90°.
	c = 13.642(3) Å	γ = 90°.
Volume	2717.0(8) $Å^3$	
Z	4	
Density (calculated)	1.517 Mg/m^3	
Absorption coefficient	0.124 mm^{-1}	
F(000)	1296	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.56 to 29.34°.	
Index ranges	-19<=h<=19, -18<=k<=19, -18<=l<=18	
Reflections collected	12492	
Independent reflections	1857 [R(int) = 0.0555]	
Completeness to theta = 29.34°	99.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1857 / 0 / 129	
Goodness-of-fit on F^2	1.152	
Final R indices [I>2sigma(I)]	R1 = 0.0612, wR2 = 0.1261	
R indices (all data)	R1 = 0.0803, wR2 = 0.1335	
Extinction coefficient	0.0004(8)	
Largest diff. peak and hole	0.332 and -0.179 $e.Å^{-3}$	

Table 89: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{24}H_{28}N_8O_{12}$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
N(1)	8623(1)	6222(1)	-165(1)	26(1)
N(2)	7851(1)	7373(1)	827(1)	27(1)
O(1)	10212(1)	6314(1)	-363(1)	34(1)
O(2)	8687(1)	8410(1)	1744(1)	34(1)
O(3)	7030(1)	6359(1)	-139(1)	40(1)
C(1)	9508(1)	6560(1)	71(1)	25(1)
C(2)	9564(1)	7183(1)	967(1)	24(1)
C(3)	8677(1)	7730(1)	1204(1)	25(1)
C(4)	7790(1)	6628(1)	158(1)	27(1)
C(6)	6955(2)	7818(2)	1130(2)	38(1)
C(7)	8549(2)	5458(2)	-897(2)	34(1)

Table 90: Bond lengths [Å] and angles [°] for $C_{24}H_{28}N_8O_{12}$

N(1)-C(1)	1.375(2)	O(1)-C(1)	1.208(2)
N(1)-C(4)	1.380(2)	O(2)-C(3)	1.210(2)
N(1)-C(7)	1.475(2)	O(3)-C(4)	1.208(2)
N(2)-C(3)	1.371(2)	C(1)-C(2)	1.507(3)
N(2)-C(4)	1.394(3)	C(2)-C(3)	1.506(2)
N(2)-C(6)	1.470(3)	C(2)-C(2)#1	1.522(3)
C(1)-N(1)-C(4)	123.74(15)	C(3)-C(2)-C(1)	115.44(15)
C(1)-N(1)-C(7)	118.43(16)	C(3)-C(2)-C(2)#1	111.71(18)
C(4)-N(1)-C(7)	117.41(16)	C(1)-C(2)-C(2)#1	112.69(12)
C(3)-N(2)-C(4)	125.10(16)	O(2)-C(3)-N(2)	121.96(17)
C(3)-N(2)-C(6)	117.99(17)	O(2)-C(3)-C(2)	121.90(17)
C(4)-N(2)-C(6)	116.90(17)	N(2)-C(3)-C(2)	115.99(16)
O(1)-C(1)-N(1)	122.18(17)	O(3)-C(4)-N(1)	121.26(18)
O(1)-C(1)-C(2)	121.48(17)	O(3)-C(4)-N(2)	120.75(18)
N(1)-C(1)-C(2)	116.08(15)	N(1)-C(4)-N(2)	117.97(16)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y+3/2,z+0

7.3.31 Crystal data for $C_{12}H_{14}N_4O_6S$ **(66)**

Table 91: Crystal data and structure refinement for $C_{12}H_{14}N_4O_6S$ **(66)**

Empirical formula	$C_{12}H_{14}N_4O_6S$	
Formula weight	342.33	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.3352(17) Å	α= 104.13(3)°
	b = 8.6688(17) Å	β= 91.67(3)°
	c = 11.701(2) Å	γ = 117.78(3)°
Volume	715.2(2) $Å^3$	
Z	2	
Density (calculated)	1.590 Mg/m^3	
Absorption coefficient	0.266 mm^{-1}	
F(000)	356	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.08 to 31.07°.	
Index ranges	-1<=h<=12, -12<=k<=11, -16<=l<=16	
Reflections collected	5403	
Independent reflections	4564 [R(int) = 0.0287]	
Completeness to theta = 31.07°	99.4 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4564 / 0 / 265	
Goodness-of-fit on F^2	1.039	
Final R indices [I>2sigma(I)]	R1 = 0.0548, wR2 = 0.1369	
R indices (all data)	R1 = 0.0921, wR2 = 0.1543	
Extinction coefficient	0.005(4)	
Largest diff. peak and hole	0.622 and -0.448 $e.Å^{-3}$	

Table 92: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{12}H_{14}N_4O_6S$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
S(1)	3898(1)	10380(1)	7051(1)	30(1)
N(1)	2188(2)	8039(2)	9656(1)	27(1)
N(2)	4607(2)	7516(2)	9021(1)	27(1)
N(3)	-637(2)	7158(2)	4578(2)	30(1)
N(4)	1658(2)	6447(2)	3928(1)	28(1)
O(1)	1011(2)	9313(2)	8643(1)	37(1)
O(2)	5816(2)	8244(2)	7401(1)	37(1)
O(3)	3203(2)	6488(2)	10514(1)	38(1)
O(4)	-198(2)	9092(2)	6401(2)	37(1)
O(5)	4400(2)	7675(2)	5141(1)	35(1)
O(6)	-1094(2)	5259(2)	2727(2)	46(1)
C(1)	2212(3)	8871(3)	8777(2)	28(1)
C(2)	3627(3)	9138(2)	8073(2)	26(1)
C(3)	4684(3)	8326(2)	8138(2)	26(1)
C(4)	3322(3)	7298(3)	9777(2)	27(1)
C(5)	5752(4)	6661(4)	9106(2)	41(1)
C(6)	783(4)	7754(4)	10430(2)	40(1)
C(7)	535(3)	8351(3)	5620(2)	27(1)
C(8)	2318(3)	8690(3)	5793(2)	26(1)
C(9)	2900(3)	7626(3)	4971(2)	27(1)
C(10)	-73(3)	6242(3)	3678(2)	31(1)
C(11)	2186(4)	5337(3)	3024(2)	37(1)
C(12)	-2542(3)	6805(4)	4369(3)	44(1)

Table 93: Bond lengths [Å] and angles [°] for $C_{12}H_{14}N_4O_6S$

S(1)-C(2)	1.7493(19)	N(4)-C(9)	1.391(3)
S(1)-C(8)	1.755(2)	N(4)-C(11)	1.471(3)
N(1)-C(1)	1.389(2)	O(1)-C(1)	1.246(3)
N(1)-C(4)	1.389(3)	O(2)-C(3)	1.311(2)
N(1)-C(6)	1.468(3)	O(3)-C(4)	1.218(2)
N(2)-C(3)	1.371(2)	O(4)-C(7)	1.316(2)
N(2)-C(4)	1.383(3)	O(5)-C(9)	1.240(2)
N(2)-C(5)	1.470(3)	O(6)-C(10)	1.222(3)
N(3)-C(7)	1.372(3)	C(1)-C(2)	1.420(3)
N(3)-C(10)	1.390(3)	C(2)-C(3)	1.370(3)
N(3)-C(12)	1.470(3)	C(7)-C(8)	1.372(3)
N(4)-C(10)	1.381(3)	C(8)-C(9)	1.422(3)

C(2)-S(1)-C(8)	101.71(9)	O(2)-C(3)-C(2)	124.91(17)
C(1)-N(1)-C(4)	123.30(16)	O(2)-C(3)-N(2)	114.78(17)
C(1)-N(1)-C(6)	119.24(18)	C(2)-C(3)-N(2)	120.30(17)
C(4)-N(1)-C(6)	117.18(17)	O(3)-C(4)-N(2)	120.85(19)
C(3)-N(2)-C(4)	121.55(17)	O(3)-C(4)-N(1)	121.96(19)
C(3)-N(2)-C(5)	120.69(17)	N(2)-C(4)-N(1)	117.19(16)
C(4)-N(2)-C(5)	117.41(16)	O(4)-C(7)-C(8)	124.77(19)
C(7)-N(3)-C(10)	121.48(17)	O(4)-C(7)-N(3)	114.77(18)
C(7)-N(3)-C(12)	121.20(19)	C(8)-C(7)-N(3)	120.47(18)
C(10)-N(3)-C(12)	117.32(19)	C(7)-C(8)-C(9)	120.20(18)
C(10)-N(4)-C(9)	123.95(17)	C(7)-C(8)-S(1)	120.98(15)
C(10)-N(4)-C(11)	116.92(18)	C(9)-C(8)-S(1)	118.78(15)
C(9)-N(4)-C(11)	119.13(18)	O(5)-C(9)-N(4)	119.60(18)
O(1)-C(1)-N(1)	119.22(18)	O(5)-C(9)-C(8)	124.20(18)
O(1)-C(1)-C(2)	124.32(18)	N(4)-C(9)-C(8)	116.20(17)
N(1)-C(1)-C(2)	116.46(18)	O(6)-C(10)-N(4)	122.2(2)
C(3)-C(2)-C(1)	120.14(17)	O(6)-C(10)-N(3)	121.0(2)
C(3)-C(2)-S(1)	120.41(15)	N(4)-C(10)-N(3)	116.83(1
C(1)-C(2)-S(1)	119.30(15)		

7.3.32 Crystal data for $C_{23}H_{33}N_6O_6S$ **(67)**

Table 94: Crystal data and structure refinement for $C_{23}H_{33}N_6O_6S$ **(67)**

Empirical formula	$C_{23}H_{33}N_6O_6S$	
Formula weight	521.61	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 12.862(3) Å	α= 90°.
	b = 13.105(3) Å	β= 99.10(3)°.
	c = 19.604(4) Å	γ = 90°.
Volume	3262.7(11) $Å^3$	
Z	4	
Density (calculated)	1.062 Mg/m^3	
Absorption coefficient	0.138 mm^{-1}	
F(000)	1108	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	2.36 to 20.00°.	
Index ranges	-1<=h<=12, 0<=k<=12, -18<=l<=18	
Reflections collected	3854	
Independent reflections	3050 [R(int) = 0.1434]	
Completeness to theta = 20.00°	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3050 / 2 / 352	
Goodness-of-fit on F^2	1.096	
Final R indices [I>2sigma(I)]	R1 = 0.0843, wR2 = 0.2406	
R indices (all data)	R1 = 0.0959, wR2 = 0.2530	
Extinction coefficient	0.010(3)	
Largest diff. peak and hole	0.600 and -0.331 $e.Å^{-3}$	

Table 95: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{23}H_{33}N_6O_6S$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
S(1)	7473(1)	6274(1)	753(1)	52(1)
N(1)	5866(3)	3730(3)	94(3)	55(1)
N(2)	5886(3)	4777(3)	-865(2)	48(1)
N(3)	10294(4)	6373(3)	126(2)	51(1)
N(4)	10565(3)	5624(3)	1224(2)	50(1)
N(5)	7585(4)	4027(4)	3053(3)	62(1)
N(6)	7446(4)	5650(4)	3047(3)	77(2)
O(1)	6635(3)	4212(3)	1165(2)	65(1)
O(2)	5114(3)	3212(3)	-980(2)	69(1)
O(3)	6844(3)	6225(3)	-803(2)	59(1)
O(4)	8669(3)	6761(3)	-427(2)	60(1)
O(5)	11948(3)	6095(3)	697(2)	62(1)
O(6)	9225(3)	5303(3)	1812(2)	63(1)
C(1)	6529(4)	5465(4)	-462(3)	51(2)
C(2)	6774(4)	5322(4)	230(3)	47(1)
C(3)	6454(4)	4414(4)	561(4)	54(2)
C(4)	5596(5)	3872(5)	-608(3)	56(2)
C(5)	5513(5)	2771(5)	368(4)	79(2)
C(6)	5624(5)	4934(5)	-1604(3)	64(2)
C(7)	9211(4)	6411(4)	116(3)	48(1)
C(8)	8812(4)	6068(4)	713(3)	51(1)
C(9)	9489(4)	5647(4)	1265(3)	49(1)
C(10)	10996(5)	6031(4)	671(3)	49(1)
C(11)	11292(5)	5178(5)	1777(3)	68(2)
C(12)	10721(5)	6731(5)	-491(3)	65(2)
C(14)	7564(5)	4848(6)	2678(4)	79(2)
C(15)	7395(6)	5337(5)	3722(4)	79(2)
C(16)	7491(6)	4315(4)	3717(3)	74(2)
C(17A)	7418(9)	6854(15)	3033(13)	95(7)
C(18A)	6333(8)	7140(6)	2747(5)	122(3)
C(19A)	8390(8)	7189(5)	2808(4)	115(3)
C(17B)	7331(14)	6607(11)	2632(10)	73(5)

C(20)	7281(10)	6016(6)	4292(5)	137(4)
C(21)	7435(10)	3561(6)	4270(4)	132(4)
C(22)	7665(6)	2948(5)	2808(3)	77(2)
C(23)	6587(7)	2585(5)	2537(4)	96(2)
C(24)	8398(7)	2883(6)	2269(4)	108(3)

Table 96: Bond lengths [Å] and angles [°] for $C_{23}H_{33}N_6O_6S$

S(1)-C(2)	1.767(5)	N(6)-C(17B)	1.489(16)
S(1)-C(8)	1.758(6)	N(6)-C(17A)	1.58(2)
N(1)-C(4)	1.379(8)	O(1)-C(3)	1.199(7)
N(1)-C(3)	1.412(7)	O(2)-C(4)	1.232(7)
N(1)-C(5)	1.466(7)	O(3)-C(1)	1.300(6)
N(2)-C(4)	1.364(7)	O(4)-C(7)	1.262(6)
N(2)-C(1)	1.384(7)	O(5)-C(10)	1.220(6)
N(2)-C(6)	1.448(7)	O(6)-C(9)	1.259(6)
N(3)-C(10)	1.362(7)	C(1)-C(2)	1.355(7)
N(3)-C(7)	1.391(7)	C(2)-C(3)	1.447(8)
N(3)-C(12)	1.482(7)	C(7)-C(8)	1.422(8)
N(4)-C(9)	1.399(7)	C(8)-C(9)	1.391(8)
N(4)-C(10)	1.398(7)	C(15)-C(16)	1.346(9)
N(4)-C(11)	1.439(7)	C(15)-C(20)	1.454(11)
N(5)-C(14)	1.300(8)	C(16)-C(21)	1.478(9)
N(5)-C(16)	1.378(7)	C(17A)-C(19A)	1.459(13)
N(5)-C(22)	1.502(8)	C(17A)-C(18A)	1.467(14)
N(6)-C(14)	1.298(9)	C(22)-C(23)	1.482(10)
N(6)-C(15)	1.397(8)	C(22)-C(24)	1.525(9)

C(2)-S(1)-C(8)	106.1(2)	C(1)-N(2)-C(6)	120.2(5)
C(4)-N(1)-C(3)	126.0(5)	C(10)-N(3)-C(7)	124.1(5)
C(4)-N(1)-C(5)	115.9(5)	C(10)-N(3)-C(12)	117.4(5)
C(3)-N(1)-C(5)	118.2(5)	C(7)-N(3)-C(12)	118.5(5)
C(4)-N(2)-C(1)	122.0(5)	C(9)-N(4)-C(10)	123.6(5)
C(4)-N(2)-C(6)	117.3(5)	C(9)-N(4)-C(11)	119.7(4)

C(10)-N(4)-C(11)	116.7(5)	N(3)-C(7)-C(8)	117.9(5)
C(14)-N(5)-C(16)	108.0(5)	C(7)-C(8)-C(9)	120.2(5)
C(14)-N(5)-C(22)	126.5(6)	C(7)-C(8)-S(1)	118.2(4)
C(16)-N(5)-C(22)	125.5(5)	C(9)-C(8)-S(1)	121.5(4)
C(14)-N(6)-C(15)	108.5(5)	O(6)-C(9)-N(4)	116.4(5)
C(14)-N(6)-C(17B)	112.9(9)	O(6)-C(9)-C(8)	125.8(5)
C(15)-N(6)-C(17B)	138.5(9)	N(4)-C(9)-C(8)	117.8(5)
C(14)-N(6)-C(17A)	143.6(10)	O(5)-C(10)-N(3)	123.1(5)
C(15)-N(6)-C(17A)	107.7(10)	O(5)-C(10)-N(4)	120.8(5)
C(17B)-N(6)-C(17A)	31.7(8)	N(3)-C(10)-N(4)	116.0(5)
O(3)-C(1)-C(2)	125.4(5)	N(6)-C(14)-N(5)	110.5(6)
O(3)-C(1)-N(2)	114.3(5)	C(16)-C(15)-N(6)	105.4(5)
C(2)-C(1)-N(2)	120.3(5)	C(16)-C(15)-C(20)	129.5(7)
C(1)-C(2)-C(3)	121.7(5)	N(6)-C(15)-C(20)	125.1(6)
C(1)-C(2)-S(1)	119.9(4)	C(15)-C(16)-N(5)	107.6(5)
C(3)-C(2)-S(1)	118.3(4)	C(15)-C(16)-C(21)	130.1(6)
O(1)-C(3)-N(1)	120.1(5)	N(5)-C(16)-C(21)	122.2(5)
O(1)-C(3)-C(2)	126.9(5)	C(19A)-C(17A)-C(18A)	128.0(18)
N(1)-C(3)-C(2)	113.1(5)	C(19A)-C(17A)-N(6)	106.7(10)
O(2)-C(4)-N(2)	122.3(5)	C(18A)-C(17A)-N(6)	106.2(10)
O(2)-C(4)-N(1)	121.3(6)	C(23)-C(22)-N(5)	108.0(6)
N(2)-C(4)-N(1)	116.4(5)	C(23)-C(22)-C(24)	112.4(6)
O(4)-C(7)-N(3)	116.3(5)	N(5)-C(22)-C(24)	110.8(5)
O(4)-C(7)-C(8)	125.8(5)		

7.3.33 Crystal data for $C_{34}H_{54}N_8O_6S$ **(68)**

Tab.97: Crystal data and structure refinement for $C_{34}H_{54}N_8O_6S$ **(68)**

Empirical formula	$C_{34}H_{54}N_8O_6S$	
Formula weight	702.91	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 10.205(2) Å	α= 90°.
	b = 22.155(4) Å	β= 104.89(3)°.
	c = 17.238(3) Å	γ = 90°.
Volume	3766.5(13) $Å^3$	
Z	4	
Density (calculated)	1.240 Mg/m^3	
Absorption coefficient	0.139 mm^{-1}	
F(000)	1512	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	2.11 to 26.08°.	
Index ranges	-12<=h<=12, -27<=k<=27, -21<=l<=21	
Reflections collected	36522	
Independent reflections	7416 [R(int) = 0.0860]	
Completeness to theta = 26.08°	99.2 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7416 / 0 / 659	
Goodness-of-fit on F^2	0.870	
Final R indices [I>2sigma(I)]	R1 = 0.0497, wR2 = 0.1261	
R indices (all data)	R1 = 0.0731, wR2 = 0.1358	
Extinction coefficient	0.0000(8)	
Largest diff. peak and hole	0.408 and -0.259 $e.Å^{-3}$	

Table 98: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{34}H_{54}N_8O_6S$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
S(1)	2697(1)	702(1)	5995(1)	47(1)
O(1)	2550(2)	1705(1)	4805(1)	47(1)
O(2)	6832(2)	2416(1)	5721(1)	84(1)
O(3)	5498(2)	939(1)	7171(1)	54(1)
O(4)	1358(2)	1673(1)	6822(1)	54(1)
O(5)	1300(2)	589(1)	9049(1)	66(1)
O(6)	3131(2)	-302(1)	7216(1)	54(1)
N(1)	9176(2)	1731(1)	4409(1)	46(1)
N(2)	10002(2)	2589(1)	4880(1)	53(1)
N(3)	2732(2)	370(1)	1596(1)	50(1)
N(4)	3132(2)	729(1)	2796(1)	48(1)
N(5)	4676(2)	2077(1)	5297(1)	48(1)
N(6)	6128(2)	1692(1)	6459(1)	52(1)
N(7)	1371(2)	1131(1)	7946(1)	43(1)
N(8)	2115(2)	141(1)	8091(1)	44(1)
C(1)	9849(2)	2018(1)	5062(1)	48(1)
C(2)	8908(2)	2134(1)	3774(1)	55(1)
C(3)	9431(2)	2671(1)	4069(2)	58(1)
C(4)	8833(2)	1080(1)	4351(2)	53(1)
C(5)	9871(3)	736(1)	4038(2)	61(1)
C(6)	8715(3)	848(1)	5153(2)	66(1)
C(7)	8130(4)	1970(2)	2950(2)	79(1)
C(8)	9483(4)	3255(2)	3650(3)	85(1)
C(9)	10621(2)	3065(1)	5466(2)	66(1)
C(10)	12085(3)	2976(2)	5767(2)	79(1)
C(11)	9921(4)	3103(2)	6156(3)	99(1)
C(12)	3424(3)	290(1)	2349(1)	59(1)
C(13)	1957(2)	886(1)	1552(1)	48(1)
C(14)	2221(2)	1112(1)	2308(1)	47(1)
C(15)	2802(3)	-32(1)	923(1)	55(1)
C(16)	2104(3)	-621(2)	990(2)	72(1)
C(17)	4276(3)	-135(2)	935(2)	80(1)

C(18)	1042(4)	1103(2)	793(2)	73(1)
C(19)	1732(4)	1677(1)	2610(2)	76(1)
C(20)	3684(2)	772(1)	3681(1)	58(1)
C(21)	3506(5)	170(2)	4060(2)	86(1)
C(22)	5109(3)	969(2)	3887(2)	83(1)
C(23)	3905(2)	1273(1)	6003(1)	39(1)
C(24)	3611(2)	1674(1)	5347(1)	39(1)
C(25)	5934(2)	2079(1)	5819(2)	56(1)
C(26)	5157(2)	1271(1)	6574(1)	43(1)
C(27)	4483(4)	2472(1)	4601(2)	67(1)
C(28)	7467(3)	1679(2)	7025(2)	75(1)
C(29)	2315(2)	697(1)	6934(1)	36(1)
C(30)	1664(2)	1188(1)	7182(1)	38(1)
C(31)	1580(2)	620(1)	8400(1)	44(1)
C(32)	2578(2)	160(1)	7379(1)	40(1)
C(33)	837(3)	1649(1)	8286(2)	58(1)
C(34)	2201(4)	-431(1)	8521(2)	66(1)

Table 99: Bond lengths [Å] and angles [°] for $C_{34}H_{54}N_8O_6S$

S(1)-C(29)	1.7593(19)	N(3)-C(15)	1.479(3)
S(1)-C(23)	1.7631(19)	N(4)-C(12)	1.322(3)
O(1)-C(24)	1.237(2)	N(4)-C(14)	1.374(3)
O(2)-C(25)	1.227(3)	N(4)-C(20)	1.487(3)
O(3)-C(26)	1.240(2)	N(5)-C(25)	1.365(3)
O(4)-C(30)	1.240(2)	N(5)-C(24)	1.426(3)
O(5)-C(31)	1.226(2)	N(5)-C(27)	1.457(3)
O(6)-C(32)	1.236(2)	N(6)-C(25)	1.370(3)
N(1)-C(1)	1.321(3)	N(6)-C(26)	1.411(3)
N(1)-C(2)	1.383(3)	N(6)-C(28)	1.461(3)
N(1)-C(4)	1.483(3)	N(7)-C(31)	1.363(3)
N(2)-C(1)	1.324(3)	N(7)-C(30)	1.429(2)
N(2)-C(3)	1.381(3)	N(7)-C(33)	1.456(3)
N(2)-C(9)	1.485(3)	N(8)-C(31)	1.362(3)
N(3)-C(12)	1.321(3)	N(8)-C(32)	1.424(3)
N(3)-C(13)	1.381(3)	N(8)-C(34)	1.459(3)

C(2)-C(3)	1.349(3)	C(14)-C(19)	1.491(3)
C(2)-C(7)	1.483(4)	C(15)-C(16)	1.506(4)
C(3)-C(8)	1.491(4)	C(15)-C(17)	1.517(4)
C(4)-C(6)	1.509(4)	C(20)-C(22)	1.472(4)
C(4)-C(5)	1.511(4)	C(20)-C(21)	1.516(4)
C(9)-C(10)	1.463(4)	C(23)-C(26)	1.398(3)
C(9)-C(11)	1.540(5)	C(23)-C(24)	1.409(3)
C(13)-C(14)	1.356(3)	C(29)-C(30)	1.398(3)
C(13)-C(18)	1.479(3)	C(29)-C(32)	1.405(3)

C(29)-S(1)-C(23)	108.45(9)	N(1)-C(1)-N(2)	108.91(19)
C(1)-N(1)-C(2)	108.59(17)	C(3)-C(2)-N(1)	106.95(19)
C(1)-N(1)-C(4)	125.97(18)	C(3)-C(2)-C(7)	129.7(2)
C(2)-N(1)-C(4)	125.34(18)	N(1)-C(2)-C(7)	123.3(2)
C(1)-N(2)-C(3)	108.69(18)	C(2)-C(3)-N(2)	106.83(19)
C(1)-N(2)-C(9)	125.0(2)	C(2)-C(3)-C(8)	130.1(3)
C(3)-N(2)-C(9)	126.26(19)	N(2)-C(3)-C(8)	123.0(3)
C(12)-N(3)-C(13)	108.63(18)	N(1)-C(4)-C(6)	110.0(2)
C(12)-N(3)-C(15)	124.63(18)	N(1)-C(4)-C(5)	109.9(2)
C(13)-N(3)-C(15)	126.74(17)	C(6)-C(4)-C(5)	112.6(2)
C(12)-N(4)-C(14)	108.35(18)	C(10)-C(9)-N(2)	111.2(2)
C(12)-N(4)-C(20)	124.20(18)	C(10)-C(9)-C(11)	111.6(3)
C(14)-N(4)-C(20)	127.42(18)	N(2)-C(9)-C(11)	111.3(2)
C(25)-N(5)-C(24)	124.36(17)	N(3)-C(12)-N(4)	109.3(2)
C(25)-N(5)-C(27)	116.5(2)	C(14)-C(13)-N(3)	106.40(17)
C(24)-N(5)-C(27)	118.8(2)	C(14)-C(13)-C(18)	130.9(2)
C(25)-N(6)-C(26)	124.42(18)	N(3)-C(13)-C(18)	122.7(2)
C(25)-N(6)-C(28)	117.8(2)	C(13)-C(14)-N(4)	107.33(18)
C(26)-N(6)-C(28)	117.6(2)	C(13)-C(14)-C(19)	130.1(2)
C(31)-N(7)-C(30)	124.33(16)	N(4)-C(14)-C(19)	122.5(2)
C(31)-N(7)-C(33)	116.31(19)	N(3)-C(15)-C(16)	110.1(2)
C(30)-N(7)-C(33)	119.35(19)	N(3)-C(15)-C(17)	109.2(2)
C(31)-N(8)-C(32)	124.72(16)	C(16)-C(15)-C(17)	110.8(2)
C(31)-N(8)-C(34)	117.11(19)	C(22)-C(20)-N(4)	110.9(2)
C(32)-N(8)-C(34)	118.17(19)	C(22)-C(20)-C(21)	112.2(3)

N(4)-C(20)-C(21)	109.3(2)	C(30)-C(29)-C(32)	122.10(17)
C(26)-C(23)-C(24)	122.28(18)	C(30)-C(29)-S(1)	120.77(14)
C(26)-C(23)-S(1)	120.99(15)	C(32)-C(29)-S(1)	116.87(14)
C(24)-C(23)-S(1)	116.19(14)	O(4)-C(30)-C(29)	126.90(18)
O(1)-C(24)-C(23)	127.29(18)	O(4)-C(30)-N(7)	116.84(17)
O(1)-C(24)-N(5)	116.91(17)	C(29)-C(30)-N(7)	116.22(16)
C(23)-C(24)-N(5)	115.78(17)	O(5)-C(31)-N(8)	121.66(19)
O(2)-C(25)-N(5)	121.4(2)	O(5)-C(31)-N(7)	122.09(19)
O(2)-C(25)-N(6)	122.2(2)	N(8)-C(31)-N(7)	116.24(17)
N(5)-C(25)-N(6)	116.46(19)	O(6)-C(32)-C(29)	127.73(18)
O(3)-C(26)-C(23)	126.62(19)	O(6)-C(32)-N(8)	116.38(17)
O(3)-C(26)-N(6)	116.83(18)	C(29)-C(32)-N(8)	115.86(16)
C(23)-C(26)-N(6)	116.55(18)		

7.3.34 Crystal data for $C_{24}H_{21}N_2O_3PS$ **(69)**

Table 100: Crystal data and structure refinement for $C_{24}H_{21}N_2O_3PS$ **(69)**

Empirical formula	$C_{24}H_{21}N_2O_3PS$	
Formula weight	448.46	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 9.879(2) Å	α= 90°.
	b = 14.163(3) Å	β= 90°.
	c = 15.090(3) Å	γ = 90°.
Volume	2111.2(7) $Å^3$	
Z	4	
Density (calculated)	1.411 Mg/m^3	
Absorption coefficient	0.259 mm^{-1}	
F(000)	936	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	3.06 to 28.06°.	
Index ranges	-1<=h<=13, -1<=k<=18, -19<=l<=19	
Reflections collected	6588	
Independent reflections	5074 [R(int) = 0.0450]	
Completeness to theta = 28.06°	99.4 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5074 / 0 / 365	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0406, wR2 = 0.0892	
R indices (all data)	R1 = 0.0696, wR2 = 0.0984	
Absolute structure parameter	0.13(8)	
Extinction coefficient	0.0008(5)	
Largest diff. peak and hole	0.392 and -0.236 $e.Å^{-3}$	

Table 101: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{24}H_{21}N_2O_3PS$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
S(1)	1871(1)	2299(1)	844(1)	31(1)
P(1)	1862(1)	823(1)	745(1)	24(1)
N(1)	-1334(2)	2560(2)	2433(2)	34(1)
N(2)	594(2)	2632(2)	3340(1)	33(1)
O(1)	-1168(2)	2367(2)	941(1)	42(1)
O(2)	-1512(2)	2729(2)	3926(1)	48(1)
O(3)	2716(2)	2539(2)	2756(1)	41(1)
C(1)	854(3)	2462(2)	1773(2)	29(1)
C(2)	1491(3)	2540(2)	2613(2)	30(1)
C(3)	-794(3)	2643(2)	3270(2)	34(1)
C(4)	-562(3)	2462(2)	1652(2)	32(1)
C(5)	1161(4)	2681(3)	4237(2)	47(1)
C(6)	-2808(3)	2541(3)	2360(3)	53(1)
C(7)	944(3)	356(2)	1664(2)	25(1)
C(8)	1555(3)	271(2)	2490(2)	36(1)
C(9)	786(3)	11(2)	3216(2)	42(1)
C(10)	-581(3)	-154(2)	3125(2)	40(1)
C(11)	-1185(3)	-86(2)	2300(2)	37(1)
C(12)	-433(3)	175(2)	1570(2)	32(1)
C(13)	1021(2)	444(2)	-257(2)	26(1)
C(14)	195(3)	1058(2)	-724(2)	36(1)
C(15)	-471(3)	748(2)	-1479(2)	44(1)
C(16)	-309(3)	-170(2)	-1761(2)	39(1)
C(17)	486(3)	-794(2)	-1288(2)	36(1)
C(18)	1159(3)	-486(2)	-531(2)	32(1)
C(19)	3572(2)	378(2)	759(2)	26(1)
C(20)	3813(3)	-566(2)	969(2)	32(1)
C(21)	5118(3)	-911(2)	941(2)	38(1)
C(22)	6178(3)	-325(2)	707(2)	39(1)
C(23)	5942(3)	613(2)	507(2)	36(1)
C(24)	4637(3)	967(2)	528(2)	30(1)

Table 102: Bond lengths [Å] and angles [°] for $C_{24}H_{21}N_2O_3PS$

S(1)-C(1)	1.740(2)	C(7)-C(12)	1.392(4)
S(1)-P(1)	2.0954(9)	C(8)-C(9)	1.383(4)
P(1)-C(7)	1.783(3)	C(9)-C(10)	1.377(4)
P(1)-C(19)	1.804(2)	C(10)-C(11)	1.384(4)
P(1)-C(13)	1.806(2)	C(11)-C(12)	1.380(4)
N(1)-C(3)	1.377(3)	C(13)-C(14)	1.386(3)
N(1)-C(4)	1.410(3)	C(13)-C(18)	1.388(4)
N(1)-C(6)	1.460(4)	C(14)-C(15)	1.386(4)
N(2)-C(3)	1.375(4)	C(15)-C(16)	1.376(4)
N(2)-C(2)	1.416(3)	C(16)-C(17)	1.381(4)
N(2)-C(5)	1.467(4)	C(17)-C(18)	1.392(4)
O(1)-C(4)	1.236(3)	C(19)-C(24)	1.387(3)
O(2)-C(3)	1.225(3)	C(19)-C(20)	1.394(3)
O(3)-C(2)	1.230(3)	C(20)-C(21)	1.380(4)
C(1)-C(4)	1.411(4)	C(21)-C(22)	1.381(4)
C(1)-C(2)	1.420(4)	C(22)-C(23)	1.383(4)
C(7)-C(8)	1.390(4)	C(23)-C(24)	1.384(4)

C(1)-S(1)-P(1)	100.82(9)	C(2)-C(1)-S(1)	118.28(19)
C(7)-P(1)-C(19)	109.73(12)	O(3)-C(2)-N(2)	118.8(2)
C(7)-P(1)-C(13)	107.85(11)	O(3)-C(2)-C(1)	126.3(2)
C(19)-P(1)-C(13)	109.67(12)	N(2)-C(2)-C(1)	114.9(2)
C(7)-P(1)-S(1)	108.48(8)	O(2)-C(3)-N(2)	121.1(3)
C(19)-P(1)-S(1)	110.08(9)	O(2)-C(3)-N(1)	121.7(3)
C(13)-P(1)-S(1)	110.98(9)	N(2)-C(3)-N(1)	117.2(2)
C(3)-N(1)-C(4)	124.4(2)	O(1)-C(4)-N(1)	118.3(2)
C(3)-N(1)-C(6)	117.2(3)	O(1)-C(4)-C(1)	126.3(2)
C(4)-N(1)-C(6)	118.4(3)	N(1)-C(4)-C(1)	115.3(2)
C(3)-N(2)-C(2)	124.4(2)	C(8)-C(7)-C(12)	120.0(2)
C(3)-N(2)-C(5)	116.9(2)	C(8)-C(7)-P(1)	120.6(2)
C(2)-N(2)-C(5)	118.7(2)	C(12)-C(7)-P(1)	119.04(19)
C(4)-C(1)-C(2)	123.7(2)	C(9)-C(8)-C(7)	119.7(3)
C(4)-C(1)-S(1)	117.9(2)	C(10)-C(9)-C(8)	120.3(3)

C(9)-C(10)-C(11)	120.0(3)
C(12)-C(11)-C(10)	120.3(3)
C(11)-C(12)-C(7)	119.6(3)
C(14)-C(13)-C(18)	120.1(2)
C(14)-C(13)-P(1)	120.7(2)
C(18)-C(13)-P(1)	119.13(19)
C(13)-C(14)-C(15)	119.9(3)
C(16)-C(15)-C(14)	119.8(3)
C(15)-C(16)-C(17)	120.7(3)
C(16)-C(17)-C(18)	119.6(3)
C(13)-C(18)-C(17)	119.7(3)
C(24)-C(19)-C(20)	120.3(2)
C(24)-C(19)-P(1)	119.85(19)
C(20)-C(19)-P(1)	119.80(18)
C(21)-C(20)-C(19)	119.5(2)
C(20)-C(21)-C(22)	120.2(3)
C(21)-C(22)-C(23)	120.4(2)
C(22)-C(23)-C(24)	120.0(3)
C(23)-C(24)-C(19)	119.7(2)

Meine akademische Ausbildung verdanke ich:

University of Jordan:

G. A. Derwish, Q. Ibraheem, A. H. Qasem, M. Fayad, M. Al-Hourani, M. Tutunji, M. Zughul, M. El-Abadelah, K. Abu Dari, M.H. Abu Zarqa, S. Sabri, M. Alawi, F. AL-Kalilli

Eberhard-Karls-Universität Tübingen:

N. Kuhn, H. A. Mayer, G. Häfelinger, J. Strähle, W. Voelter

Lebenslauf

Persönliche Daten:

Name	Kamal Sweidan
Geburtstag	26. April 1973
Geburtsort	Kuwait
Staatsangehörigkeit	Jordanisch
Familienstand	Verheiratet, zwei Kinder

Schulbildung:

1979 – 1983	Grundschule in Kuwait
1984– 1990	Gymnasium in Kuwait
Juli 1992	Abitur in Amman, Jordanien

Hochschulausbildung:

09/1992 – 06/ 1996	B. Sc. Studium an der "University of Jordan" in Amman, Jordanien
06/1996	B. Sc. Degree
09/1996 – 01/1999	M. Sc. Studium an der "University of Jordan" in Amman, Jordanien
01/1999	M.Sc. Examen mit dem Thema " Use of Duckweeds for Removal of Heavy Metals and Organic Compounds from Wastewater " bei Prof. Dr. Manar Fayyad am Institut für Chemie der "University of Jordan" Amman, Jordanien

04/2003 – 4/2006	Dissertation unter der Leitung von Herrn Prof. Dr. N. Kuhn am Institut für Anorganische Chemie der Universität Tübingen mit dem Thema: "Derivate des Imidazols und der Barbitursäure - Heterozyklische Carben - Fragmente mit π-Donor und Akzeptor Funktion".

Beschäftigung

09/1996 – 01/1999	"Teaching Assistant" an der "University of Jordan", Amman, Jordanien
08/1998 – 02/2001	Forschungs- und Entwicklungs- Supervisor an der Pharmazeutischen Firma "Hikma Pharmaceuticals", Amman, Jordanien.
03/2001 – 01/2003	Forschungs- und Entwicklungs- Supervisor an der Pharmazeutischen Firma "Tabuk Pharmaceuticals ", Tabuk, Saudi Arabia.
07/2004 - 08/2004	Assistent im Chemiepraktikum für den Studiengang Biologie