# Synthesis and Crystal Structure of Schiff Bases Based on AMTTO (AMTTO = 4-Amino-6-methyl-3-thio-3,4-dihydro-1,2,4-triazin-5(2H)-one) 

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Reaction of 4-amino-6-methyl-3-thio-3,4-dihydro-1,2,4-triazin-5(2H)-one (1) with 2-methoxybenzaldehyde, 4-methoxybenzaldehyde, 2-pyridinecarbaldehyde, and 2,4-dichlorobenzaldehyde under classical heating and microwave irradiation in a solventfree system led to the corresponding imines (Schiff bases) 2-5. All synthesized compounds have been characterized by IR and NMR spectroscopy, mass spectrometry and by X-ray diffraction studies.

Key words: 1,2,4-Triazine, 1,2,4-Triazole, Schiff Base, Microwave Irradiation

## Introduction

During the last few decades, there has been considerable interest in the chemistry of Schiff base compounds [1,2]. Schiff bases, containing different donor atoms, also find use in analytical applications and metal coordination [3-5]. Since many compounds containing sulfur and nitrogen atoms are antihypertensive [6], analgesic [7], antiinflammatory [8], sedative [9], or fungicidal [10], synthesis of the corresponding heterocyclic compounds could be of interest from the viewpoint of chemical reactivity and biological activity. 1,2,4-Triazines and 1,2,4-triazoles are well known heterocyclic thiones derived from thiocarbohydrazide. Some of their derivatives exhibit biological activity and have been used for various purposes such as herbicides, neutral antibiotics, antibacterials etc. The heterocyclic thiones exist in thione and thiol tautomeric forms. Therefore, there has been considerable interest in the coordination properties of both the neutral thione and the deprotonated thiol ligand [11]. In our earlier investigations we have shown that 4-amino-6-methyl-1,2,4-triazine-3-thione-5(2H)-one (1) and 4-amino-5-methyl-1,2,4-triazole-3(4H)-thione can react with aldehydes to give Schiff bases (Scheme 1). We studied their behavior toward silver(I) and copper(I) ions [12-14]. In this paper, we wish to report the molecular structures of new Schiff bases based on 1 .


Scheme 1.

## Results and Discussion

Imines 2-5 were prepared from amine $\mathbf{1}$ and the appropriate aldehyde (Scheme 1). Table 1 shows the crystallographic data of $\mathbf{2}, \mathbf{3}, \mathbf{4} \cdot \mathrm{MeOH}$, and 5. Selected bond lengths and angles are listed in Table 2. Imine 2 (Fig. 1) crystallizes in the monoclinic space group $P 2_{1} / n, 3$ (Fig. 3) in the triclinic space group $P \overline{1}, \mathbf{4} \cdot \mathrm{MeOH}$ (Fig. 4) in the orthorhombic space group Pbca and 5 (Fig. 6) in the monoclinic space group $P 2_{1} / c$.
The bond lengths S1-C1 and O1-C2 of 164.2(3) and $122.3(3) \mathrm{pm}(\mathbf{2}), 165.9(2)$ and $121.0(2) \mathrm{pm}(\mathbf{3})$, $165.9(2)$ and $120.0(2) \mathrm{pm}(4 \cdot \mathrm{MeOH})$, and $166.6(2)$

Table 1. Crystallographic data for 2, 3, 4• MeOH and 5.

| Compound | 2 | 3 | 4. MeOH | 5 |
| :---: | :---: | :---: | :---: | :---: |
| Empirical formula | $\mathrm{C}_{12} \mathrm{H}_{12} \mathrm{~N}_{4} \mathrm{O}_{2} \mathrm{~S}$ | $\mathrm{C}_{12} \mathrm{H}_{12} \mathrm{~N}_{4} \mathrm{O}_{2} \mathrm{~S}$ | $\mathrm{C}_{11} \mathrm{H}_{13} \mathrm{~N}_{5} \mathrm{O}_{2} \mathrm{~S}$ | $\mathrm{C}_{11} \mathrm{H}_{8} \mathrm{Cl}_{2} \mathrm{~N}_{4} \mathrm{OS}$ |
| Formula mass | 276.31 | 276.31 | 279.32 | 315.18 |
| Crystal size (mm) | $0.55 \times 0.25 \times 0.18$ | $0.68 \times 0.4 \times 0.28$ | $0.38 \times 0.16 \times 0.11$ | $0.32 \times 0.24 \times 0.07$ |
| Crystal system | monoclinic | triclinic | orthorhombic | monoclinic |
| Space group | $P 2_{1} / n$ | $P \overline{1}$ | Pbca | $P 2_{1} / \mathrm{c}$ |
| $a$ [pm] | 806.9(1) | 928.2(1) | 786.6(1) | 1345.1(2) |
| $b$ [pm] | 1377.5(3) | 1025.5(1) | 1631.9(2) | 817.1(1) |
| $c[\mathrm{pm}]$ | 1181.5(2) | 1382.8(2) | 2051.2(1) | 1267.2(2) |
| $\alpha\left[{ }^{\circ}\right]$ | 90 | 95.72(1) | 90 | 90 |
| $\left.\beta{ }^{\circ}{ }^{\circ}\right]$ | 106.37(1) | 102.17(1) | 90 | 104.50(1) |
| $\gamma\left[{ }^{\circ}\right]$ | 90 | 91.22(1) | 90 | 90 |
| Volume $\left[\mathrm{pm}^{3} \cdot 10^{6}\right.$ ] | 1260.0(4) | 1279.0(3) | 2633.0(5) | 1348.4(3) |
| Z | 4 | 4 | 8 | 4 |
| $D_{\text {calcd. }}\left[\mathrm{g} \mathrm{cm}^{-3}\right]$ | 1.457 | 1.435 | 1.409 | 1.553 |
| Absorption correction | numerical | numerical | numerical | numerical |
| $\mu_{\text {MoK }}$ [ $\left.\mathrm{cm}^{-1}\right]$ | 2.6 | 2.6 | 2.5 | 6.3 |
| Temperature [K] | 193 | 193 | 193 | 193 |
| $2 \theta$ Range | 52.27 | 52.59 | 52.52 | 51.90 |
| Index range |  |  |  |  |
| $h$ | $-9 \rightarrow 9$ | $-11 \rightarrow 10$ | $-9 \rightarrow 9$ | $-16 \rightarrow 16$ |
| $k$ | $-17 \rightarrow 17$ | $-12 \rightarrow 12$ | $-20 \rightarrow 20$ | $-9 \rightarrow 10$ |
| $l$ | $-14 \rightarrow 14$ | $-17 \rightarrow 17$ | $-25 \rightarrow 25$ | $-15 \rightarrow 15$ |
| Reflections collected | 12242 | 18878 | 35678 | 18395 |
| Unique reflections ( $R_{\text {int }}$ ) | 2374 (0.0949) | 5184 (0.0575) | 2645 (0.0878) | 2628 (0.0515) |
| Reflections with $F_{\mathrm{o}} \geq 4 \sigma\left(F_{\mathrm{o}}\right)$ | 1300 | 3957 | 1919 | 1853 |
| Parameters | 178 | 439 | 224 | 204 |
| $R_{1}$ | 0.036 | 0.0368 | 0.0347 | 0.0246 |
| $w R_{2}$ (all data) | $0.0717^{\text {a }}$ | $0.1025^{\text {b }}$ | $0.0846^{\text {c }}$ | $0.0506^{\text {d }}$ |
| $\left.\underline{\text { Largest diff. peak [(e pm }}{ }^{-3}\right) \cdot 10^{-6}$ ] | 0.22 | 0.30 | 0.16 | 0.21 |

${ }^{\mathrm{a}} w=1 /\left[\sigma^{2}\left(F_{\mathrm{o}}{ }^{2}\right)+(0.0289 P)^{2}\right] ; P=\left[\max \left(F_{\mathrm{o}}{ }^{2}, 0\right)+2{F_{\mathrm{c}}}^{2}\right] / 3 ; \quad{ }^{\mathrm{b}} w=1 /\left[\sigma^{2}\left(F_{\mathrm{o}}{ }^{2}\right)+(0.073 P)^{2}\right] ; \quad{ }^{\mathrm{c}} w=1 /\left[\sigma^{2}\left(F_{\mathrm{o}}{ }^{2}\right)+(0.0499 P)^{2}\right] ; \quad{ }^{\mathrm{d}} w=$ $1 /\left[\sigma^{2}\left(F_{0}^{2}\right)+(0.0265 P)^{2}\right]$.


Fig. 1. Molecular structure of 2 (thermal ellipsoids at the $50 \%$ probability level).
and $121.1(2) \mathrm{pm}(5)$ are indicative of a high double bond character as also observed in $\mathbf{1}$ [15]. The iminic C5-N2 bond distance (126.6(2)-127.6(3) pm) is in the range observed in similar compounds such as 2-acetylthiophene-thiosemicarbazone (129.2(3) pm), 2-acetylthiophene-4-phenyl-thiosemicarbazone (128.2(3) pm) [16] and 4-[(4-chloro-benzylidene)
amino]-3,4-dihydro-6-methyl-3-thioxo[1,2,4]triazine-3,4-5(2H)-5-one [14].

In all compounds the six-membered heterocyclic ring skeleton is planar. The dihedral angle between the "best planes" through the phenyl ring and the thiotriazine ring is $62^{\circ}$ for $2,85^{\circ}$ for $4 \cdot \mathrm{MeOH}$, and $85^{\circ}$ for 5 , respectively. The bond lengths in the heterocyclic ring indicate a delocalization of $\pi$-electrons. A medium strong hydrogen bond in 2 links the oxygen atom of one molecule to the NH group of the adjacent one (N4-H1…O1a: 297.7(2) pm) and is responsible for the building of chains along [101] (Fig. 2).

In 3, two heterocycles are linked together via hydrogen bonding between their NH groups and sulfur atoms, which leads to dimers with a centrosymmetric structure ( $\mathrm{N}-\mathrm{H} \cdots \mathrm{S}: 327.5(2) \mathrm{pm}$ ). In addition, each of the heterocycles is also linked via hydrogen bonding to the NH group of another molecule using its endocyclic nitrogen atom ( $\mathrm{N}-\mathrm{H} \cdots \mathrm{N}$ : 307.8(2) pm) (Fig. 3).

In $4 \cdot \mathrm{MeOH}$, two Schiff base molecules are linked together via the oxygen atom of the solvent molecule

Table 2. Selected bond lengths [pm] and bond angles $\left[{ }^{\circ}\right]$ in $\mathbf{2 , 3}, \mathbf{4} \cdot \mathrm{MeOH}$ and $\mathbf{5}$.

| $\mathbf{2}$ |  | $\mathbf{3}$ |  | $\mathbf{4}$ |  | $\mathbf{5}$ | $166.6(2)$ |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| S1-C1 | $164.2(3)$ | S1-C1 | $165.9(2)$ | S1-C1 | $165.9(2)$ | S1-C1 | $136.8(2)$ |
| N1-C1 | $137.9(3)$ | N1-C1 | $138.3(2)$ | N1-C1 | $138.0(2)$ | N1-C1 | $134.8(2)$ |
| N4-C1 | $135.4(3)$ | N3-C1 | $134.8(2)$ | N4-C1 | $134.8(2)$ | N4-C1 | $142.6(2)$ |
| N1-N2 | $142.6(2)$ | N1-N2 | $141.8(2)$ | N1-N2 | $142.0(2)$ | N1-N2 | $126.6(2)$ |
| N2-C5 | $127.6(3)$ | N2-C5 | $127.4(2)$ | N2-C5 | $127.0(2)$ | N2-C5 | $140.4(2)$ |
| N1-C2 | $139.7(3)$ | N1-C2 | $140.7(2)$ | N1-C2 | $140.2(2)$ | N1-C2 | $129.0(2)$ |
| N3-C3 | $129.2(3)$ | N3-C3 | $128.9(2)$ | N3-C3 | $129.4(2)$ | N3-C3 | $121.1(2)$ |
| O1-C2 | $122.3(3)$ | O1-C2 | $121.0(2)$ | O1-C2 | $120.0(2)$ | O1-C2 | $122.9(1)$ |
| S1-C1-N1 | $124.0(2)$ | S1-C1-N1 | $124.2(1)$ | S1-C1-N1 | $123.9(1)$ | S1-C1-N1 | $123.2(1)$ |
| S1-C1-N4 | $122.4(2)$ | S1-C1-N3 | $121.6(1)$ | S1-C1-N4 | $122.2(1)$ | S1-C1-N4 | $124.9(1)$ |
| C1-N1-C2 | $124.4(2)$ | C1-N1-C2 | $123.7(1)$ | C1-N1-C2 | $124.2(1)$ | C1-N1-C2 | $113.2(1)$ |
| N1-N2-C5 | $113.5(2)$ | N1-N2-C5 | $115.0(1)$ | N1-N2-C5 | $112.5(1)$ | N1-N2-C5 | $118.9(2)$ |
| N2-N1-C1 | $115.9(2)$ | N2-N1-C1 | $114.6(2)$ | N2-N1-C1 | $117.9(1)$ | N2-N1-C1 | $127.4(2)$ |
| N3-N4-C1 | $127.5(2)$ | N4-N3-C1 | $128.0(2)$ | N3-N4-C1 | $127.2(2)$ | N3-N4-C1 | $117.5(1)$ |
| N4-N3-C3 | $117.7(2)$ | N3-N4-C3 | $116.9(1)$ | N4-N3-C3 | $118.1(1)$ | N4-N3-C3 | $121.8(2)$ |
| O1-C2-N1 | $122.1(2)$ | O1-C2-N1 | $122.3(2)$ | O1-C2-N1 | $121.6(2)$ | O1-C2-N1 | 10 |



Fig. 2. Perspective view of the crystal structure of 2. The molecules of $\mathbf{2}$ are linked in the direction [101].
by hydrogen bonding (N4-H1 $\cdots \mathrm{O} 2: 271.8(2)$ and $\mathrm{O} 2-$ $\mathrm{H} 2 \cdots \mathrm{~N} 5 \mathrm{a} 277.5(2) \mathrm{pm}$ ) (Fig. 4). This coordination mode leads to formation of chains along [100] (Fig. 5).

A weak hydrogen bonding links the sulfur atom of one molecule of 5 to the NH-group of the adjacent one (N4-H1 $\cdots$ S1A 326.9(2) pm) and is responsible for the centrosymmetric structure of the aggregate (Fig. 6).
The IR spectra of the compounds show characteristic bands at $1606 \mathrm{~cm}^{-1}(\mathbf{2}), 1605 \mathrm{~cm}^{-1}(\mathbf{3}), 1606 \mathrm{~cm}^{-1}$


Fig. 3. Tetrameric arrangement through hydrogen bonding in 3 (thermal ellipsoids at the $50 \%$ probability level).


Fig. 4. Molecular structure of $\mathbf{4} \cdot \mathrm{MeOH}$ (thermal ellipsoids at the $40 \%$ probability level).
$(4 \cdot \mathrm{MeOH})$ and $1614 \mathrm{~cm}^{-1}(\mathbf{5})$, which can be assigned as stretching vibration bands of the azomethine group $(v(\mathrm{~N}=\mathrm{CH}))$. A similar range is also reported in the literature [1]. ${ }^{1} \mathrm{H}$ NMR spectra of the compounds show sharp peaks (singlets) at $\delta=8.88$ (2), 8.99 (3), 8.72 $(\mathbf{4} \cdot \mathrm{MeOH})$, and $\delta=8.79(\mathbf{5})$, which can be assigned to the azomethine proton.


Fig. 5. Graphical representation of the unit cell of $4 \cdot \mathrm{MeOH}$. The molecules of $\mathbf{4} \cdot \mathrm{MeOH}$ are linked in the direction [100].


Fig. 6. Dimeric arrangement of $\mathbf{5}$ by hydrogen bonding (thermal ellipsoids at the $40 \%$ probability level).

## Experimental Section

## General remarks

1, 2 and 5 were prepared according to literature procedures [8, 17]. IR spectra were recorded on a Shimadzu spectrometer 470 ( KBr pellets, Nujol mulls). ${ }^{1} \mathrm{H}$ and ${ }^{13} \mathrm{C}$ NMR spectra were recorded on a Bruker AX 200 spectrometer
using TMS as an external standard. For the EI mass spectra, a Varian CH7A mass spectrometer ( 70 eV ) was used. All chemicals and solvents were purchased from Merck and Fluka and were used without further purification or drying.

General procedure for the reaction of $\mathbf{1}$ with aldehydes under classical heating

A solution of $1(4 \mathrm{mmol})$ in $\mathrm{EtOH}(30 \mathrm{~mL})$ was treated with an aldehyde in a molar ratio of $1: 1.5$ and the resulting mixture was acidified by $37 \%$ hydrochloric acid ( 3 drops). The reaction mixture was refluxed for the indicated time. The solid residue was filtered, washed with cold ethanol ( 10 mL ) and recrystallized from EtOH or MeOH .

General procedures for the reaction of 1 with aldehydes under microwave irradiation in a solventfree system
$\mathbf{1}(1 \mathrm{mmol})$ was mixed with aldehyde ( 1 mmol ) in a molar ratio of $1: 1.5$ in a beaker. The beaker was placed in a microwave oven for the appropriate time. The reaction mixture was dissolved in ethanol or methanol ( 2 mL ), the solid residue was filtered, washed with cold ethanol $(5 \mathrm{~mL})$ and recrystallized from EtOH .

4-\{[(1E)-(4-Methoxyphenyl)methylene]amino\}-6-methyl-3-thioxo-3,4-dihydro-1,2,4-triazin-5(2H)-one (3)

3 was obtained from 1 and 4-methoxybenzaldehyde as a pale yellow crystalline solid, and the reaction time was 12 h under classical heating or 180 s under microwave irradiation. Both reactions led to the same compound. Yield: $91 \%$. - M.p. $208-210{ }^{\circ} \mathrm{C}$. - IR ( KBr disc): $v=3143$ $(\mathrm{NH}), 1693(\mathrm{C}=\mathrm{O}), 1606\left(\mathrm{~N}=\mathrm{CH}_{\text {imine }}\right), 1565\left(\mathrm{CN}_{\text {triazine }}\right)$ $\mathrm{cm}^{-1} .-{ }^{1} \mathrm{H}$ NMR (acetone): $\delta=2.22\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{CH}_{3}\right), 3.15$ (s, $3 \mathrm{H}, \mathrm{OCH}_{3}$ ), $6.93(\mathrm{~d}, 2 \mathrm{H}, \operatorname{Ar}-H, J=7.5 \mathrm{~Hz}$ ), 7.25 (d, $2 \mathrm{H}, \operatorname{Ar}-H, J=8.18 \mathrm{~Hz}), 8.72\left(\mathrm{~s}, 1 \mathrm{H}, \mathrm{CH}_{\mathrm{imine}}\right), 10.00$ (s, $1 \mathrm{H}, \mathrm{N} H_{\text {triazine }}$ ). $-\mathrm{MS}: m / z=276\left[\mathrm{M}^{+}\right], 143\left[\mathrm{M}^{+}\right.$$\mathrm{ArCN}], 133,119,103\left[\mathrm{C}_{3} \mathrm{H}_{7} \mathrm{~N}_{3} \mathrm{O}^{+}\right], 90,77,42\left[\mathrm{C}_{2} \mathrm{H}_{4} \mathrm{~N}^{+}\right]$. $\mathrm{C}_{12} \mathrm{H}_{12} \mathrm{~N}_{4} \mathrm{O}_{2} \mathrm{~S}$ (276.31): calcd. C 52.17, H 4.36, N 20.25; found: C 51.92, H 4.23, N 19.68.

6-Methyl-4-\{[(1E)-(2-pyridyl)methylene]amino\}-3-thioxo-3,4-dihydro-1,2,4-triazin-5(2H)-one (4)

4 was obtained from 1 and pyridine-2-carbaldehyde. The reaction time for 4 was 14 h under classical heating or 180 s under microwave irradiation. Both reactions led to the same compound, which was recrystallized from methanol to give $4 \cdot \mathrm{MeOH}$. Yield: $82 \%$. - M. p. $148-149{ }^{\circ} \mathrm{C}$. - IR (KBr disc): $v=3215(\mathrm{NH}), 1683(\mathrm{C}=\mathrm{O}), 1614\left(\mathrm{~N}=\mathrm{CH}_{\text {imine }}\right)$, $1582\left(\mathrm{CN}_{\text {triazine }}\right) \mathrm{cm}^{-1} .-{ }^{1} \mathrm{H}$ NMR (acetone): $\delta=2.19$ (s, $3 \mathrm{H}, \mathrm{CH}_{3}$ ), $7.62-8.67(\mathrm{~m}, 4 \mathrm{H}, \mathrm{Ar}-H), 8.79(\mathrm{~s}, 1 \mathrm{H}$, $\left.\mathrm{C} H_{\text {imine }}\right), 13.71\left(\mathrm{~s}, 1 \mathrm{H}, \mathrm{N} H_{\text {triazine }}\right) .-\mathrm{MS}: m / z=247$ $\left[\mathrm{M}^{+}\right], 143\left[\mathrm{M}-\mathrm{ArCN}^{+}\right], 137,122,102\left[\mathrm{C}_{3} \mathrm{H}_{7} \mathrm{~N}_{3} \mathrm{O}^{+}\right], 90,77$,
$42\left[\mathrm{C}_{2} \mathrm{H}_{4} \mathrm{~N}^{+}\right] .-\mathrm{C}_{10} \mathrm{H}_{9} \mathrm{~N}_{5} \mathrm{OS}$ (247.28; 4): calcd. C 48.58, H 3.64, N 28.34; found: C 48.76, H 3.48, N 28.12.

## Crystal structure analyses of 2, 3, 4•MeOH and 5

The crystals of $\mathbf{2}, \mathbf{3}, \mathbf{4} \cdot \mathrm{MeOH}$ and $\mathbf{5}$ were covered with perfluorinated oil and mounted on the top of a glass capillary under a flow of cold gaseous nitrogen (Stoe-IPDS II diffractometer, graphite-monochromated $\mathrm{Mo} K_{\alpha}$ radiation, $\lambda=71.073 \mathrm{pm}$ ). The intensities were corrected for Lorentz and polarization effects. In addition, numerical absorption corrections were applied for all compounds. The structures were solved by Direct Methods using SHELXS-97
[18] and refined against $F^{2}$ by full-matrix least-squares using the program SHELXL-97 [19]. The positions of carbon bonded hydrogen atoms (except H 1 ) in 2 were calculated and refined with a common displacement parameter. H1 atoms in 2 and all hydrogen atoms in 3, 4• MeOH and 5 were included with a free refinement. Programs used were SHELXS-97 [18], SHELXL-97 [19], SHELXTL-Plus [20], ORTEP [21], and PLATON [22]. Further details can be obtained from The Cambridge Crystallographic Data Centre by quoting the numbers CCDC 617517 (2), 617518 (3), 617519 (4 • MeOH) and 617520 (5) via www.ccdc.cam.ac.uk/ data_request/cif.
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