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Efficient and Stereoselective Synthesis of α-Hydrazino Tetrazoles through a Pseudo Five-component Domino Reaction

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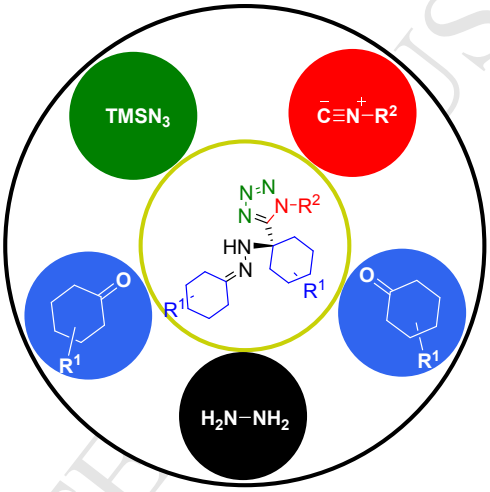


Abstract:

A concise and efficient route for the diastereoselective synthesis of new and interesting α-hydrazino tetrazoles has been developed through a pseudo five-component domino reaction using the isocyanide-based one-pot multicomponent reaction. The products were obtained by a facile Ugi-azide pseudo five-component reaction of cyclic ketones, hydrazine hydrate, trimethylsilyl azide, and corresponding isocyanides without any catalyst in good yields and with high bond forming efficiency at room temperature.

Keywords: Ugi-azide pseudo five-component reaction, α-hydrazino tetrazoles, isocyanide-based one-pot multicomponent reaction.

Dedicated to Prof. Mohammad Yalpani on the occasion of his birthday

Introduction

Tetrazoles represent an important class of heterocyclic nitrogen compound with a wide range of biological properties such as antimicrobial, antibacterial, antifungal, analgestic, anti-inflammatory, antiviral, and anti-cancer activities.[1] In recent years, considerable attention has been drawn to 1,5-disubstituted tetrazoles, the more common tetrazole derivatives, due to their biological importance and functional properties.[2] As such, they are important in biology and medicine as NADPH oxidase inhibitors, glucokinase activators, hepatitis C virus (HCV) serine protease NS3 inhibitors, calcitonin gene-related peptide receptor antagonists and antimigraine agents.[3] They have also attracted a great deal of attention in the field of peptide chemistry as conformational mimics of *cis*-amide bonds.[4] Prescribed agents featuring the 1,5-disubstituted tetrazole scaffold include the anti-inflammatory cilostazol, the anti-microbial latamoxef, the circulatory and respiratory stimulant pentylenetetrazol and the anti-diabetic nojiritetrazole.[5]

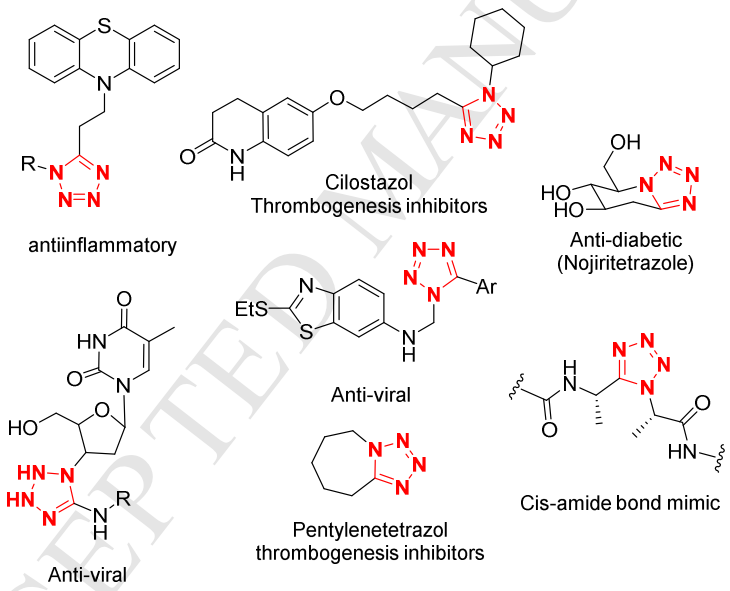
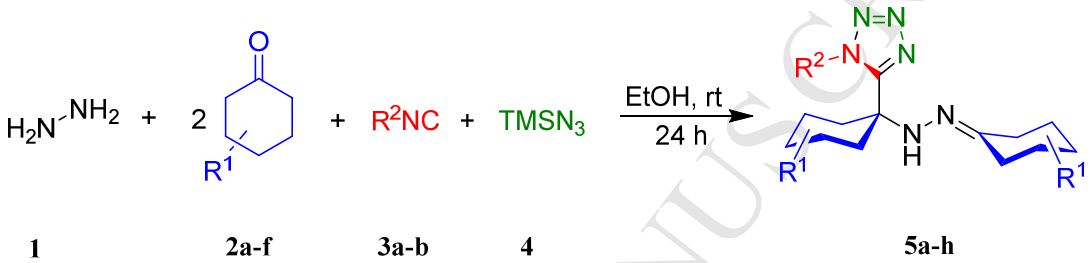


Figure. 1 1,5-Disubstituted tetrazoles as conformational mimics of *cis*-amide bonds, and as medicinally important compounds.

There has been considerable interest in the synthesis of 1,5-disubstituted tetrazoles owning to their diverse pharmacological activities and numerous approaches to this scaffold have been disclosed in the literature. Major synthetic strategies utilized for the preparation of 1,5-disubstituted tetrazoles are via intermolecular cycloaddition reactions, and isocyanide-based multicomponent reactions.[6] Among the various pathways toward 1,5-disubstituted tetrazole derivatives, the isocyanide-based multicomponent reactions (IMCRs) are most relevant by virtue of their synthetic potential, inherent atom efficiency, convergent nature, ease of implementation, and the generation of molecular

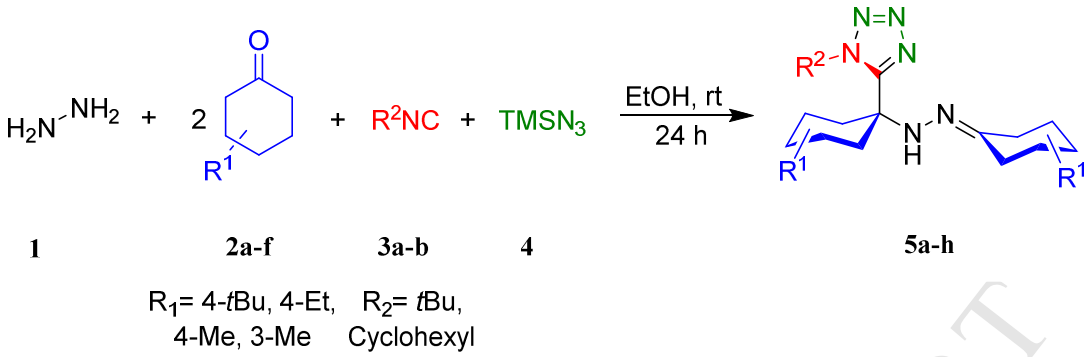
diversity.[7] Recently, we have developed a non-catalytic diastereoselective approach to these important heterocyclic nitrogen compounds through a facile Ugi-azide four-component reaction.[8] As part of our continuing efforts to improve the preparation of biologically important 1,5-disubstituted tetrazoles by means of isocyanide-based multicomponent reactions, herein we describe a concise and efficient route for the diastereoselective synthesis of new and interesting α-hydrazino tetrazoles via a facile Ugi-azide pseudo five-component reaction of two equivalents of cyclic ketones, hydrazine hydrate, isocyanides and trimethylsilyl azide (Scheme 1).



Scheme 1. Stereoselective synthesis of α-hydrazino tetrazoles **5a-h** through a facile Ugi-azide pseudo five-component reaction

Results and discussion

The Ugi-azide pseudo five-component reaction of hydrazine hydrate **1**, 2 equivalents of 4-*tert*-butyl cyclohexanone **2a**, *tert*-butyl isocyanide **3a** and trimethylsilyl azide **4** in ethanol was selected as the model reaction. The reaction mixture was stirred at room temperature to produce the corresponding α-hydrazino tetrazole (**5a**) in 89% yield (Table 1). At the outset of this study, our efforts were focused on finding appropriate reaction conditions to perform the proposed reaction. Consequently, the model reaction was carried out in various solvents. The results are summarized in Table 1. The use of acetonitrile, tetrahydrofuran, and dichloromethane in place of ethanol/methanol as the reaction solvent decreased the yield of the desired product (entries 1-3). Therefore, ethanol was used as the reaction solvent.



Scheme 2. The model reaction for the synthesis of **5a** through the Ugi-azide pseudo five-component reaction

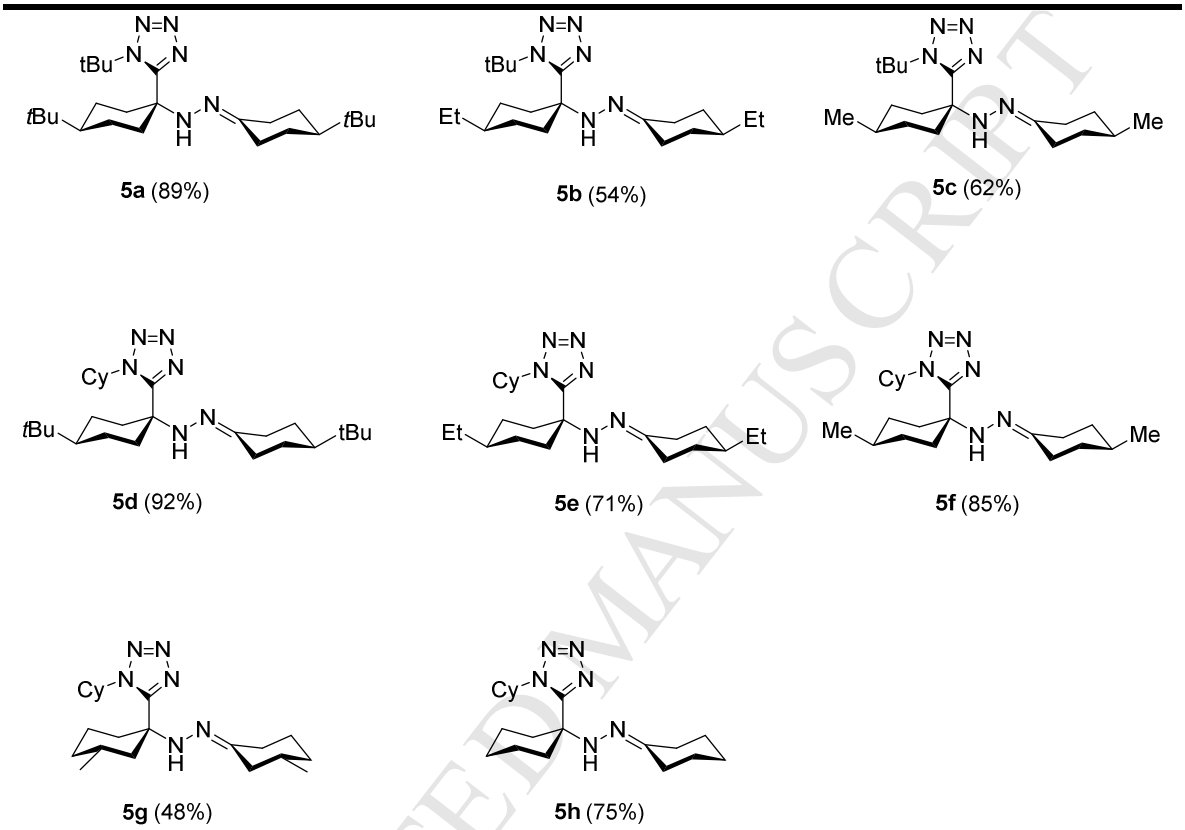
Table 1. Optimization of the solvent reaction

Entry	Solvent	Yield (%)[a]
1	THF	17
2	CH_2Cl_2	23
3	CH_3CN	35
4	CH_3OH	89
5	CH_3CH_2OH	89

[a] Isolated yields

Furthermore, when TMS-N_3 in the reaction was replaced by NaN_3 we could isolate only 37% of **5a**. In another attempt, the ratio of isocyanides and also trimethylsilyl azide was raised to 2.5 equivalents in the model reaction to obtain the product containing two tetrazole ring moieties. However, the sole product was **5a:** the desired product could not form possibly due to stereoelectronic effects. With optimized reaction conditions in hand for the preparation of **5a**, we decided to check the scope of the reaction with various cyclic ketones and isocyanides. The results obtained are summarized in Table 2. In our initial studies, the formation of single atropisomers were determined by ^{1}H NMR spectroscopic data on the crude reaction products through the single -NH signal and the single C<u>H</u>-N signal of the corresponding products, resonating at δ 4.30-4.60 and δ 5.00-5.20 respectively (Table 2).

Table 2. The facile Ugi-azide pseudo five-component reaction for the diastereoselective synthesis of hydrazino tetrazoles **5a-h**



The structures of the products **5a-h** were deduced from their IR, ^{1}H NMR, ^{13}C NMR and ESI-HRMS spectra. For example, the ^{1}H NMR spectrum of **5a** consisted of a singlet for the distinguished -NH peak at δ 4.36 ppm, two singlets for two *tert*-butyl groups at δ 0.82 and 0.86 and a singlet at δ 1.78 ppm for the –N-*tert*-butyl group. The proton decoupled ^{13}C NMR spectrum of **5a** showed 24 distinct resonances in agreement with the proposed structure. The distinguished peaks resonated at δ 63.2 and 64.4 ppm related to –C-N groups. The C=N tetrazole and imine group resonated at δ 152.7 and 157.7 ppm, respectively.

Meanwhile, the structures of **5a** and **5d** were subsequently confirmed by single-crystal X-ray crystallographic data (Fig. 2). As shown in Fig. 2 the orientation of tetrazole scaffold is axial, and the hydrazine group is in the equatorial position.

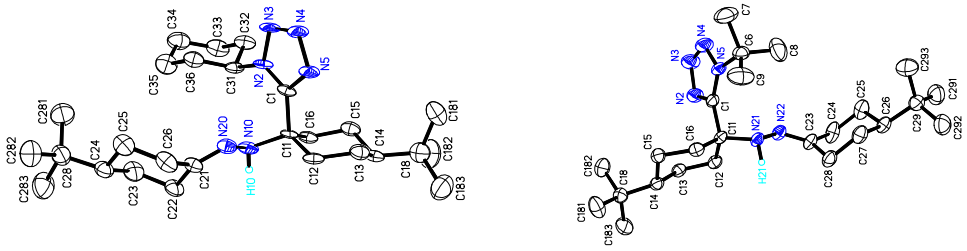
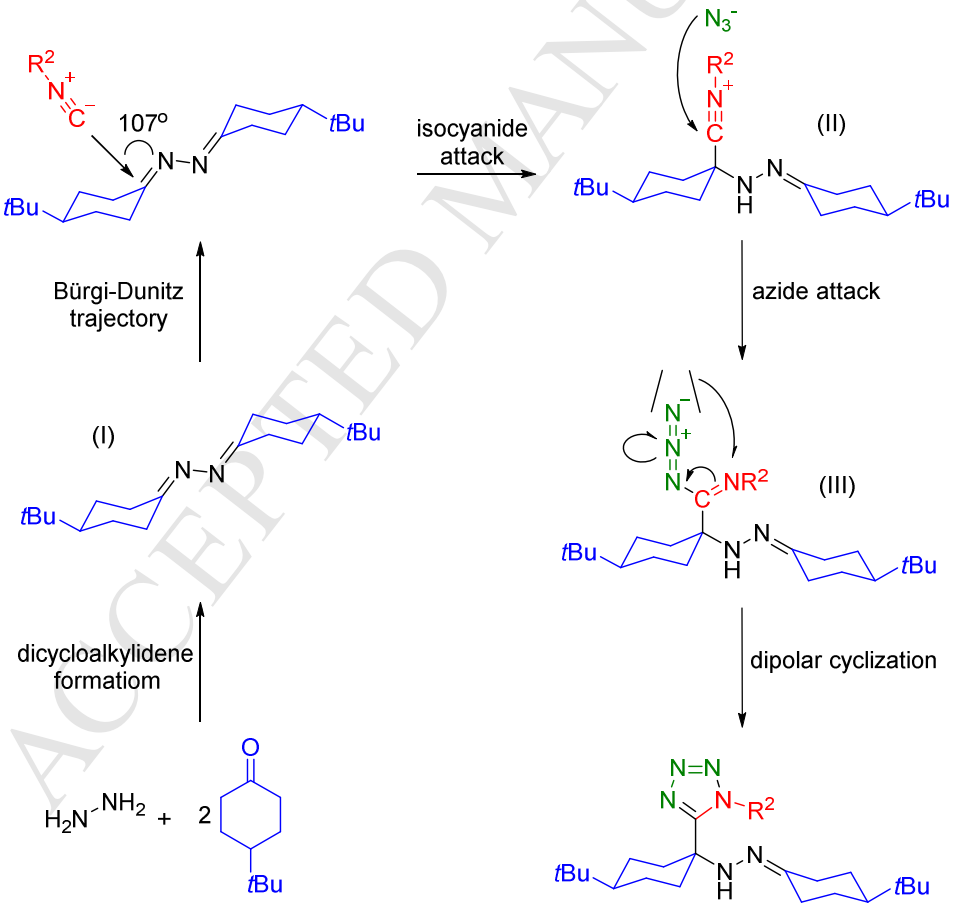


Fig 2 The ORTEP structure of **5a** and **5d**

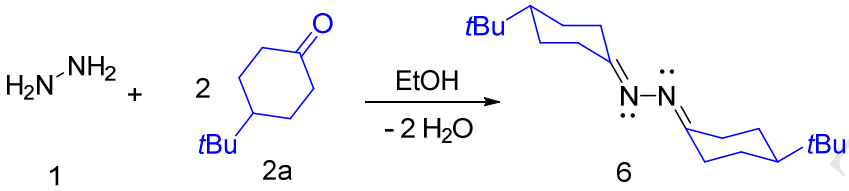
The C=N bond lengths in **5a** and **5d** (1.277 Å, 1.266 Å, respectively) could confirm the existence of imine bonds in the desired structures.

A mechanistic rationalization for this reaction is provided in Scheme 3. The first step seems to be the formation of the diimines, followed by nucleophilic attack of isocyanide and trimethylsilyl azide leading to the formation of the intermediate, which undergoes cyclization to afford the corresponding products.



Scheme 3. Proposed mechanism for the synthesis of α-hydrazino tetrazoles **5a-h** through pseudo-five-component reaction

To confirm the formation of the diimines, the reaction of two equivalents of 4-*tert*-butyl cyclohexanone and 1 equivalent of hydrazine hydrate was investigated leading to the formation of diimin**e 6** in the *anti*-configuration, the most stable conformation.



Scheme 4. Synthesis of diimine **6** through the reaction of 4-*tert*-butyl cyclohexanone and hydrazine hydrate

In the next step, there are two possibilities for the nucleophilic addition of isocyanide and trimethylsilyl azide to the diimine intermediate through an axial or equatorial approach. In our previous report regarding the synthesis of α-hydrazido tetrazoles through the Ugi-azide one-pot four-component reaction, the transition state for axial attack of isocyanide suffered from steric clashes between the 1,3 axial hydrogens and the incoming isocyanide reagent and the addition occurred via equatorial attack.[8] In contrast to our previously reported article, here, the addition of isocyanide through equatorial attack is not suitable due to the a) torsional strain due to eclipsing interactions between the hydrazonyl group and also β C-H hydrogens; b) less stability of the product as it is not the thermodynamic product (Fig.3); c) based on the Cieplack model the axial nucleophilic addition of isocyanide is more preferable due to effective stabilizing orbitals interaction (the C-H bond has more donation character compared to C-C bond)[9] (Fig.4).

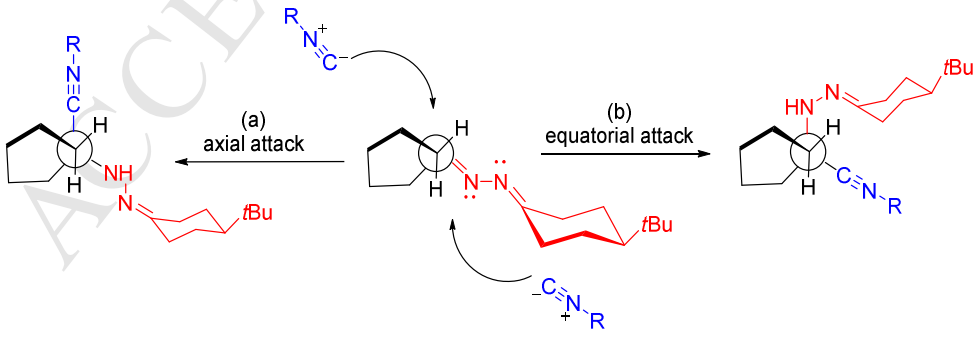


Fig 3. Proposed addition reaction of isocyanide to diamine

Fig4. The Cieplak model for the effective stabilizing interaction for axial nucleophilic addition[9]

In conclusion, we have developed a convenient, simple and efficient diastereoselective synthesis of thermodynamically stable α-hydrazino tetrazoles via a facile Ugi-azide pseudo five-component reaction. The present methodology offers several advantages, such as simple procedure with an easy work-up, high yields, high atom-economy, high bond forming efficiency and carrying out the reaction in one-pot reaction conditions at room temperature.

Experimental Section:

Commercially available materials were used without further purification. Melting points were determined on an Electrothermal 9100 apparatus and were uncorrected. IR spectra were obtained on an ABB FT-IR FTLA 2000 spectrometer. ^{1}H NMR and ^{13}C NMR spectra were run on Bruker DRX-300 AVANCE spectrometers at 300 MHz for ^{1}H NMR, 75 MHz for ^{13}C NMR. DMSO-d_6 and $CDCl_3$ was used as solvent. High resolution mass spectra were recorded on Mass-ESI-POS (Apex Qe-FT- ICR instrument) spectrometer.

General procedure for the synthesis of α-hydrazino tetrazoles 5a-h:

To a solution of hydrazine hydrate **1** (1 mmol), cyclic ketone **2a-f** (2 mmol) in ethanol (5 mL) was added. The mixture was stirred for 30 min then isocyanide **3a-b** (1.2 mmol) and trimethylsilyl azide **4** (1 mmol) was added to the mixture. The mixture was stirred for 24h at ambient temperature. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 1:3), the precipitate was filtered off and then washed with methanol.

Spectroscopic data for compounds **5a-h:**

1-(*tert*-Butyl)-5- ((1r,4S)-4- (*tert*-butyl)-1-(2 -((S,*E*)-4 -(*tert*-butyl)cyclohexylidene) hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5a**): Colorless powder; 383 mg (89%), mp: 209-211^{o}C, IR ν_{max} (KBr, cm^{-1}): 3258, 1639; ^{1}H NMR (300 MHz, $CDCl_3$): 0.82 (*s*, 9H, $3CH_3$, *t*-Bu), 0.86 (*s*, 9H, $3CH_3$, *t*-Bu), 1.07-1.72 (*m*, 8H, $4CH_2$, $H_{Cyclohexyl}$), 1.78 (*s*, 9H, $3CH_3$, *t*-Bu), 1.82-2.01 (*m*, 4H, $H_{Cyclohexyl}$), 2.28-2.50 (*m*, 2H, $H_{Cyclohexyl}$), 2.60-2.81 (*m*, 4H, $H_{Cyclohexyl}$), 4.36

(*s*, 1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): 23.9, 24.2, 25.1, 26.3, 27.4, 27.5, 27.7, 31.2, 32.3, 32.4, 34.8, 37.9, 38.5, 47.4, 47.6, 59.3, 64.4, 152.6, 157.8; HR-MS (ESI): calc. for $C_{25}H_{47}N_6$ $[M+H]^+$ 431.3857, found 431.3857; calc. for $C_{25}H_{46}N_6Na$ $[M+Na]^+$ 453.3677, found 453.3676; calc. for $C_{25}H_{46}N_6K$ $[M+K]^+$ 469.3416, found 469.3415.

colourless crystal (plate), dimensions 0.370 x 0.230 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.2048(7) Å, b=29.336(3) Å, c=14.4809(17) Å, alpha=90 deg, beta=98.484(4) deg, gamma=90 deg, V=2607.0(5) $Å^3$, rho=1.097 g/cm^3, T=200(2) K, $Theta_{max}$= 26.022 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.15 and a completeness of 100.0% to a resolution of 0.81Å, 16371 reflections measured, 5139 unique (R(int)=0.0805), 3027 observed (I > 2□(I)), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using SADABS based on the Laue symmetry of the reciprocal space, mu=0.07mm^{-1}, T_{min}=0.90, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package, 285 parameters refined, hydrogen atoms were treated using appropriate riding models, except H21 at N21, which was refined isotropically, goodness of fit 0.99 for observed reflections, final residual values R1(F)=0.057, $wR(F^2)$=0.109 for observed reflections, residual electron density -0.22 to 0.21 $eÅ^{-3}$. CCDC 103930 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

1-(*tert*-Butyl)-5-((1*r*,4S)-4-ethyl-1-(2-((S,*E*)-4-ethyl cyclohexylidene) hydrazinyl) cyclohexyl) -1*H*-tetrazole (**5b**): Waxy liquid; 202 mg (54%), IR □$_{max}$ (KBr, cm^{-1}): 3362, 1713; ^{1}H NMR (300 MHz, $CDCl_3$): 0.85 (*t*, *J* = 7.0 Hz, 3H, CH_3), 0.91 (*t*, *J* = 7.0 Hz, 3H, CH_3), 0.94-1.03 (*m*, 2H, $H_{Cyclohexyl}$), 1.22-1.37 (*m*, 7H, $H_{Cyclohexyl}$), 1.61-1.88 (*m*, 8H, $H_{Cyclohexyl}$), 1.78 (*s*, 9H, $3CH_3$, *t*-Bu), 2.00-2.23(*m*, 2H, $H_{Cyclohexyl}$), 2.53-2.67 (*m*, 3H, $H_{Cyclohexyl}$), 4.54 (*brs*, 1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): 11.4, 11.6, 11.7, 24.4, 28.1, 28.2, 28.9, 31.2, 31.3, 31.5, 32.8, 34.4, 35.6, 35.9, 37.5, 38.6, 59.4, 64.3, 152.7, 158.4; HR-MS (ESI): calc. for $C_{21}H_{39}N_6$ $[M+H]^+$ 375.3360, found 375.3357; calc. for $C_{21}H_{38}N_6Na$ $[M+Na]^+$ 397.3038, found 397.3035; calc. for $C_{21}H_{38}N_6K$ $[M+K]^+$ 413.2787, found 413.2785.

1-(*tert*-Butyl)-5-((1*r*,4S)- 4-methyl-1- (2 -((S, *E*)-4-methylcyclohexylidene) hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5c**): Colorless solid; 215 mg (62%), mp: 120-123 ^{o}C, IR $\square_{max}$ (KBr, cm^{-1}): 3255, 1632; ^{1}H NMR (300 MHz, $CDCl_3$): 0.89 (*d*, 6H, *J* = 6.3 Hz, $2CH_3$), 0.92-1.70 (*m*, 10H, $H_{cyclohexyl}$), 1.74 (*s*, 9H, *t*-Bu), 1.76-2.66 (*m*, 8H, $H_{cyclohexyl}$), 4.62 (*s*, 1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): 20.9, 21.6, 24.3, 30.3, 30.5, 30.6, 31.0, 31.2, 32.0, 33.8, 34.5, 35.2, 35.6, 59.1, 64.3, 76.6, 152.2, 158.5; HR-MS (ESI): HR-MS (ESI): calc. for $C_{19}H_{35}N_6$ $[M+H]^+$ 347.2428, found 347.2428.

5-((1r,4S)-4-(*tert*-Butyl)-1-(2-((S,*E*)-4-(*tert*-Butyl)cyclohexylidene)hydrazinyl) cyclohexyl)-1-cyclohexyl-1*H*-tetrazole (**5d**): Colorless solid; 420 mg (92%), mp: 239-241 ^{o}C, IR $\square_{max}$ (KBr, cm^{-1}): 3253, 1642; ^{1}H NMR (300 MHz, $CDCl_3$): 0.77 (*s*, 9H, $3CH_3$, *t*-Bu), 0.84 (*s*, 9H, $3CH_3$, *t*-Bu), 0.88-1.50 (*m*, 10H, $H_{Cyclohexyl}$), 1.53-1.82 (*m*, 6H, $H_{Cyclohexyl}$), 1.83-2.24 (*m*, 9H, $H_{Cyclohexyl}$), 2.28-2.79 (*m*, 3H, $H_{Cyclohexyl}$), 4.49 (*s*, 1H, NH), 4.90-5.11 (*m*, 1H, CNH); ^{13}C NMR (75 MHz, $CDCl_3$): 21.6, 22.0, 24.8, 25.0, 25.7, 26.5, 27.3, 27.7, 32.3, 32.5, 33.1, 33.2, 34.2, 34.8, 46.4, 47.4, 56.8, 58.9, 153.4, 158.5; HR-MS (ESI): calc. for $C_{27}H_{49}N_6$ $[M+H]^+$ 457.40144, found 457.40132; calc. for $C_{27}H_{49}N_6Na$ $[M+Na]^+$ 479.3834, found 479.3833; calc. for $C_{27}H_{48}KN_6$ $[M+K]^+$ 495.3573, found 495.3572.

Colourless crystal (needle), dimensions 0.420 x 0.050 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.345(2) Å, b=31.355(11) Å, c=14.286(5) Å, alpha=90 deg, beta=99.872(11) deg, gamma=90 deg, V=2799.8(17) Å^3, rho=1.083 g/cm^3, T=200(2) K, $Theta_{max}$= 21.260 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.63and a completeness of 61.5% to a resolution of 0.98Å, 11459 reflections measured, 3120 unique (R(int)=0.1049), 1898 observed (I > 2□(I)), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using SADABS based on the Laue symmetry of the reciprocal space, mu=0.06mm^{-1}, T_{min}=0.82, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package, 302 parameters refined, hydrogen atoms were treated using appropriate riding models, except H10 at N10, which was refined isotropically, goodness of fit 1.01 for observed reflections, final residual values R1(F)=0.074, wR(F^2)=0.159 for observed reflections, residual electron density -0.33 to 0.32 eÅ^{-3}. CCDC 103931 contains the supplementary crystallographic data for this paper.

These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

1-Cyclohexyl-5- ((1*r*,4S)-4-ethyl-1 -(2 -((S,*E*)-4-ethylcyclohexylidene)hydrazinyl) cyclohexyl)-1H-tetrazole (**5e**): Colorless solid; 216 mg (71%), mp: 129-130 ^{o}C, IR $\square_{max}$ (KBr, cm^{-1}): 3244, 1739, 1635; ^{1}H NMR (300 MHz, $CDCl_3$): 0.84 (*t*, 3H, *J* = 7.2 Hz, CH_3), 0.87 (*t*, 3H, *J* = 7.2 Hz, CH_3), 0.97-1.02 (*m*, 3H, $H_{Cyclohexyl}$), 1.78-1.38 (*m*, 10H, $H_{Cyclohexyl}$), 1.63-2.24 (*m*, 16H, $H_{Cyclohexyl}$), 2.53-2.70 (*m*, 3H, $H_{Cyclohexyl}$), 4.59 (*s*,1H, NH), 5.07-5.14 (*m*, 1H, CNH); ^{13}C NMR (75 MHz, $CDCl_3$): 11.5, 11.6, 24.3, 25.0, 25.6, 28.5, 28.6, 28.8, 31.3, 32.6, 33.2, 34.4, 35.3, 35.7, 38.5, 58.3, 59.1, 153.6.2; HR-MS (ESI): calc. for $C_{23}H_{41}N_6$ $[M+H]^+$ 401.3388, found 401.3387; calc. for $C_{23}H_{40}N_6$ $[M+Na]^+$ 423.3197, found 432.3193; calc. for $C_{23}H_{40}KN_6$ $[M+K]^+$ 439.2947, found 439.2946

1-Cyclohexyl-5 -((1*r*,4S)-4-methyl-1-(2 -((S, *E*)-4-methylcyclohexylidene) hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5f**) Colorless solid; 291 mg (78%), mp: 167-169 ^{o}C, IR $\square_{max}$ (KBr, cm^{-1}): 3240, 1739, 1631; ^{1}H NMR (300 MHz, $CDCl_3$): 0.85 (*d*, 3H, *J* = 6.4 Hz, CH_3), 0.88 (*d*, 3H, *J* = 6.5 Hz, CH_3), 0.93-1.56 (*m*, 10H, $H_{Cyclohexyl}$), 1.58-1.78 (*m*, 8H, $H_{Cyclohexyl}$), 1.79-2.20 (*m*, 8H, $H_{Cyclohexyl}$), 2.45-2.66 (*m*, 2H, $H_{Cyclohexyl}$), 4.6 (*s*, 1H, NH), 5.10 (*m*, 1H, NCH); ^{13}C NMR (75 MHz, $CDCl_3$): 21.4, 21.6, 24.3, 25.0, 25.6, 30.8, 30.9, 31.7, 31.8, 33.1, 33.2, 33.7, 34.5, 35.1, 35.3, 35.8, 56.9, 58.0, 59.1, 153.3, 156.2, HR-MS (ESI): calc. for $C_{21}H_{37}N_6$ $[M+H]^+$ 373.3074, found 373.3074; calc. for $C_{21}H_{36}N_6Na$ $[M+Na]^+$ 395.2894, found 395.2894; calc. for $C_{21}H_{36}KN_6$ $[M+K]^+$ 411.2633, found 411.2633.

1-Cyclohexyl-5- ((1R, 3R)-3-methyl-1- (2-((S, *Z*)-3-methylcyclohexylidene)hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5g**): Colorless solid; 231 mg (65%), mp: 145-147 ^{o}C, IR $\square_{max}$ (KBr, cm^{-1}): 3238, 1740, 1632; ^{1}H NMR (300 MHz, $CDCl_3$) 0.85-0.95 (*m*, 6H, CH_3), 1.00-1.55 (*m*, 10H, $H_{Cyclohexyl}$), 1.63-1.79 (*m*, 6H, $H_{Cyclohexyl}$), 1.87-2.21 (*m*, 9H, $H_{Cyclohexyl}$), 2.48-2.65 (*m*, 3H, $H_{Cyclohexyl}$), 4.56 (*s*, 1H, NH), 5.05-5.13 (*m*, 1H, CHN); ^{13}C NMR (75 MHz, $CDCl_3$): 22.2, 22.4, 22.5, 25.0, 25.6, 25.7, 29.0, 32.3, 33.2, 33.3, 34.1, 34.4, 34.7, 35.9, 44.4, 58.9, 59.1, 59.2, 153.3, 156.1; 156.1; HR-MS (ESI): calc. for $C_{21}H_{37}N_6$ $[M+H]^+$ 373.3074, found 373.3074; calc. for $C_{21}H_{36}N_6Na$ $[M+Na]^+$ 395.2894, found 395.2894; calc. for $C_{21}H_{36}KN_6$ $[M+K]^+$ 411.2633, found 411.2633.

1-Cyclohexyl-5-(1-(2-cyclohexylidenehydrazinyl)cyclohexyl)-1H-tetrazole(**5h**): Colorless powder; 258 mg (75%), mp: 147-150 ^{o}C, IR ν_{max} (KBr, cm^{-1}): 3239, 1631; ^{1}H NMR (300 MHz, $CDCl_3$): 1.27-1.63 (*m*, 15H, CH_2, $H_{Cyclohexyl}$), 1.67-2.25 (*m*, 15H, CH_2, $H_{Cyclohexyl}$), 4.96 (*s*, 1H, NH), 5.11 (*m*, 1H, CNH); ^{13}C NMR (75 MHz, CDCl3): 21.6, 24.9, 25.0, 25.1, 25.1, 25.4, 25.7, 25.8, 25.9, 27.0, 33.2, 34.4, 35.2, 57.6, 59.0, 153.4, 157.8; HR-MS (ESI): calc. for $C_{19}H_{33}N_6[M+1]^+$ 345.2762, found 345.2761; calc. for $C_{19}H_{33}N_6Na[M+Na]^+$ 367.2581, found 367.2581; calc. for $C_{19}H_{33}KN_6[M+1]^+$ 383.23207, found 383.23200.

Acknowledgment:

We gratefully acknowledge Iran National Science Foundation (INSF) for their financial support.

ASSOCIATED CONTENT

Supporting Information

Copies of ^{1}H NMR, ^{13}C NMR, HRMS spectra for compounds **5a-h**; and crude ^{1}H NMR spectra for the mixture of diastereomers in compounds **5a**, **5d**, **5e** and **5f**; X-ray crystal data for compounds **5a**, and **5d**. This material is available free of charge via the Internet at http://pubs.acs.org.

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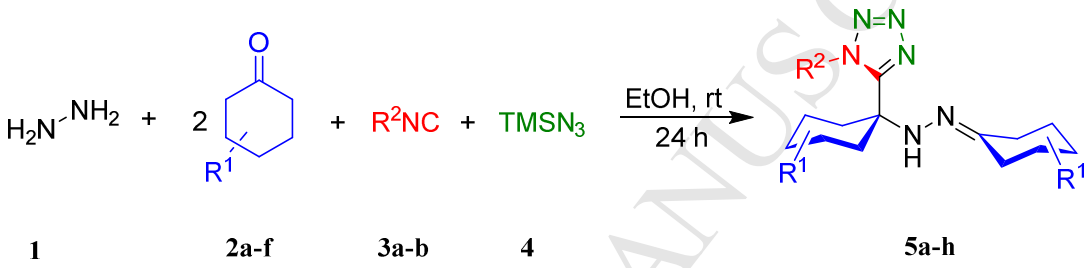
Graphical Abstract

Efficient and Stereoselective Synthesis of α-Hydrazino Tetrazoles through a Pseudo Five-component Domino Reaction

Ali Nikbakht, Sorour Ramezanpour, Saeed Balalaie,* Frank Rominger



1 **2a-f** **3a-b** **4** **5a-h**

Supporting information

Efficient and Stereoselective Synthesis of α-Hydrazino Tetrazoles through a Pseudo Five-component Domino Reaction

Ali Nikbakht, [a] Sorour Ramezanpour, [a] Saeed Balalaie,* [a,b] Frank Rominger [c]

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[b]Medical Biology Research Center, Kermanshah University of Medical Sciences, Kermanshah, Iran

[c]Organisch-Chemisches Institut der Universitaet Heidelberg, Im Neuenheimer Feld 270, 69120 Heidelberg, Germany

General information

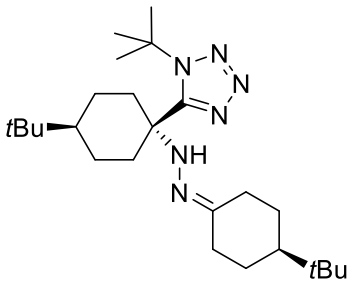
Experimental Section:

Commercially available materials were used without further purification. Melting points were determined on an Electrothermal 9100 apparatus and were uncorrected. IR spectra were obtained on an ABB FT-IR FTLA 2000 spectrometer. ^{1}H NMR and ^{13}C NMR spectra were run on Bruker DRX-300 AVANCE spectrometers at 300 MHz for ^{1}H NMR, 75 MHz for ^{13}C NMR. DMSO-d_6 and $CDCl_3$ was used as solvent. High resolution mass spectra were recorded on Mass-ESI-POS (Apex Qe-FT- ICR instrument) spectrometer.

General procedure for the synthesis of α-hydrazino tetrazoles 5a-h:

To a solution of hydrazine hydrate **1** (1 mmol), cyclic ketone **2a-f** (2 mmol) in 5 mL ethanol was added. The mixture was stirred for 30 min then isocyanide **3a-b** (1.2 mmol) and trimethylsilyl azide **4** (1 mmol) was added to the mixture. The mixture was stirred for 24h at ambient temperature. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 1:3), the precipitate was filtered off and then washed with methanol.

Spectroscopic data for compounds **5a-h:**

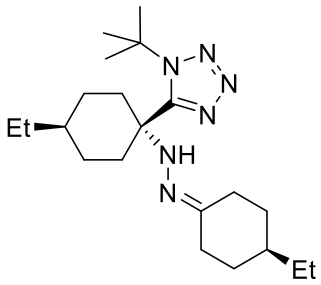


5a (89%)

1-(*tert*-Butyl)-5- ((1r,4S)-4- (*tert*-butyl)-1-(2 -((S,*E*)-4 -(*tert*-butyl)cyclohexylidene) hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5a**): Colorless powder; 383 mg (89%), mp: 209-211°C, IR υ_{max} (KBr, cm^{-1}): 3258, 1639; ^{1}H NMR (300 MHz, $CDCl_3$): 0.82 (*s*, 9H, $3CH_3$, *t*-Bu), 0.86 (*s*, 9H, $3CH_3$, *t*-Bu), 1.07-

1.72 (*m*, 8H, $4CH_2$, $H_{Cyclohexyl}$), 1.78 (*s*, 9H, $3CH_3$, *t*-Bu), 1.82-2.01 (*m*, 4H, $H_{Cyclohexyl}$), 2.28-2.50 (*m*, 2H, $H_{Cyclohexyl}$), 2.60-2.81 (*m*, 4H, $H_{Cyclohexyl}$), 4.36 (*s*, 1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): 23.9, 24.2, 25.1, 26.3, 27.4, 27.5, 27.7, 31.2, 32.3, 32.4, 34.8, 37.9, 38.5, 47.4, 47.6, 59.3, 64.4, 152.6, 157.8; HR-MS (ESI): calc. for $C_{25}H_{47}N_6$ $[M+H]^+$ 431.3857, found 431.3857; calc. for $C_{25}H_{46}N_6Na$ $[M+Na]^+$ 453.3677, found 453.3676; calc. for $C_{25}H_{46}N_6K$ $[M+K]^+$ 469.3416, found 469.3415.

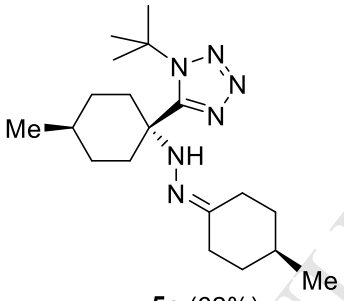
colourless crystal (plate), dimensions 0.370 x 0.230 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.2048(7) Å, b=29.336(3) Å, c=14.4809(17) Å, alpha=90 deg, beta=98.484(4) deg, gamma=90 deg, V=2607.0(5) $Å^3$, rho=1.097 g/cm^3, T=200(2) K, $Theta_{max}$= 26.022 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.15and a completeness of 100.0% to a resolution of 0.81Å, 16371 reflections measured, 5139 unique (R(int)=0.0805), 3027 observed (I > 2σ(I)), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using SADABS based on the Laue symmetry of the reciprocal space, mu=0.07mm^{-1}, T_{min}=0.90, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package, 285 parameters refined, hydrogen atoms were treated using appropriate riding models, except H21 at N21, which was refined isotropically, goodness of fit 0.99 for observed reflections, final residual values R1(F)=0.057, $wR(F^2)$=0.109 for observed reflections, residual electron density -0.22 to 0.21 $eÅ^{-3}$. CCDC 103930 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



5b (54%)

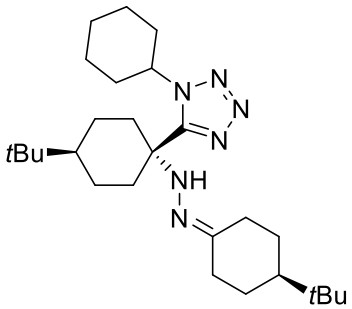
1-(*tert*-Butyl)-5-((1*r*,4S)-4-ethyl-1-(2- ((S,*E*)-4-ethyl cyclohexylidene) hydrazinyl) cyclohexyl) -1*H*-tetrazole (**5b**): Waxy liquid; 202 mg (54%), IR υ_{max} (KBr, cm^{-1}): 3362, 1713; ^{1}H NMR (300 MHz, $CDCl_3$): 0.85 (*t*, *J* = 7.0 Hz, 3H, CH_3), 0.91 (*t*, *J* = 7.0 Hz, 3H, CH_3), 0.94-1.03

(*m*, 2H, $H_{Cyclohexyl}$), 1.22-1.37 (*m*, 7H, $H_{Cyclohexyl}$), 1.61-1.88 (*m*, 8H, $H_{Cyclohexyl}$), 1.78 (*s*, 9H, $3CH_3$, *t*-Bu), 2.00-2.23(*m*, 2H, $H_{Cyclohexyl}$), 2.53-2.67 (*m*, 3H, $H_{Cyclohexyl}$), 4.54 (*brs*, 1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): 11.4, 11.6, 11.7, 24.4, 28.1, 28.2, 28.9, 31.2, 31.3, 31.5, 32.8, 34.4, 35.6, 35.9, 37.5, 38.6, 59.4, 64.3, 152.7, 158.4; HR-MS (ESI): calc. for $C_{21}H_{39}N_6$ $[M+H]^+$ 375.3360, found 375.3357; calc. for $C_{21}H_{38}N_6Na$ $[M+Na]^+$ 397.3038, found 397.3035; calc. for $C_{21}H_{38}N_6K$ $[M+K]^+$ 413.2787, found 413.2785.



5c (62%)

1-(*tert*-Butyl)-5-((1*r*,4S)- 4-methyl-1- (2 -((S, *E*)-4-methylcyclohexylidene) hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5c**): Colorless solid; 215 mg (62%), mp: 120-123 ^{o}C, IR υ_{max} (KBr, cm^{-1}): 3255, 1632; ^{1}H NMR (300 MHz, $CDCl_3$): 0.89 (*d*, 6H, *J* = 6.3 Hz, $2CH_3$), 0.92-1.70 (*m*, 10H, $H_{cyclohexyl}$), 1.74 (*s*, 9H, *t*-Bu), 1.76-2.66 (*m*, 8H, $H_{cyclohexyl}$), 4.62 (*s*, 1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): 20.9, 21.6, 24.3, 30.3, 30.5, 30.6, 31.0, 31.2, 32.0, 33.8, 34.5, 35.2, 35.6, 59.1, 64.3, 76.6, 152.2, 158.5; HR-MS (ESI): HR-MS (ESI): calc. for $C_{19}H_{35}N_6$ $[M+H]^+$ 347.2428, found 347.2428.

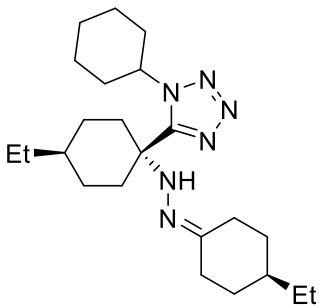


5d (92%)

5-((1r,4S)-4-(*tert*-Butyl)-1-(2-((S,*E*)-4-(*tert*-Butyl)cyclohexylidene)hydrazinyl) cyclohexyl)-1-cyclohexyl-1*H*-tetrazole (**5d**): Colorless solid; 420 mg (92%), mp: 239-241 ^{o}C, IR υ_{max} (KBr, cm^{-1}): 3253, 1642; ^{1}H NMR (300 MHz, $CDCl_3$): 0.77 (*s*, 9H, $3CH_3$, *t*-Bu), 0.84 (*s*, 9H, $3CH_3$, *t*-Bu), 0.88-1.50 (*m*, 10H, $H_{Cyclohexyl}$), 1.53-1.82 (*m*, 6H, $H_{Cyclohexyl}$), 1.83-2.24 (*m*, 9H, $H_{Cyclohexyl}$),

2.28-2.79 (*m*, 3H, $H_{Cyclohexyl}$), 4.49 (*s*, 1H, NH), 4.90-5.11 (*m*, 1H, CNH); ^{13}C NMR (75 MHz, $CDCl_3$): 21.6, 22.0, 24.8, 25.0, 25.7, 26.5, 27.3, 27.7, 32.3, 32.5, 33.1, 33.2, 34.2, 34.8, 46.4, 47.4, 56.8, 58.9, 153.4, 158.5; HR-MS (ESI): calc. for $C_{27}H_{49}N_6$ $[M+H]^+$ 457.40144, found 457.40132; calc. for $C_{27}H_{49}N_6Na$ $[M+Na]^+$ 479.3834, found 479.3833; calc. for $C_{27}H_{48}KN_6$ $[M+K]^+$ 495.3573, found 495.3572.

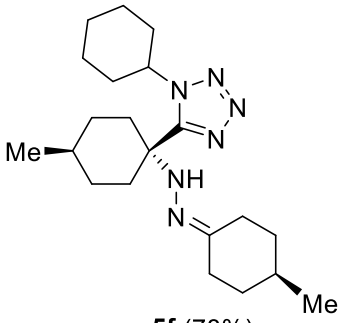
colourless crystal (needle), dimensions 0.420 x 0.050 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.345(2) Å, b=31.355(11) Å, c=14.286(5) Å, alpha=90 deg, beta=99.872(11) deg, gamma=90 deg, V=2799.8(17) $Å^3$, rho=1.083 g/cm^3, T=200(2) K, $Theta_{max}$= 21.260 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.63and a completeness of 61.5% to a resolution of 0.98Å, 11459 reflections measured, 3120 unique (R(int)=0.1049), 1898 observed ($I > 2\sigma(I)$), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using SADABS[1] based on the Laue symmetry of the reciprocal space, mu=0.06mm^{-1}, T_{min}=0.82, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package[2], 302 parameters refined, hydrogen atoms were treated using appropriate riding models, except H10 at N10, which was refined isotropically, goodness of fit 1.01 for observed reflections, final residual values R1(F)=0.074, $wR(F^2)$=0.159 for observed reflections, residual electron density -0.33 to 0.32 $eÅ^{-3}$. CCDC 103931 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



5e (71%)

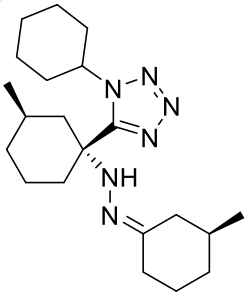
1-Cyclohexyl-5- ((1*r*,4S)-4-ethyl-1 -(2 -((S,*E*)-4-ethylcyclohexylidene)hydrazinyl) cyclohexyl)-1H-tetrazole (**5e**): Colorless solid; 216 mg (71%), mp: 129-130 ^oC, IR υ_{max} (KBr, cm^{-1}): 3244, 1739, 1635; ^{1}H NMR (300 MHz, $CDCl_3$): 0.84 (*t*, 3H, *J* = 7.2 Hz, CH_3), 0.87 (*t*, 3H, *J* = 7.2 Hz, CH_3), 0.97-1.02 (*m*, 3H, $H_{Cyclohexyl}$), 1.78-1.38 (*m*, 10H, $H_{Cyclohexyl}$), 1.63-2.24 (*m*, 16H, $H_{Cyclohexyl}$), 2.53-2.70 (*m*, 3H, $H_{Cyclohexyl}$), 4.59 (*s*,1H, NH), 5.07-5.14 (*m*, 1H, CNH); ^{13}C NMR

(75 MHz, $CDCl_3$): 11.5, 11.6, 24.3, 25.0, 25.6, 28.5, 28.6, 28.8, 31.3, 32.6, 33.2, 34.4, 35.3, 35.7, 38.5, 58.3, 59.1, 153.6.2; HR-MS (ESI): calc. for $C_{23}H_{41}N_6$ $[M+H]^+$ 401.3388, found 401.3387; calc. for $C_{23}H_{40}N_6$ $[M+Na]^+$ 423.3197, found 432.3193; calc. for $C_{23}H_{40}KN_6$ $[M+K]^+$ 439.2947, found 439.2946.



5f (78%)

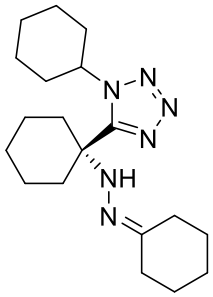
1-Cyclohexyl-5 -((1*r*,4S)-4-methyl-1-(2 -((S, *E*)-4-methylcyclohexylidene) hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5f**) Colorless solid; 291 mg (78%), mp: 167-169 ^oC, IR υ_{max} (KBr, cm^{-1}): 3240, 1739, 1631; ^1H NMR (300 MHz, $CDCl_3$): 0.85 (*d*, 3H, *J* = 6.4 Hz, CH_3), 0.88 (*d*, 3H, *J* = 6.5 Hz, CH_3), 0.93-1.56 (*m*, 10H, $H_{Cyclohexyl}$), 1.58-1.78 (*m*, 8H, $H_{Cyclohexyl}$), 1.79-2.20 (*m*, 8H, $H_{Cyclohexyl}$), 2.45-2.66 (*m*, 2H, $H_{Cyclohexyl}$), 4.6 (*s*, 1H, NH), 5.10 (*m*, 1H, NCH); ^{13}C NMR (75 MHz, $CDCl_3$): 21.4, 21.6, 24.3, 25.0, 25.6, 30.8, 30.9, 31.7, 31.8, 33.1, 33.2, 33.7, 34.5, 35.1, 35.3, 35.8, 56.9, 58.0, 59.1, 153.3, 156.2, HR-MS (ESI): calc. for $C_{21}H_{37}N_6$ $[M+H]^+$ 373.3074, found 373.3074; calc. for $C_{21}H_{36}N_6Na$ $[M+Na]^+$ 395.2894, found 395.2894; calc. for $C_{21}H_{36}KN_6$ $[M+K]^+$ 411.2633, found 411.2633.



5g (65%)

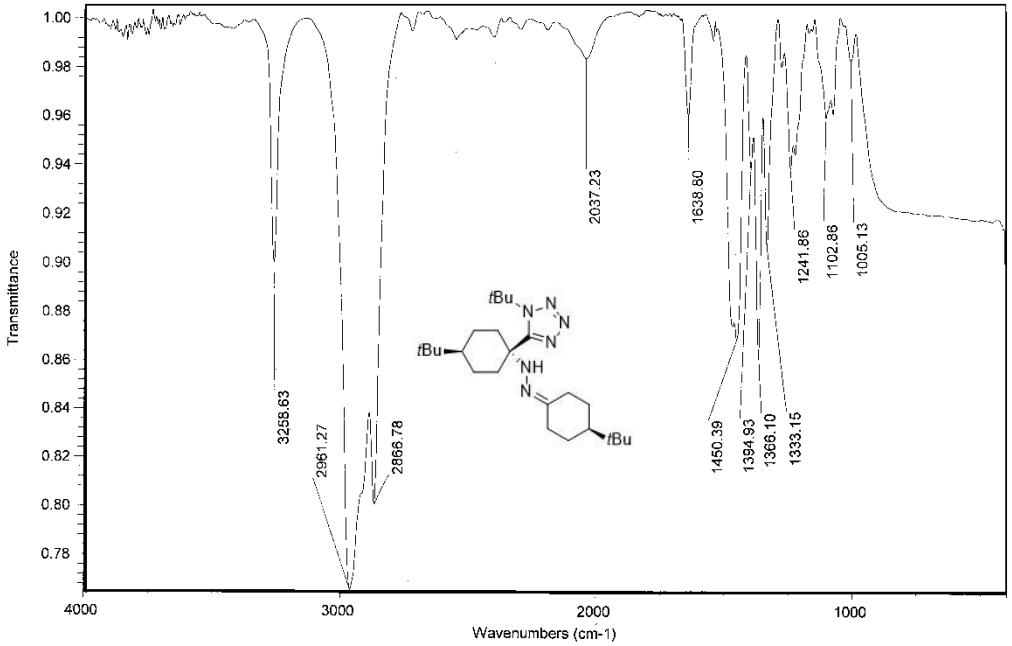
1-Cyclohexyl-5- ((1R, 3R)-3-methyl-1- (2-((S, *Z*)-3-methylcyclohexylidene)hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5g**): Colorless solid; 231 mg (65%), mp: 145-147 ^oC, IR υ_{max} (KBr, cm^{-1}): 3238, 1740, 1632; ^1H NMR (300 MHz, $CDCl_3$) 0.85-0.95 (*m*, 6H, CH_3), 1.00-1.55 (*m*, 10H, $H_{Cyclohexyl}$), 1.63-1.79 (*m*, 6H, $H_{Cyclohexyl}$), 1.87-2.21 (*m*, 9H, $H_{Cyclohexyl}$), 2.48-2.65 (*m*, 3H, $H_{Cyclohexyl}$), 4.56 (*s*, 1H, NH), 5.05-5.13 (*m*, 1H, CHN); ^{13}C NMR (75 MHz, $CDCl_3$): 22.2, 22.4,

22.5, 25.0, 25.6, 25.7, 29.0, 32.3, 33.2, 33.3, 34.1, 34.4, 34.7, 35.9, 44.4, 58.9, 59.1, 59.2, 153.3, 156.1; 156.1; HR-MS (ESI): calc. for $C_{21}H_{37}N_6$ $[M+H]^+$ 373.3074, found 373.3074; calc. for $C_{21}H_{36}N_6Na$ $[M+Na]^+$ 395.2894, found 395.2894; calc. for $C_{21}H_{36}KN_6$ $[M+K]^+$ 411.2633, found 411.2633.

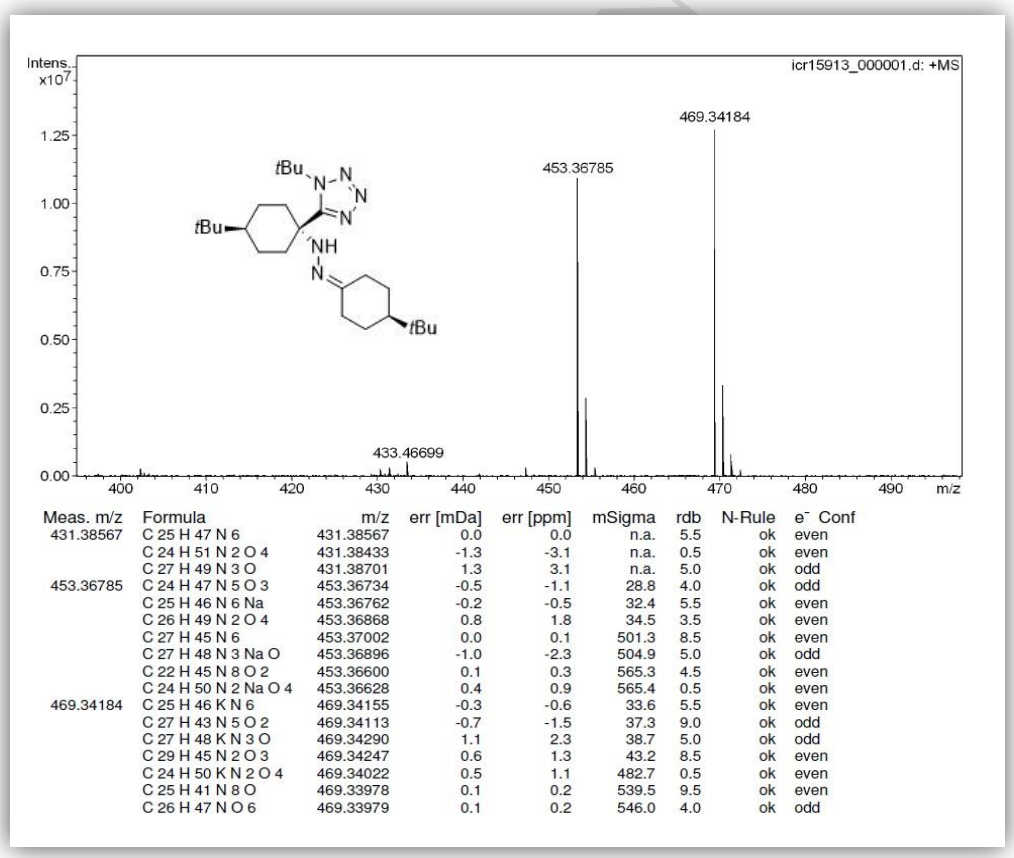


5h (75%)

1-Cyclohexyl-5-(1-(2-cyclohexylidenehydrazinyl)cyclohexyl)-1H-tetrazole(**5h**): Colorless powder; 258 mg (75%), mp: 147-150 ^oC, IR υ_{max} (KBr, cm^{-1}): 3239, 1631; ^1H NMR (300 MHz, $CDCl_3$): 1.27-1.63 (*m*, 15H, CH_2, $H_{Cyclohexyl}$), 1.67-2.25 (*m*, 15H, CH_2, $H_{Cyclohexyl}$), 4.96 (*s*, 1H, NH), 5.11 (*m*, 1H, CNH); ^{13}C NMR (75 MHz, CDCl3): 21.6, 24.9, 25.0, 25.1, 25.1, 25.4, 25.7, 25.8, 25.9, 27.0, 33.2, 34.4, 35.2, 57.6, 59.0, 153.4, 157.8; HR-MS (ESI): calc. for $C_{19}H_{33}N_6[M+1]^+$ 345.2762, found 345.2761; calc. for $C_{19}H_{33}N_6Na[M+Na]^+$ 367.2581, found 367.2581; calc. for $C_{19}H_{33}KN_6[M+1]^+$ 383.23207, found 383.23200.

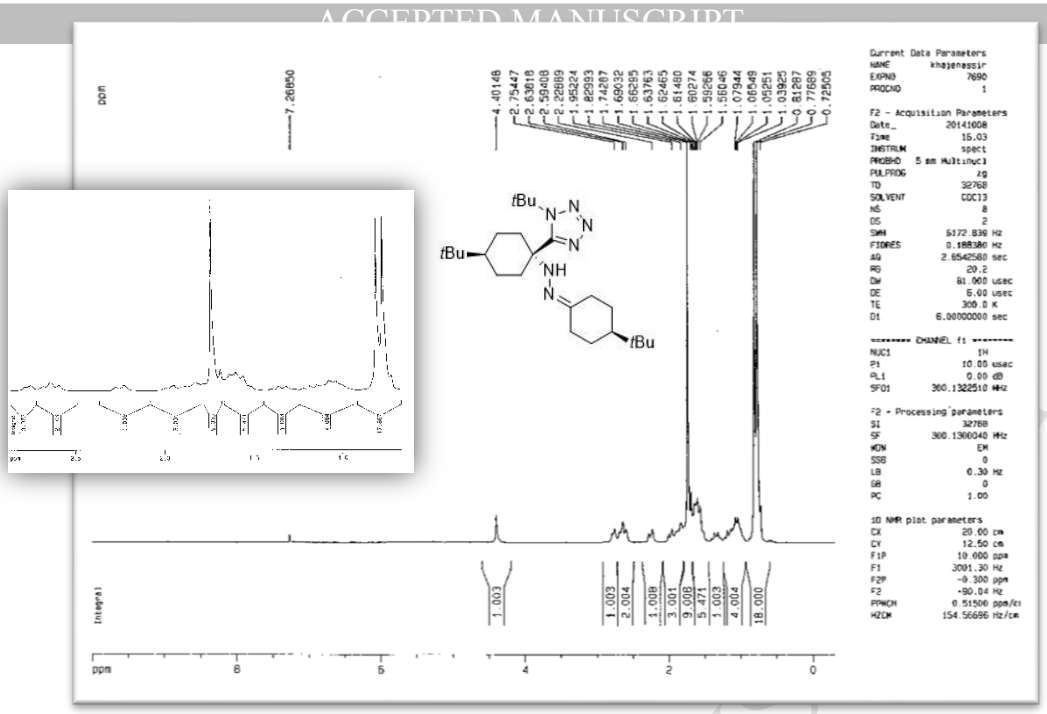


IR (KBr) **(5a)**

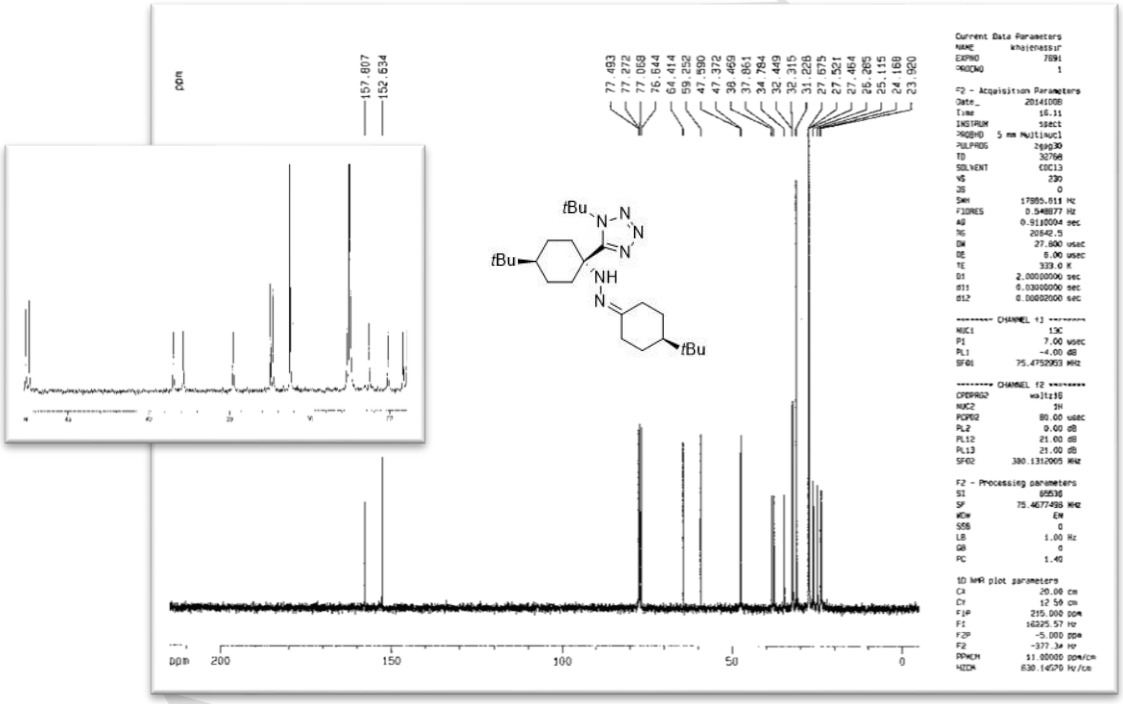


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
431.38567	C 25 H 47 N 6	431.38567	0.0	0.0	n.a.	5.5	ok	even
	C 24 H 51 N 2 O 4	431.38433	-1.3	-3.1	n.a.	0.5	ok	even
	C 27 H 49 N 3 O	431.38701	1.3	3.1	n.a.	5.0	ok	odd
453.36785	C 24 H 47 N 5 O 3	453.36734	-0.5	-1.1	28.8	4.0	ok	odd
	C 25 H 46 N 6 Na	453.36762	-0.2	-0.5	32.4	5.5	ok	even
	C 26 H 49 N 2 O 4	453.36868	0.8	1.8	34.5	3.5	ok	even
	C 27 H 45 N 6	453.37002	0.0	0.1	501.3	8.5	ok	even
	C 27 H 48 N 3 Na O	453.36896	-1.0	-2.3	504.9	5.0	ok	odd
	C 22 H 45 N 8 O 2	453.36600	0.1	0.3	565.3	4.5	ok	even
	C 24 H 50 N 2 Na O 4	453.36628	0.4	0.9	565.4	0.5	ok	even
469.34184	C 25 H 46 K N 6	469.34155	-0.3	-0.6	33.6	5.5	ok	even
	C 27 H 43 N 5 O 2	469.34113	-0.7	-1.5	37.3	9.0	ok	odd
	C 27 H 48 K N 3 O	469.34290	1.1	2.3	38.7	5.0	ok	odd
	C 29 H 45 N 2 O 3	469.34247	0.6	1.3	43.2	8.5	ok	even
	C 24 H 50 K N 2 O 4	469.34022	0.5	1.1	482.7	0.5	ok	even
	C 25 H 41 N 8 O	469.33978	0.1	0.2	539.5	9.5	ok	even
	C 26 H 47 N O 6	469.33979	0.1	0.2	546.0	4.0	ok	odd

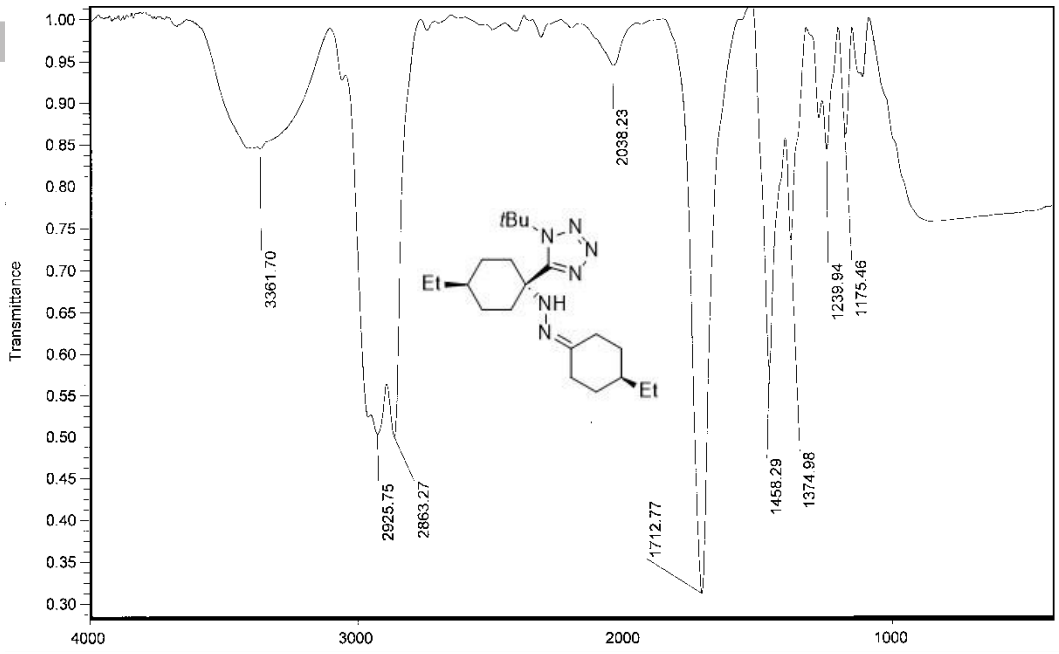
ESI-HRMS **(5a)**



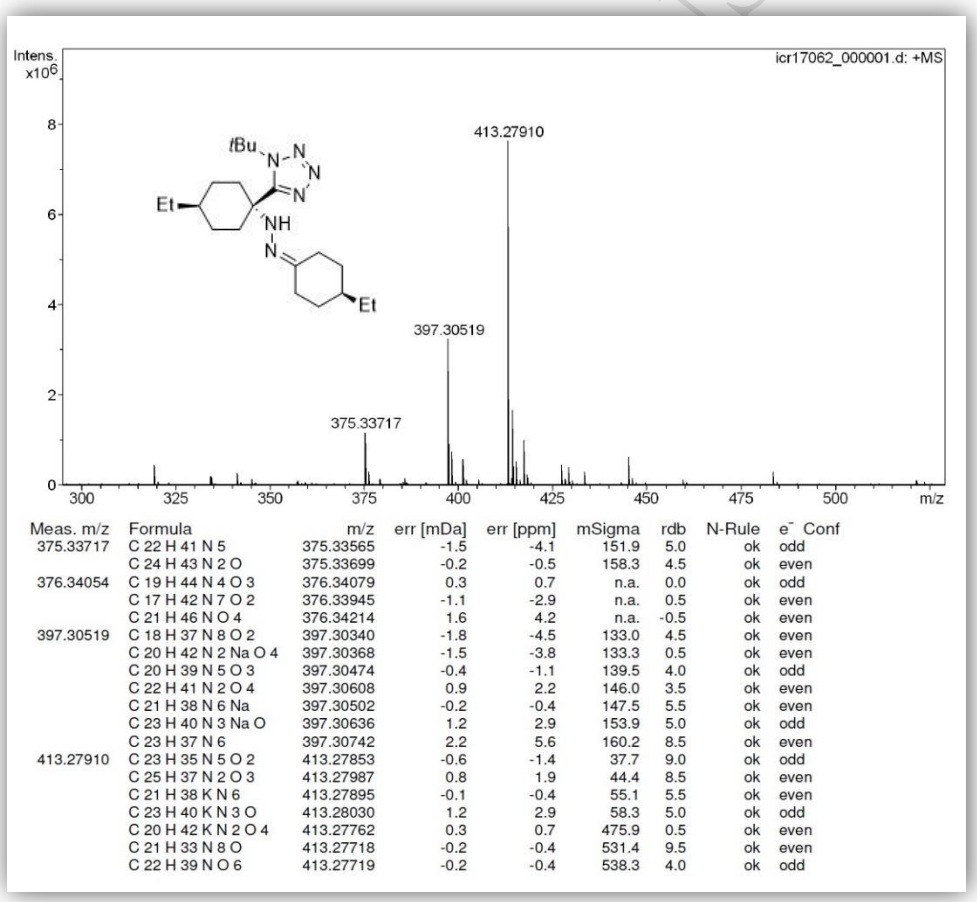
^{1}HNMR (300MHz, $CDCl_3$) **(5a)**



^{13}CNMR (75MHz, $CDCl_3$) **(5a)**

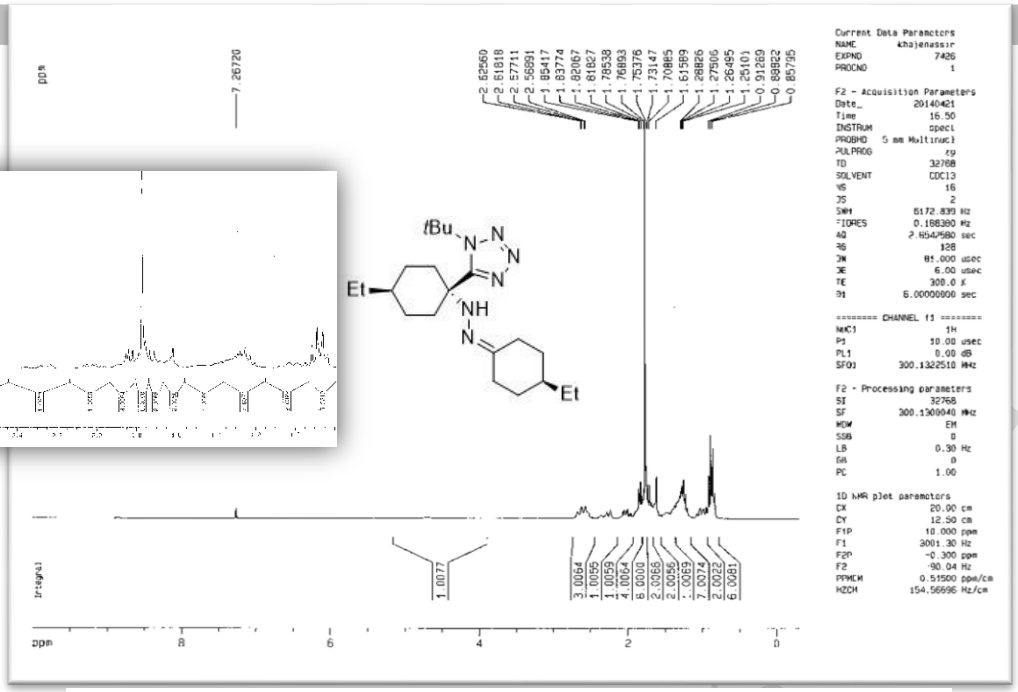


IR (KBr) **(5b)**

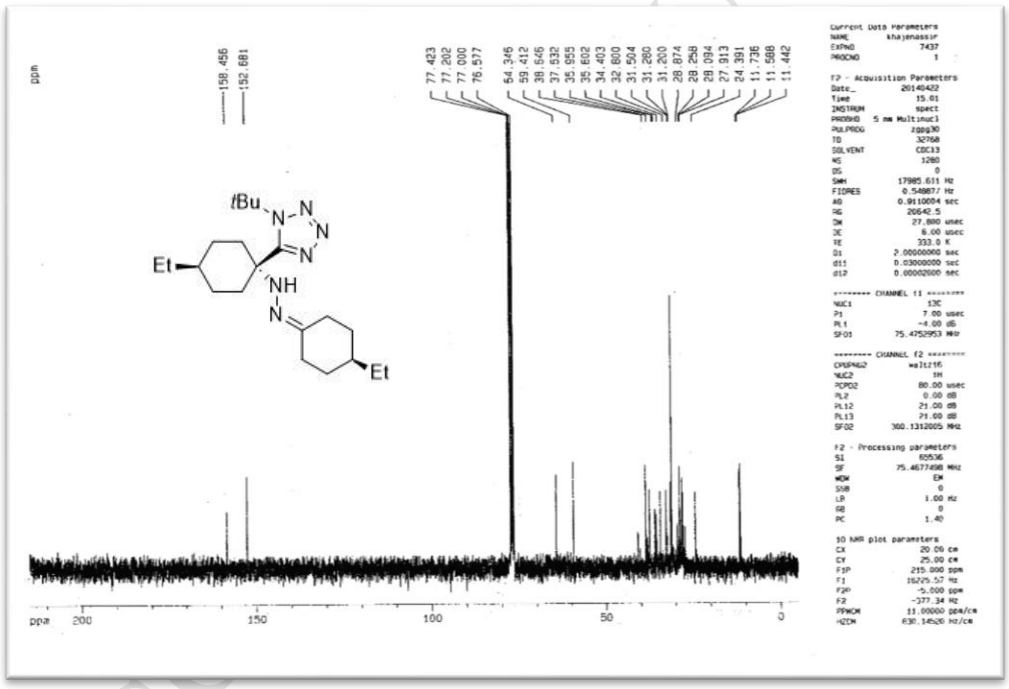


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
375.33717	C 22 H 41 N 5	375.33565	-1.5	-4.1	151.9	5.0	ok	odd
	C 24 H 43 N 2 O	375.33699	-0.2	-0.5	158.3	4.5	ok	even
376.34054	C 19 H 44 N 4 O 3	376.34079	0.3	0.7	n.a.	0.0	ok	odd
	C 17 H 42 N 7 O 2	376.33945	-1.1	-2.9	n.a.	0.5	ok	even
	C 21 H 46 N O 4	376.34214	1.6	4.2	n.a.	-0.5	ok	even
397.30519	C 18 H 37 N 8 O 2	397.30340	-1.8	-4.5	133.0	4.5	ok	even
	C 20 H 42 N 2 Na O 4	397.30368	-1.5	-3.8	133.3	0.5	ok	even
	C 20 H 39 N 5 O 3	397.30474	-0.4	-1.1	139.5	4.0	ok	odd
	C 22 H 41 N 2 O 4	397.30608	0.9	2.2	146.0	3.5	ok	even
	C 21 H 38 N 6 Na	397.30502	-0.2	-0.4	147.5	5.5	ok	even
	C 23 H 40 N 3 Na O	397.30636	1.2	2.9	153.9	5.0	ok	odd
	C 23 H 37 N 6	397.30742	2.2	5.6	160.2	8.5	ok	even
413.27910	C 23 H 35 N 5 O 2	413.27853	-0.6	-1.4	37.7	9.0	ok	odd
	C 25 H 37 N 2 O 3	413.27987	0.8	1.9	44.4	8.5	ok	even
	C 21 H 38 K N 6	413.27895	-0.1	-0.4	55.1	5.5	ok	even
	C 23 H 40 K N 3 O	413.28030	1.2	2.9	58.3	5.0	ok	odd
	C 20 H 42 K N 2 O 4	413.27762	0.3	0.7	475.9	0.5	ok	even
	C 21 H 33 N 8 O	413.27718	-0.2	-0.4	531.4	9.5	ok	even
	C 22 H 39 N O 6	413.27719	-0.2	-0.4	538.3	4.0	ok	odd

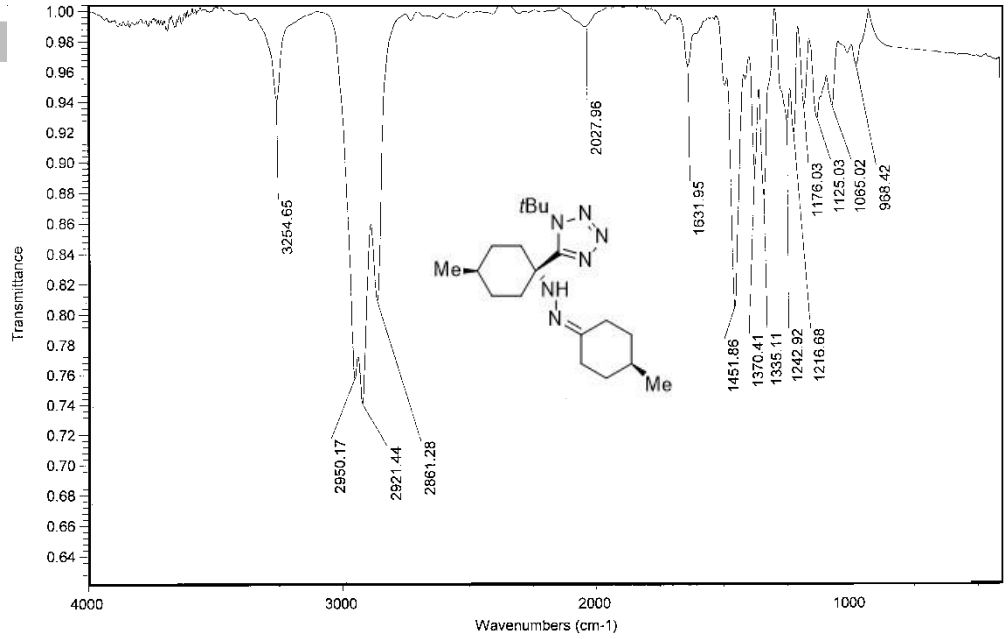
ESI-HRMS **(5b)**



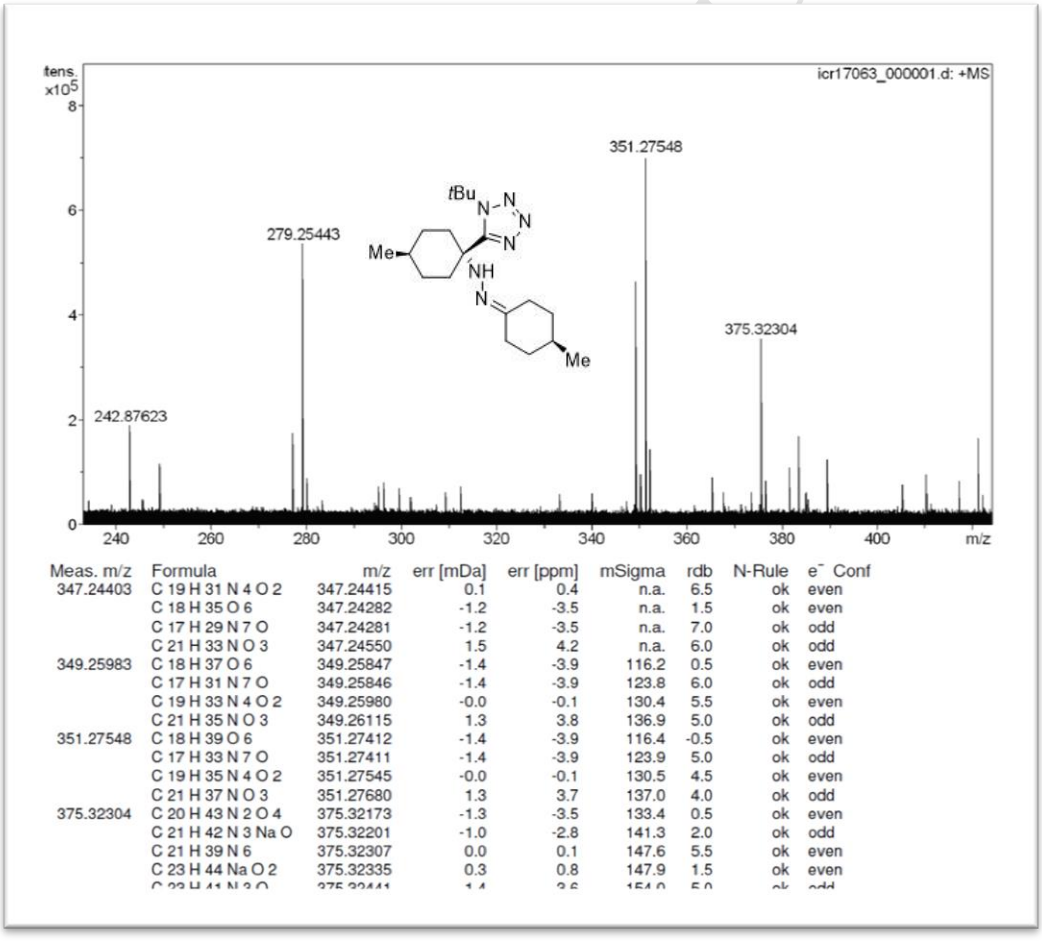
^{1}HNMR (300MHz, $CDCl_3$) **(5b)**



^{13}CNMR (75MHz, $CDCl_3$) **(5b)**

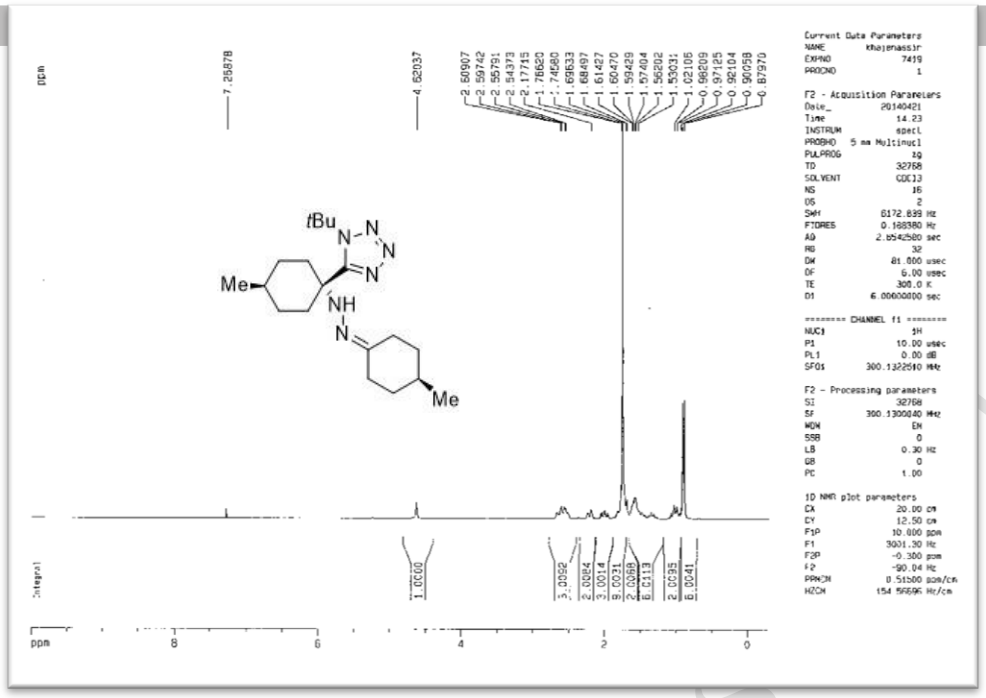


IR (KBr) **(5c)**

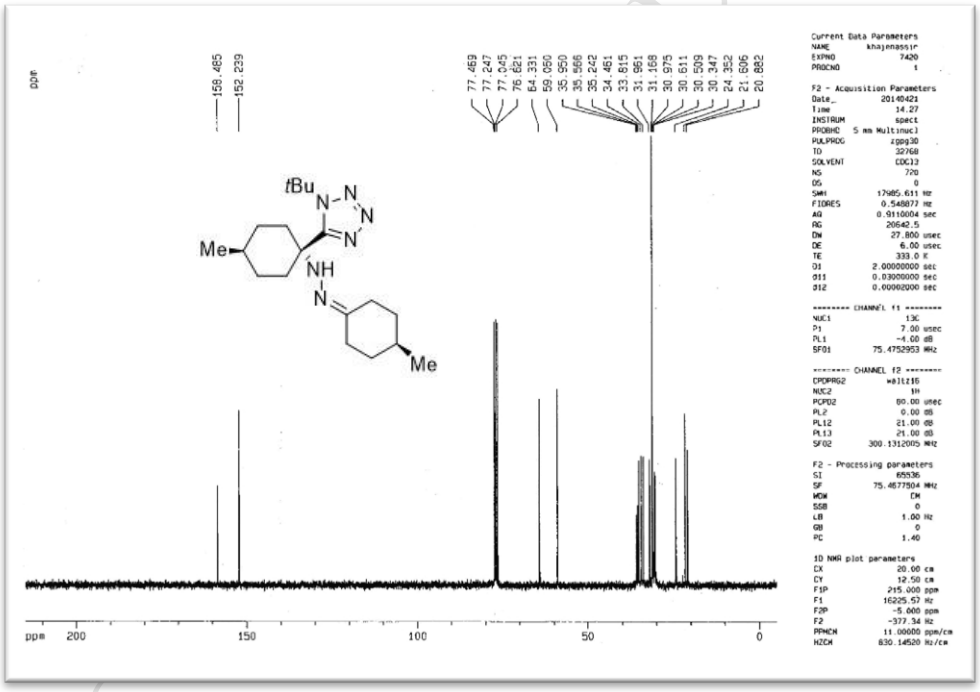


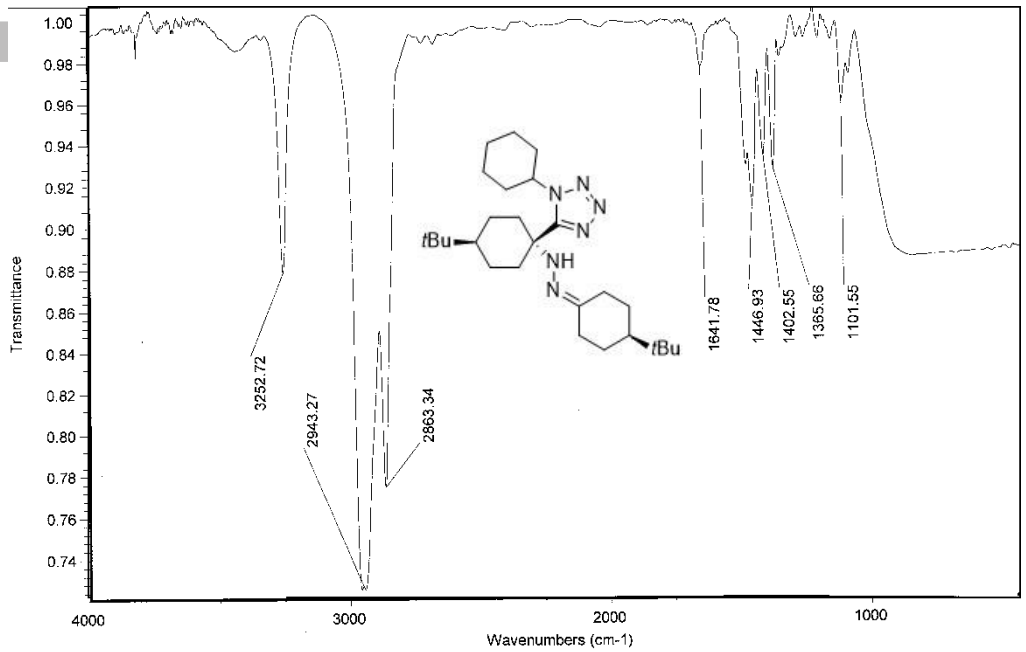
Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
347.24403	C 19 H 31 N 4 O 2	347.24415	0.1	0.4	n.a.	6.5	ok	even
	C 18 H 35 O 6	347.24282	-1.2	-3.5	n.a.	1.5	ok	even
	C 17 H 29 N 7 O	347.24281	-1.2	-3.5	n.a.	7.0	ok	odd
	C 21 H 33 N O 3	347.24550	1.5	4.2	n.a.	6.0	ok	odd
349.25983	C 18 H 37 O 6	349.25847	-1.4	-3.9	116.2	0.5	ok	even
	C 17 H 31 N 7 O	349.25846	-1.4	-3.9	123.8	6.0	ok	odd
	C 19 H 33 N 4 O 2	349.25980	-0.0	-0.1	130.4	5.5	ok	even
	C 21 H 35 N O 3	349.26115	1.3	3.8	136.9	5.0	ok	odd
351.27548	C 18 H 39 O 6	351.27412	-1.4	-3.9	116.4	-0.5	ok	even
	C 17 H 33 N 7 O	351.27411	-1.4	-3.9	123.9	5.0	ok	odd
	C 19 H 35 N 4 O 2	351.27545	-0.0	-0.1	130.5	4.5	ok	even
	C 21 H 37 N O 3	351.27680	1.3	3.7	137.0	4.0	ok	odd
375.32304	C 20 H 43 N 2 O 4	375.32173	-1.3	-3.5	133.4	0.5	ok	even
	C 21 H 42 N 3 Na O	375.32201	-1.0	-2.8	141.3	2.0	ok	odd
	C 21 H 39 N 6	375.32307	0.0	0.1	147.6	5.5	ok	even
	C 23 H 44 Na O 2	375.32335	0.3	0.8	147.9	1.5	ok	even

ESI-HRMS **(5c)**

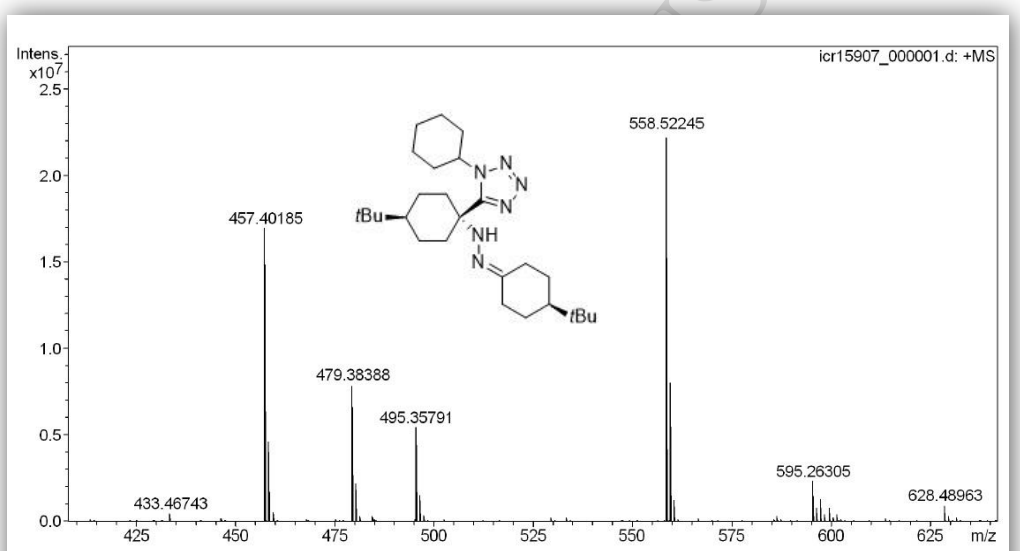


^{1}HNMR (300MHz, $CDCl_3$) **(5c)**



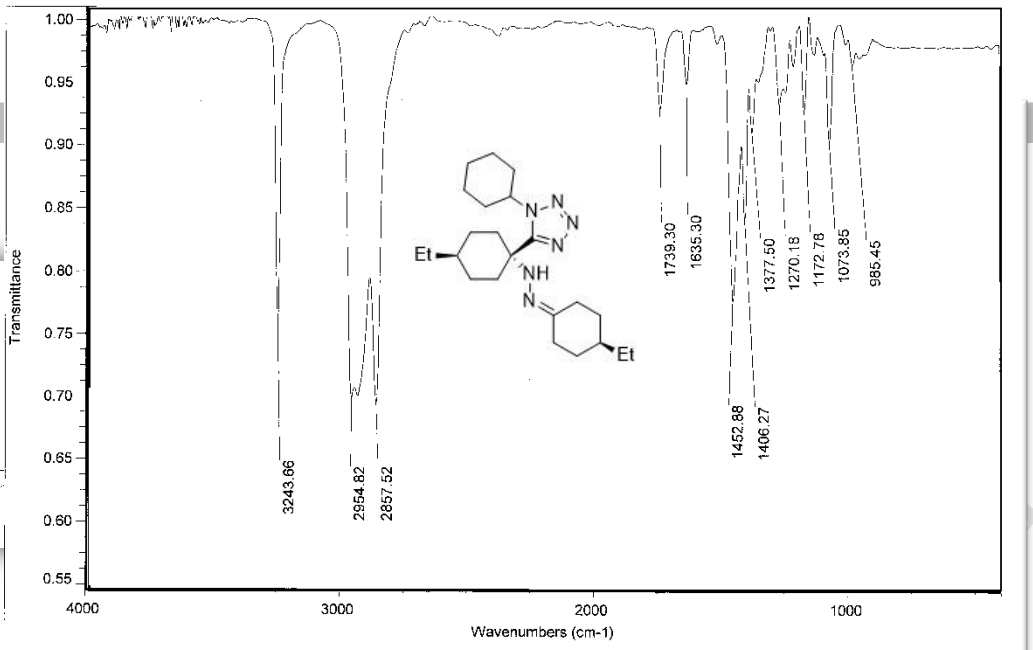


IR (KBr) **(5d)**

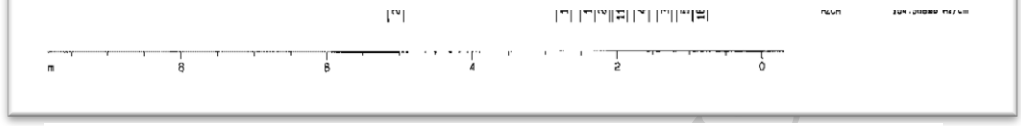


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
457.40185	C 27 H 49 N 6	457.40132	-0.5	-1.2	23.9	6.5	ok	even
	C 29 H 51 N 3 O	457.40266	0.8	1.8	29.6	6.0	ok	odd
	C 24 H 51 N 5 O 3	457.39864	0.2	0.5	166.0	2.0	ok	odd
	C 31 H 53 O 2	457.40401	-0.1	-0.2	492.4	5.5	ok	even
	C 26 H 53 N 2 O 4	457.39998	0.1	0.3	547.5	1.5	ok	even
479.38388	C 27 H 48 N 6 Na	479.38327	-0.6	-1.3	32.4	6.5	ok	even
	C 26 H 49 N 5 O 3	479.38299	-0.9	-1.9	33.5	5.0	ok	odd
	C 28 H 51 N 2 O 4	479.38433	0.5	0.9	34.3	4.5	ok	even
	C 29 H 50 N 3 Na O	479.38461	0.7	1.5	37.4	6.0	ok	odd
	C 23 H 51 N 4 O 6	479.38031	-1.2	-2.5	157.9	0.5	ok	even
	C 22 H 45 N 11 O	479.38031	-1.2	-2.6	165.3	6.0	ok	odd
	C 24 H 50 N 5 Na O 3	479.38059	-0.9	-2.0	165.9	2.0	ok	odd
	C 31 H 49 N 3 O	479.38701	0.8	1.7	477.3	9.0	ok	odd
	C 29 H 47 N 6	479.38567	-0.5	-1.1	480.8	9.5	ok	even
	C 31 H 52 Na O 2	479.38595	-0.2	-0.5	481.0	5.5	ok	even
	C 25 H 53 N O 7	479.38165	0.1	0.3	483.9	0.0	ok	odd
	C 24 H 47 N 8 O 2	479.38165	0.1	0.2	552.4	5.5	ok	even
	C 26 H 52 N 2 Na O 4	479.38193	0.4	0.8	552.5	1.5	ok	even
495.35791	C 18 H 45 N 11 O 5	495.35997	2.1	4.1	28.3	2.0	ok	odd

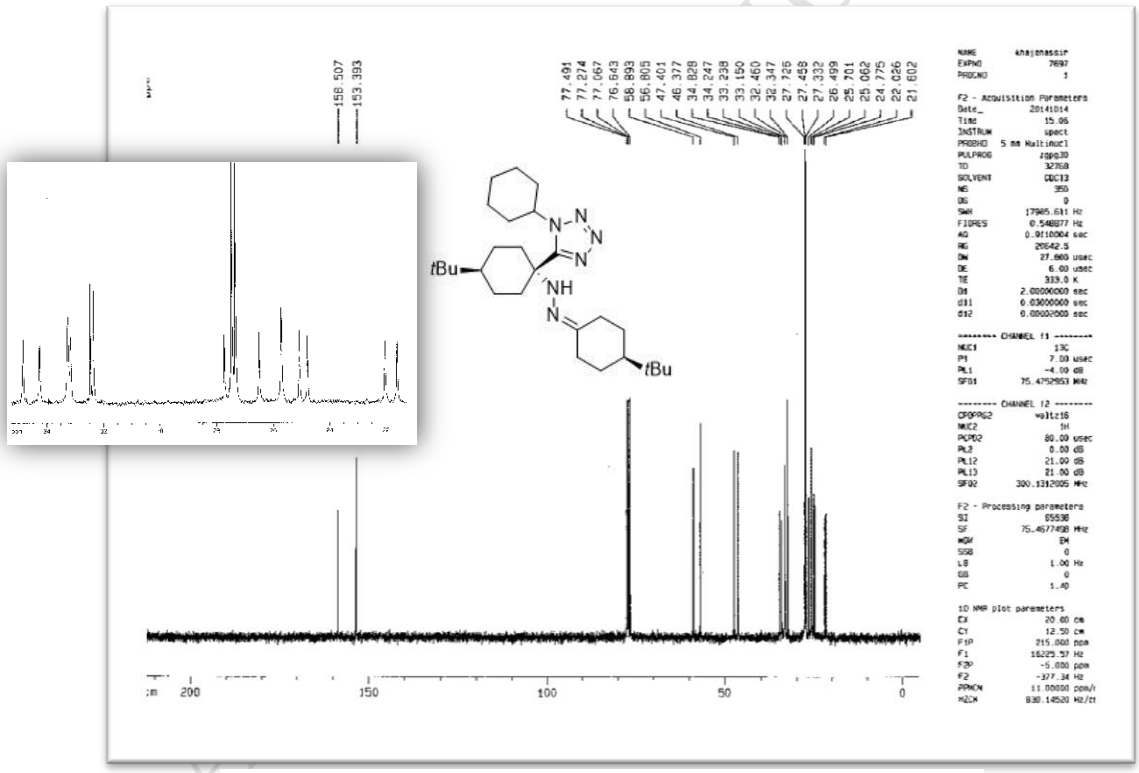
ESI-HRMS **(5d)**



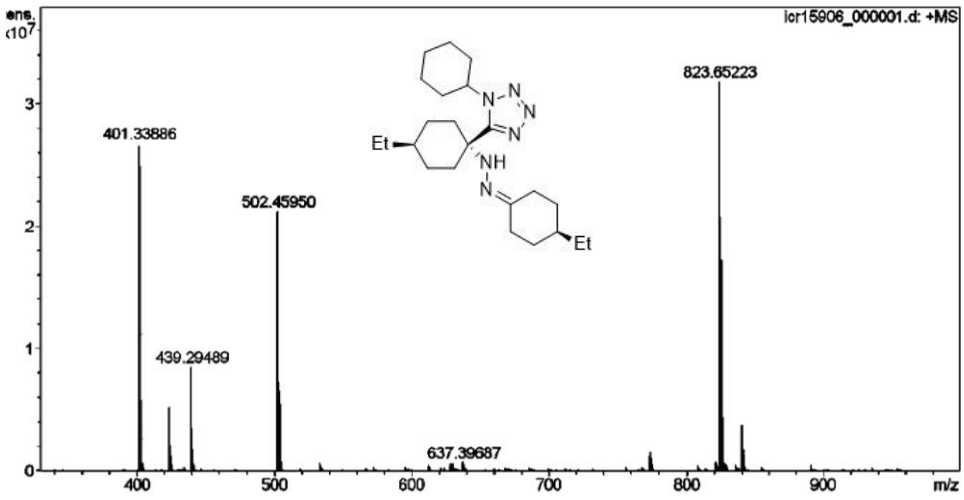
IR (KBr) **(5e)**



^{1}HNMR (300MHz, $CDCl_3$) **(5d)**

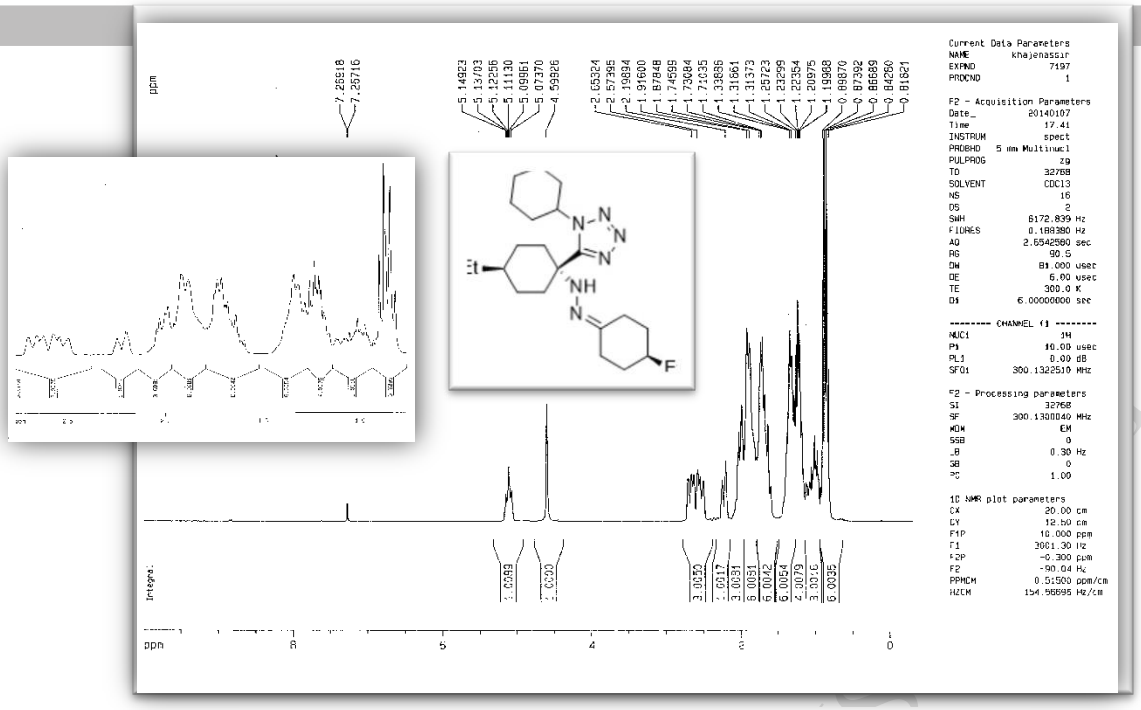


^{13}CNMR (75MHz, $CDCl_3$) **(5d)**

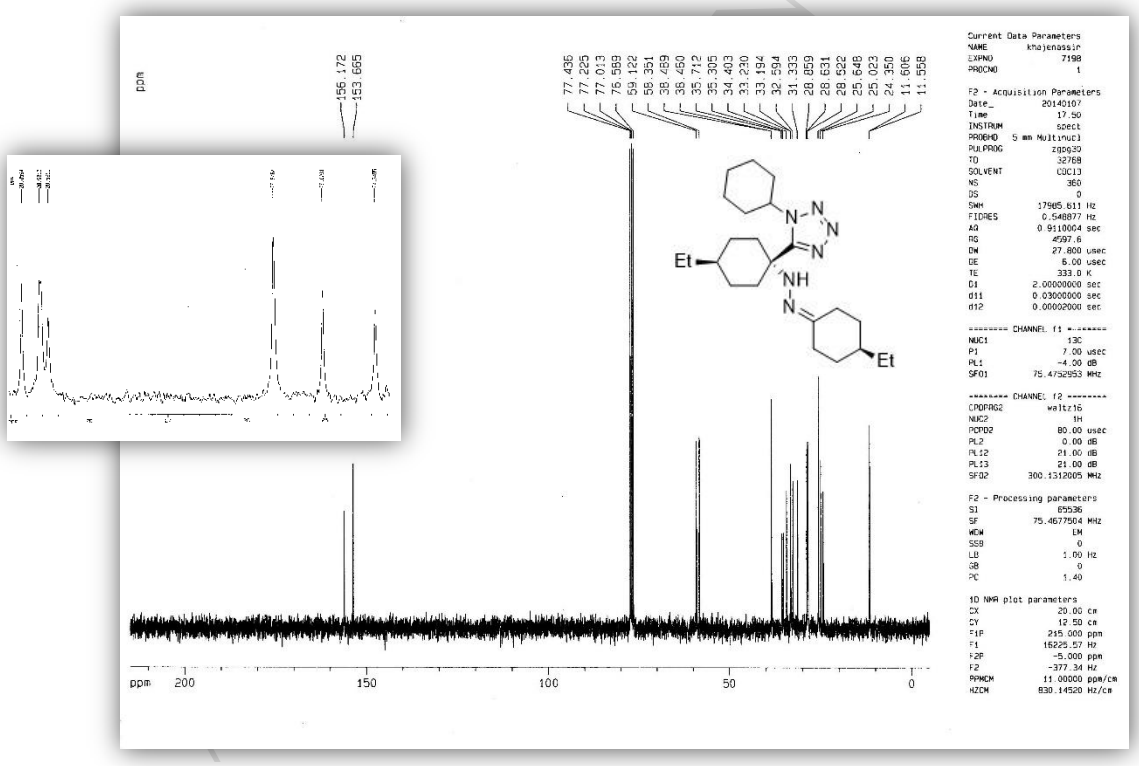


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
401.33886	C 20 H 43 N 5 O 3	401.33604	-0.2	-0.5	139.8	2.0	ok	odd
	C 27 H 45 O 2	401.34141	0.0	0.0	611.6	5.5	ok	even
	C 23 H 41 N 6	401.33872	-0.1	-0.4	648.8	6.5	ok	even
	C 22 H 45 N 2 O 4	401.33738	1.1	2.8	658.3	1.5	ok	even
	C 25 H 43 N 3 O	401.34006	0.0	0.0	709.6	6.0	ok	odd
423.32095	C 20 H 39 N 8 O 2	423.31905	-1.9	-4.5	20.4	5.5	ok	even
	C 22 H 44 N 2 Na O 4	423.31933	-1.6	-3.8	22.9	1.5	ok	even
	C 21 H 45 N O 7	423.31905	-1.9	-4.5	23.8	0.0	ok	odd
	C 22 H 41 N 5 O 3	423.32039	-0.6	-1.3	25.5	5.0	ok	odd
	C 23 H 40 N 6 Na	423.32067	-0.3	-0.7	29.4	6.5	ok	even
	C 24 H 43 N 2 O 4	423.32173	0.8	1.8	31.3	4.5	ok	even
	C 25 H 42 N 3 Na O	423.32201	1.1	2.5	35.1	6.0	ok	odd
	C 27 H 44 Na O 2	423.32335	2.4	5.7	37.0	5.5	ok	even
	C 25 H 39 N 6	423.32307	2.1	5.0	41.0	9.5	ok	even
	C 20 H 42 N 5 Na O 3	423.31799	-3.0	-7.0	139.7	2.0	ok	odd

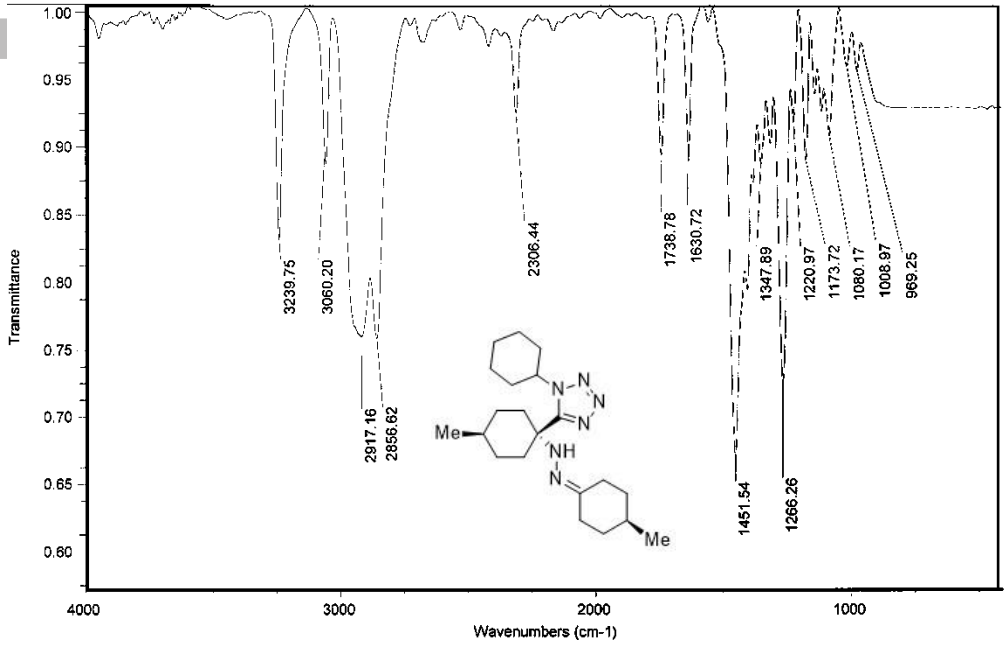
ESI-HRMS **(5e)**



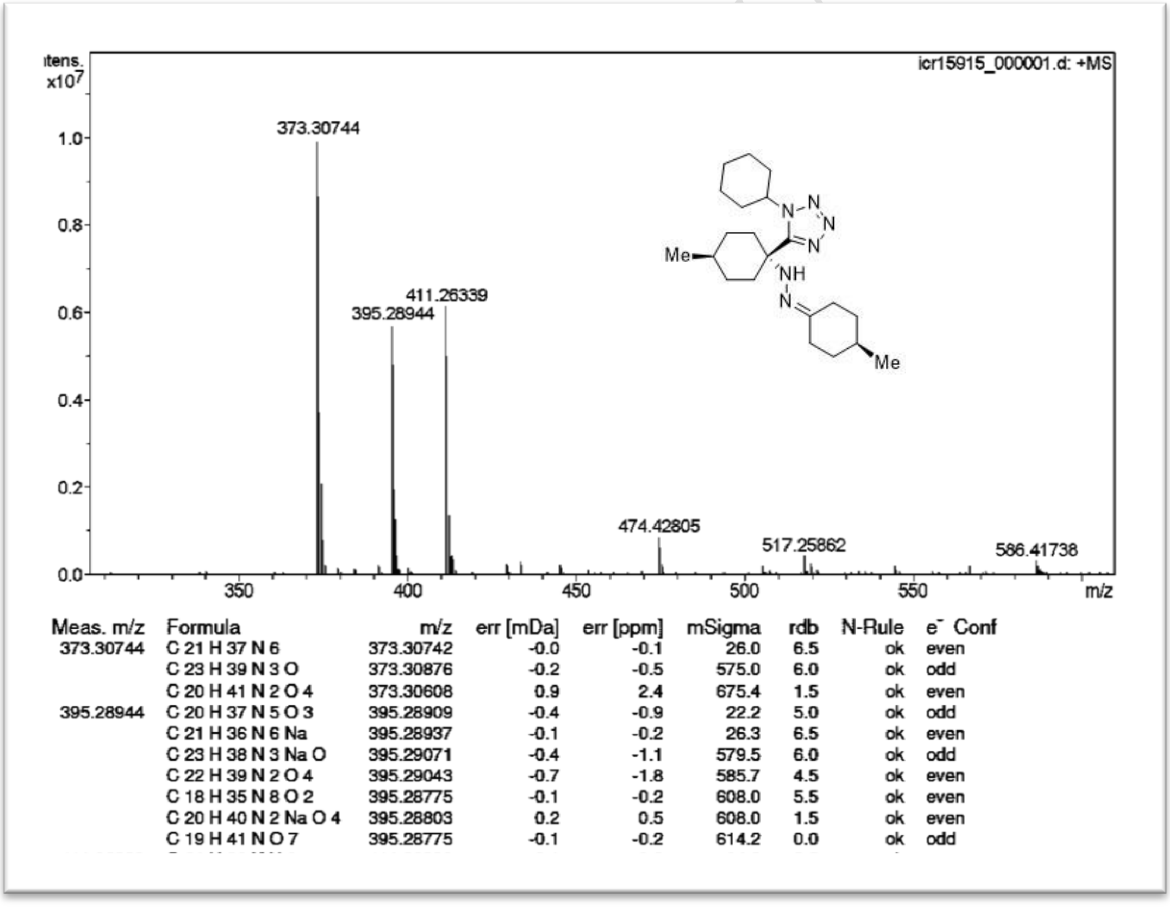
^{1}HNMR (300MHz, $CDCl_3$) **(5e)**



^{13}CNMR (75MHz, $CDCl_3$) **(5e)**

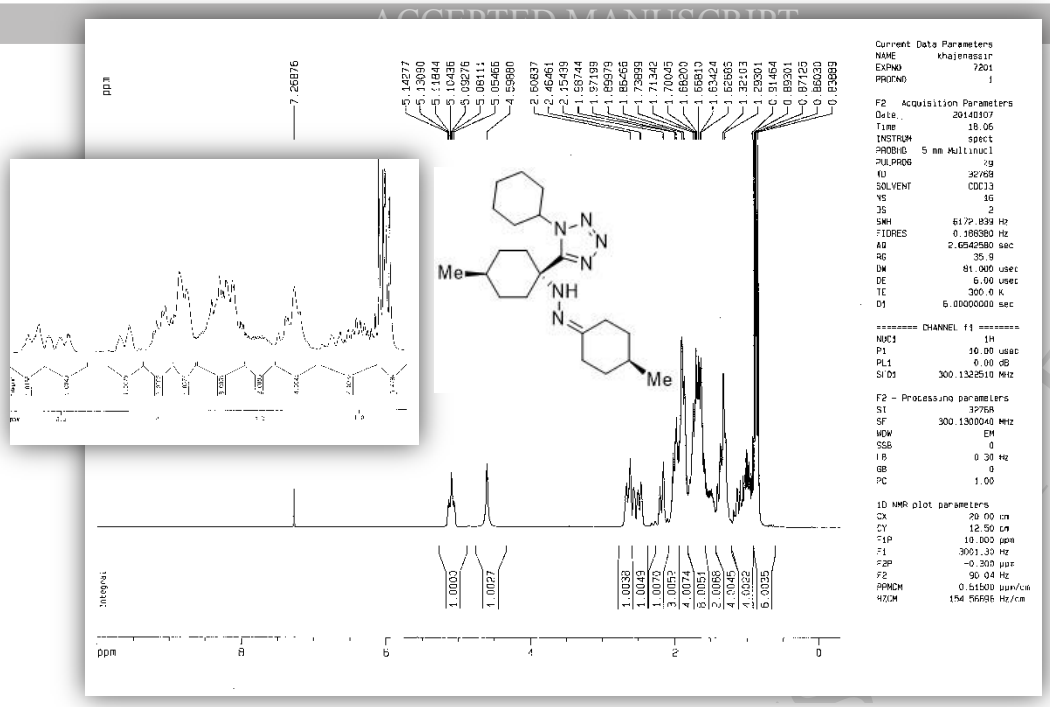


IR (KBr) **(5f)**

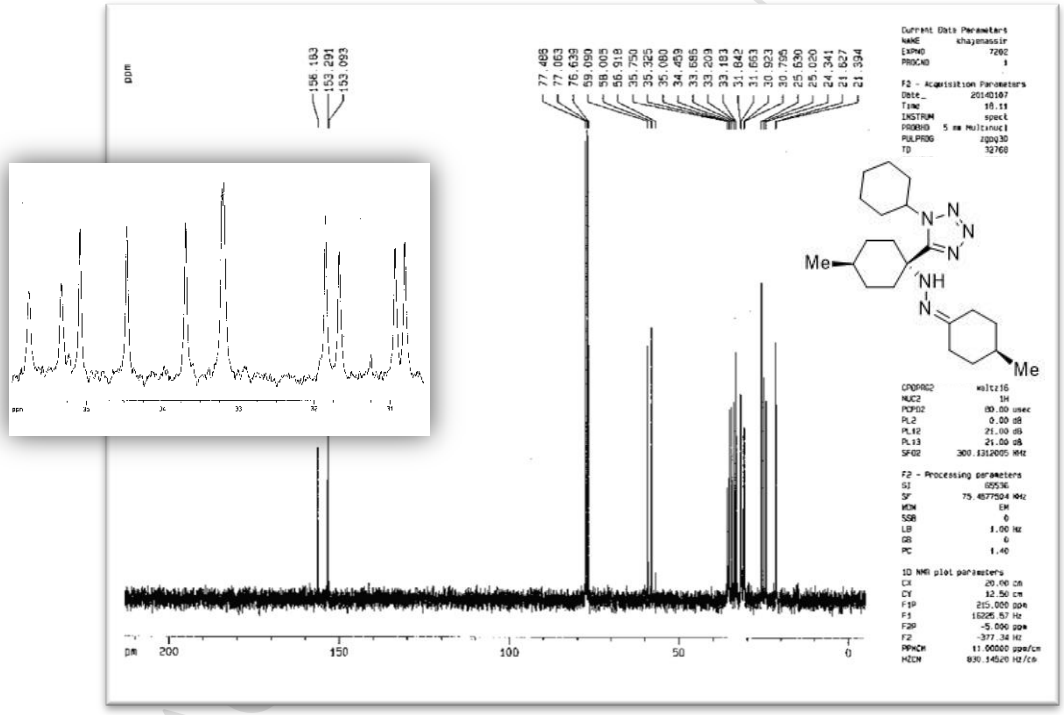


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
373.30744	C 21 H 37 N 6	373.30742	-0.0	-0.1	26.0	6.5	ok	even
	C 23 H 39 N 3 O	373.30876	-0.2	-0.5	575.0	6.0	ok	odd
	C 20 H 41 N 2 O 4	373.30608	0.9	2.4	675.4	1.5	ok	even
395.28944	C 20 H 37 N 5 O 3	395.28909	-0.4	-0.9	22.2	5.0	ok	odd
	C 21 H 36 N 6 Na	395.28937	-0.1	-0.2	26.3	6.5	ok	even
	C 23 H 38 N 3 Na O	395.29071	-0.4	-1.1	579.5	6.0	ok	odd
	C 22 H 39 N 2 O 4	395.29043	-0.7	-1.8	585.7	4.5	ok	even
	C 18 H 35 N 8 O 2	395.28775	-0.1	-0.2	608.0	5.5	ok	even
	C 20 H 40 N 2 Na O 4	395.28803	0.2	0.5	608.0	1.5	ok	even
	C 19 H 41 N O 7	395.28775	-0.1	-0.2	614.2	0.0	ok	odd

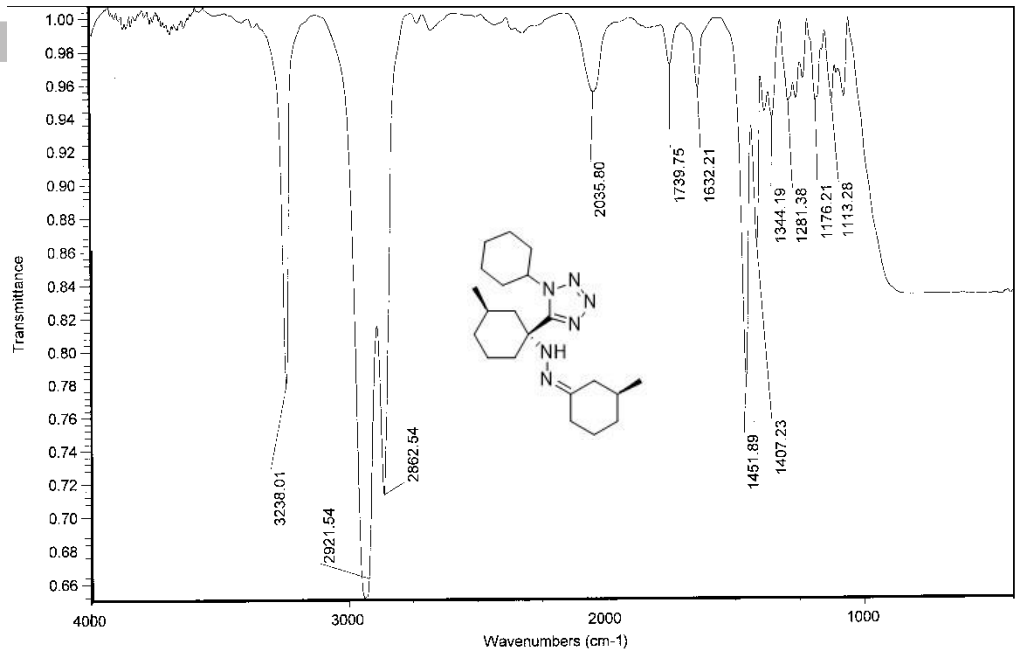
ESI-HRMS **(5f)**



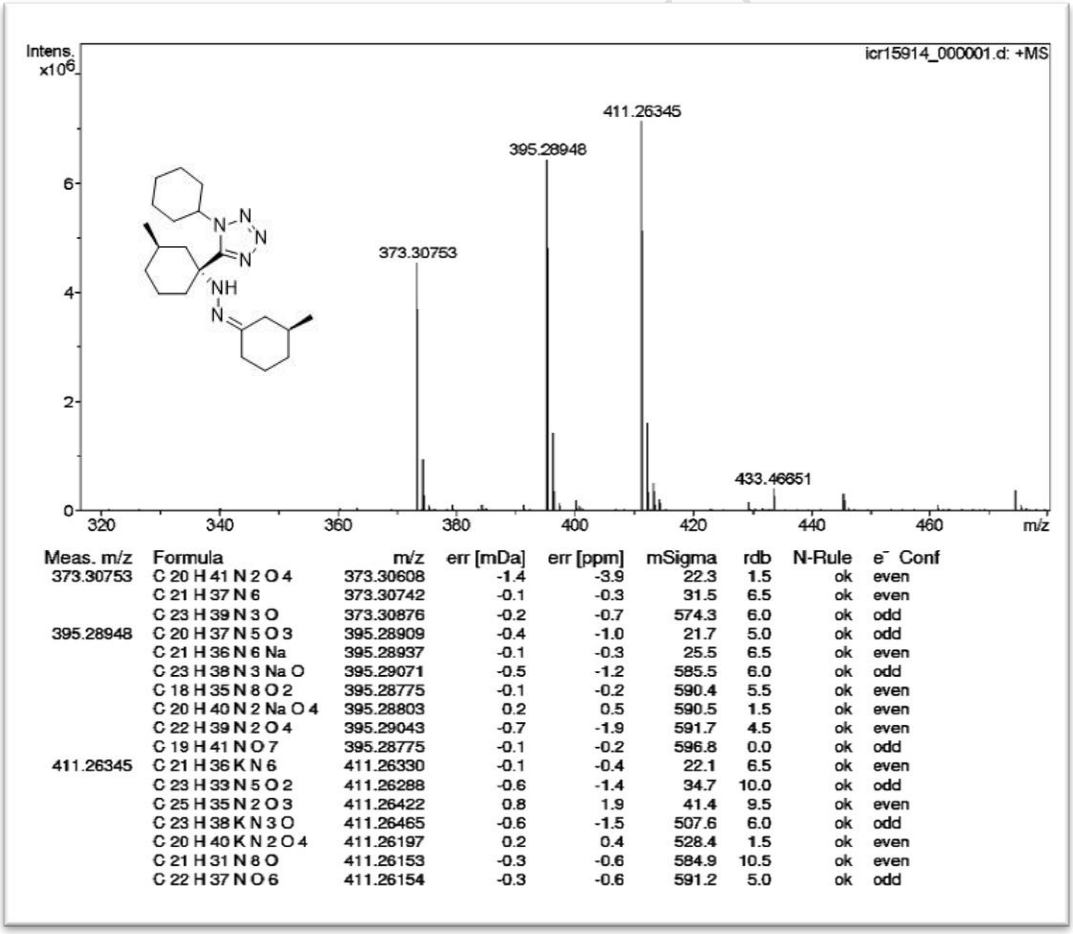
^{1}HNMR (300MHz, $CDCl_3$) **(5f)**



^{13}CNMR (75MHz, $CDCl_3$) **(5f)**

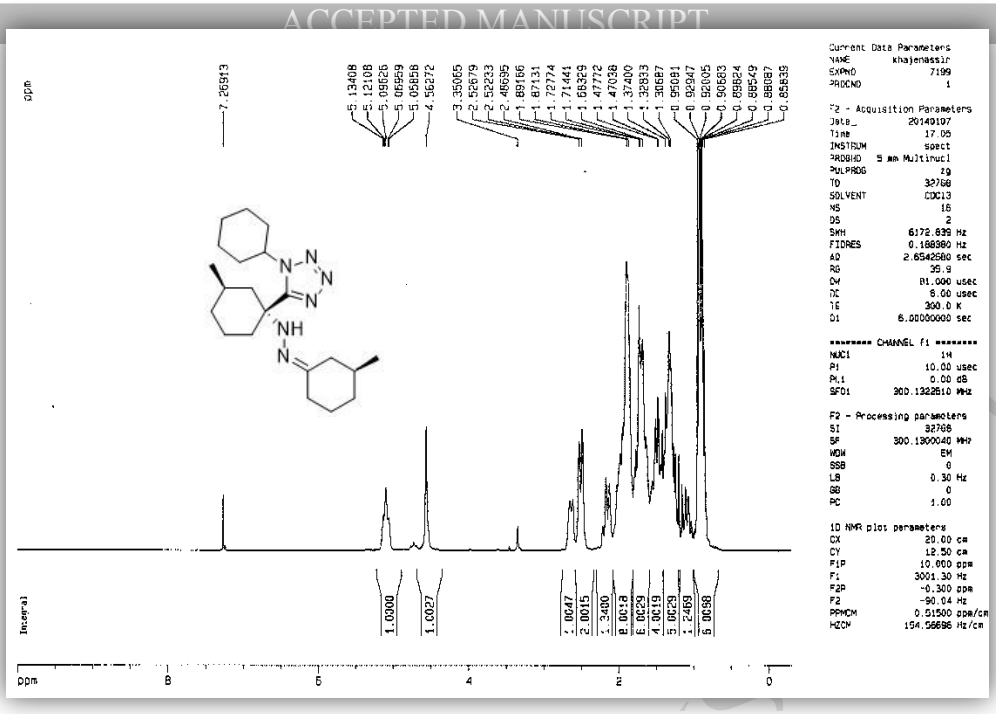


IR (KBr) **(5g)**

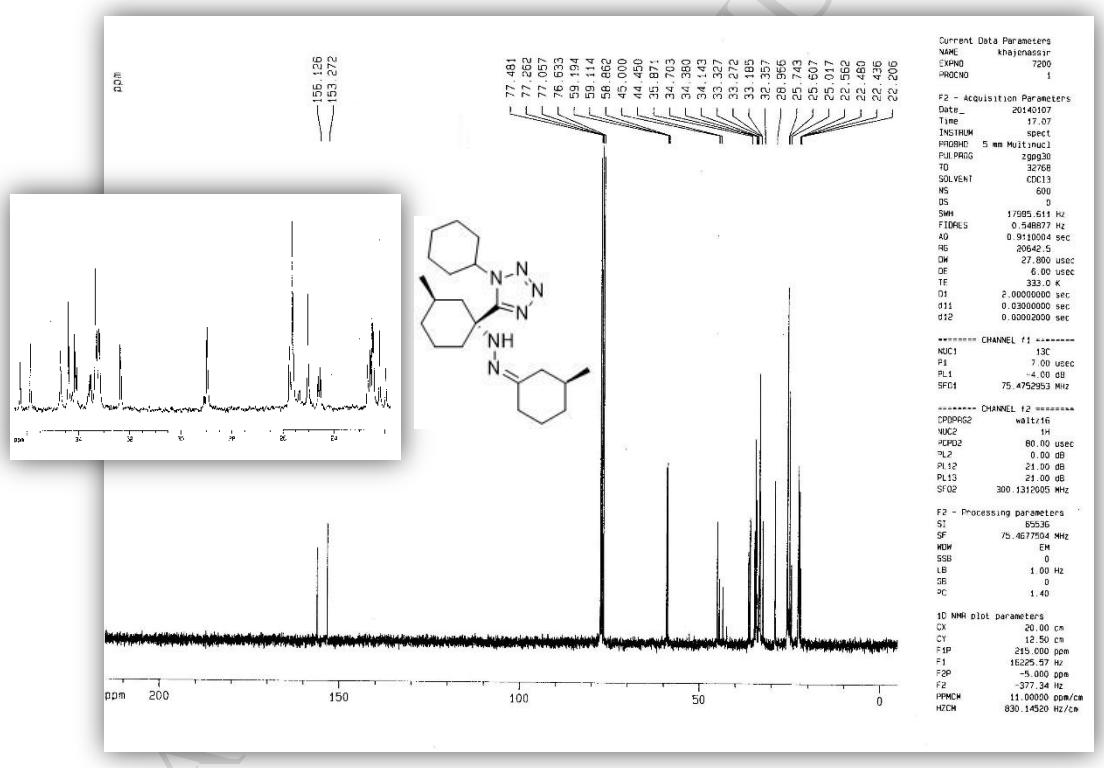


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
373.30753	C 20 H 41 N 2 O 4	373.30608	-1.4	-3.9	22.3	1.5	ok	even
	C 21 H 37 N 6	373.30742	-0.1	-0.3	31.5	6.5	ok	even
	C 23 H 39 N 3 O	373.30876	-0.2	-0.7	574.3	6.0	ok	odd
395.28948	C 20 H 37 N 5 O 3	395.28909	-0.4	-1.0	21.7	5.0	ok	odd
	C 21 H 36 N 6 Na	395.28937	-0.1	-0.3	25.5	6.5	ok	even
	C 23 H 38 N 3 Na O	395.29071	-0.5	-1.2	585.5	6.0	ok	odd
	C 18 H 35 N 8 O 2	395.28775	-0.1	-0.2	590.4	5.5	ok	even
	C 20 H 40 N 2 Na O 4	395.28803	0.2	0.5	590.5	1.5	ok	even
	C 22 H 39 N 2 O 4	395.29043	-0.7	-1.9	591.7	4.5	ok	even
	C 19 H 41 N O 7	395.28775	-0.1	-0.2	596.8	0.0	ok	odd
411.26345	C 21 H 36 K N 6	411.26330	-0.1	-0.4	22.1	6.5	ok	even
	C 23 H 33 N 5 O 2	411.26288	-0.6	-1.4	34.7	10.0	ok	odd
	C 25 H 35 N 2 O 3	411.26422	0.8	1.9	41.4	9.5	ok	even
	C 23 H 38 K N 3 O	411.26465	-0.6	-1.5	507.6	6.0	ok	odd
	C 20 H 40 K N 2 O 4	411.26197	0.2	0.4	528.4	1.5	ok	even
	C 21 H 31 N 8 O	411.26153	-0.3	-0.6	584.9	10.5	ok	even
	C 22 H 37 N O 6	411.26154	-0.3	-0.6	591.2	5.0	ok	odd

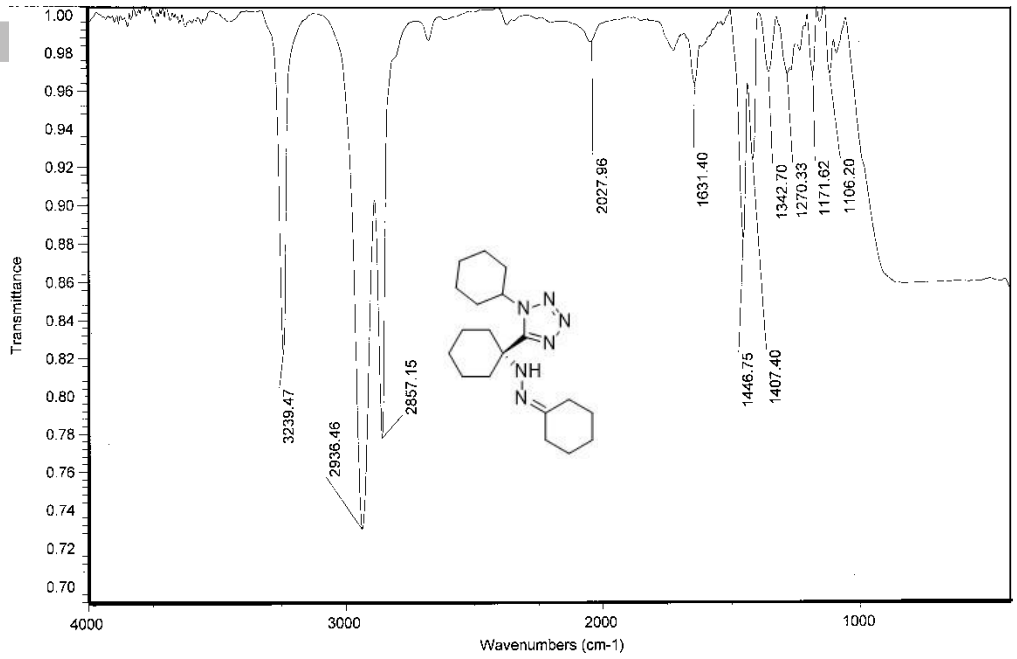
ESI-HRMS **(5g)**



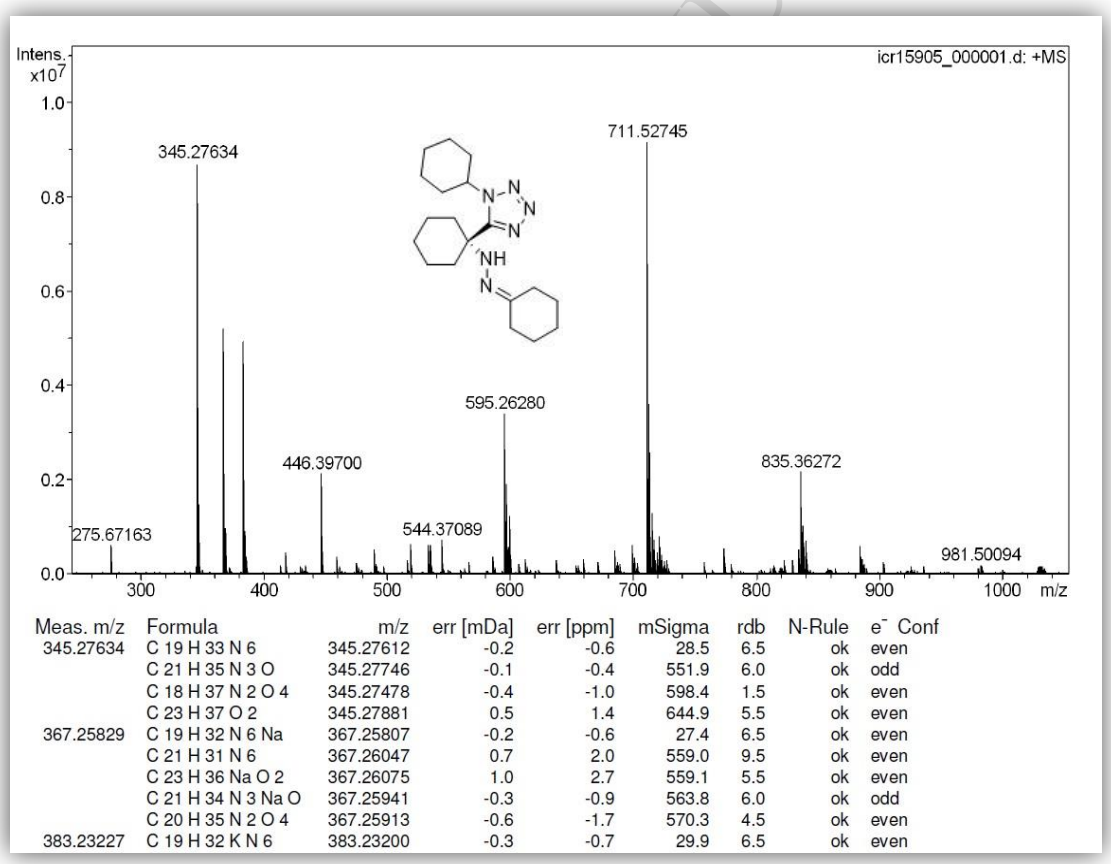
^{1}HNMR (300MHz, $CDCl_3$) **(5g)**



^{13}CNMR (75MHz, $CDCl_3$) **(5g)**

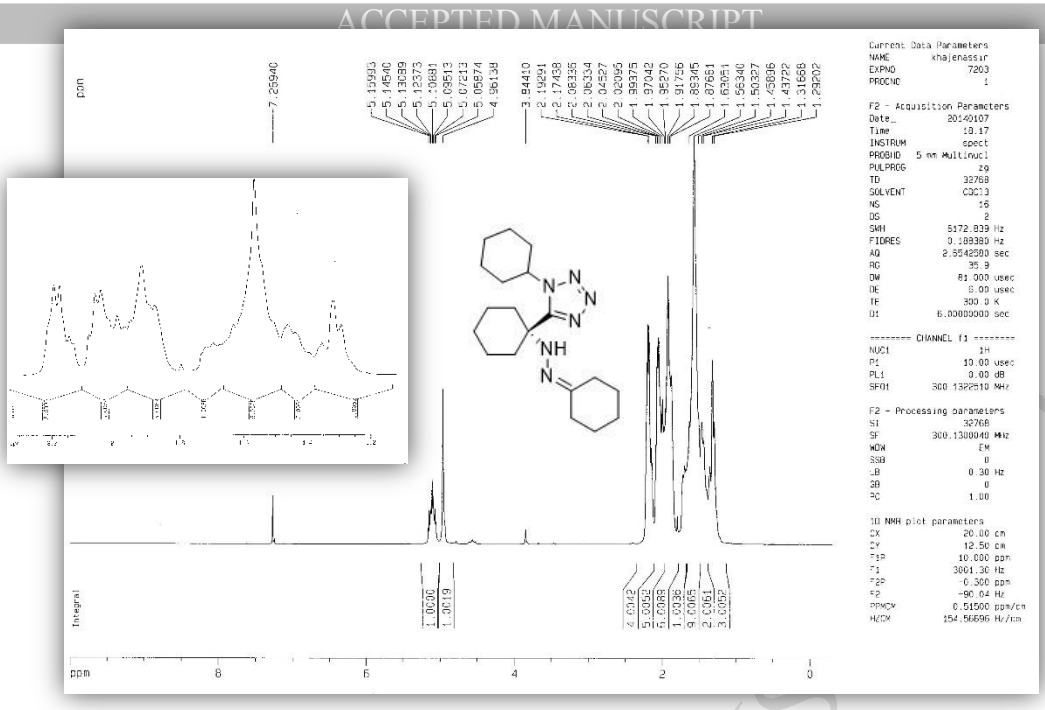


IR (KBr) **(5h)**

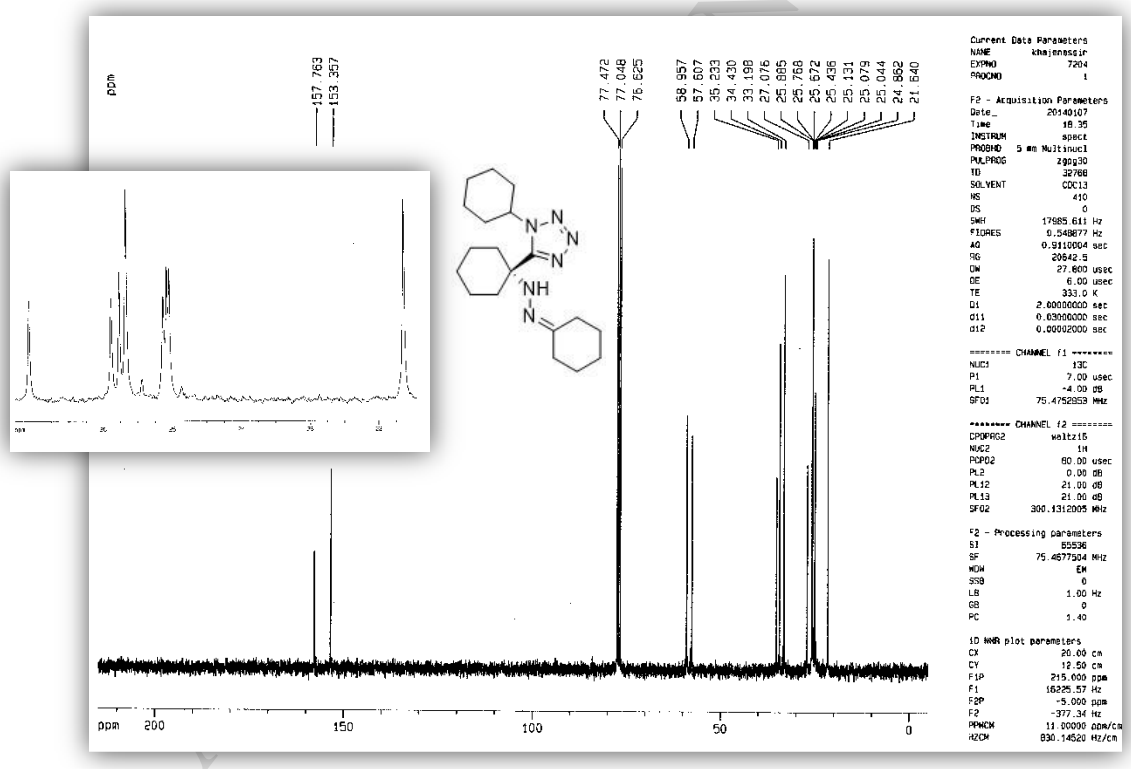


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
345.27634	C 19 H 33 N 6	345.27612	-0.2	-0.6	28.5	6.5	ok	even
	C 21 H 35 N 3 O	345.27746	-0.1	-0.4	551.9	6.0	ok	odd
	C 18 H 37 N 2 O 4	345.27478	-0.4	-1.0	598.4	1.5	ok	even
	C 23 H 37 O 2	345.27881	0.5	1.4	644.9	5.5	ok	even
367.25829	C 19 H 32 N 6 Na	367.25807	-0.2	-0.6	27.4	6.5	ok	even
	C 21 H 31 N 6	367.26047	0.7	2.0	559.0	9.5	ok	even
	C 23 H 36 Na O 2	367.26075	1.0	2.7	559.1	5.5	ok	even
	C 21 H 34 N 3 Na O	367.25941	-0.3	-0.9	563.8	6.0	ok	odd
	C 20 H 35 N 2 O 4	367.25913	-0.6	-1.7	570.3	4.5	ok	even
383.23227	C 19 H 32 K N 6	383.23200	-0.3	-0.7	29.9	6.5	ok	even

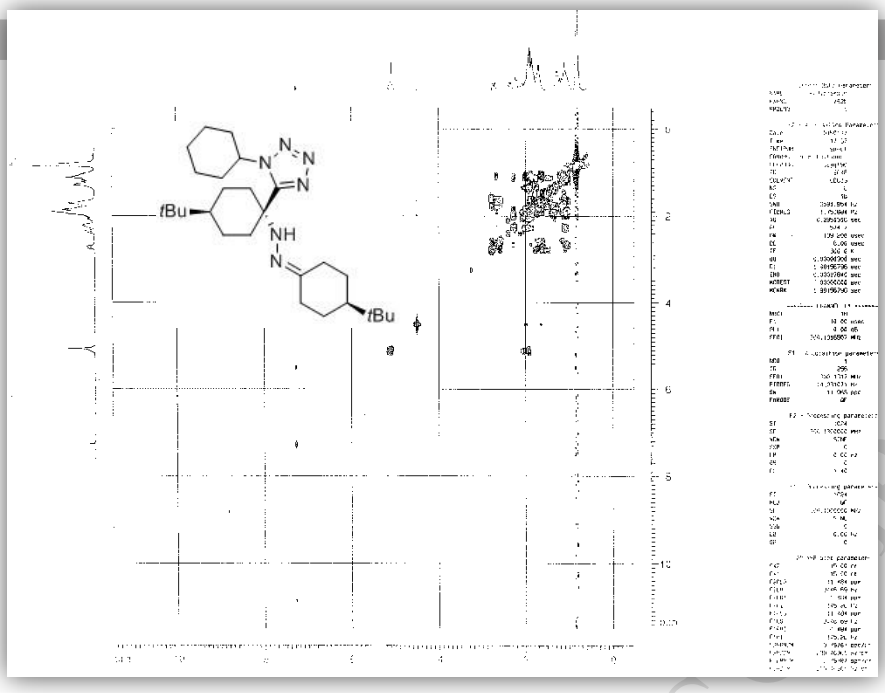
ESI-HRMS **(5h)**



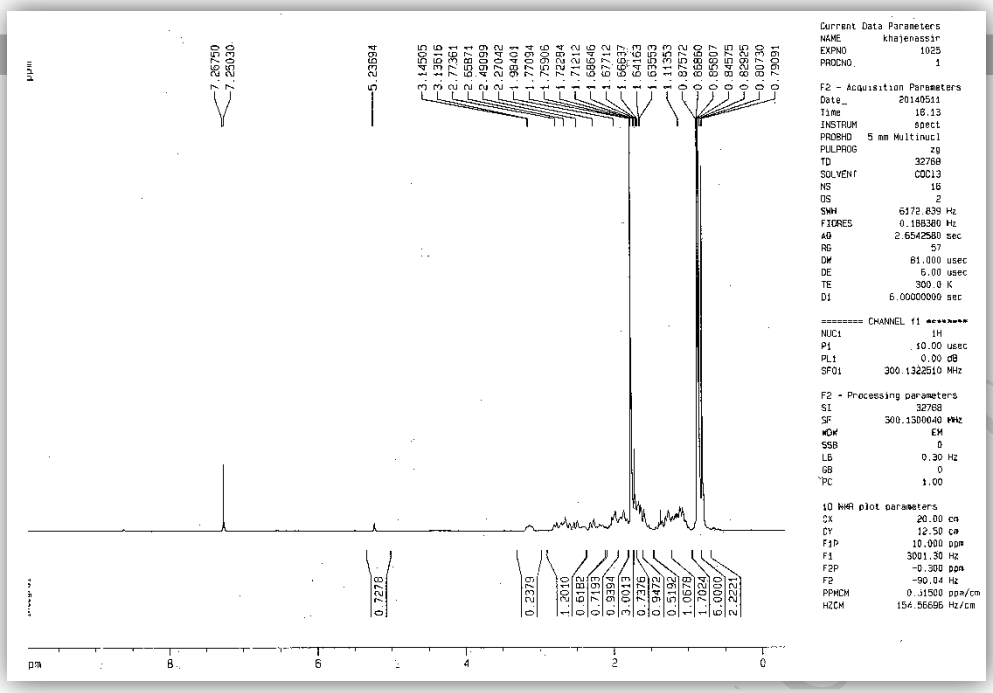
^{1}HNMR (300MHz, $CDCl_3$) **(5h)**



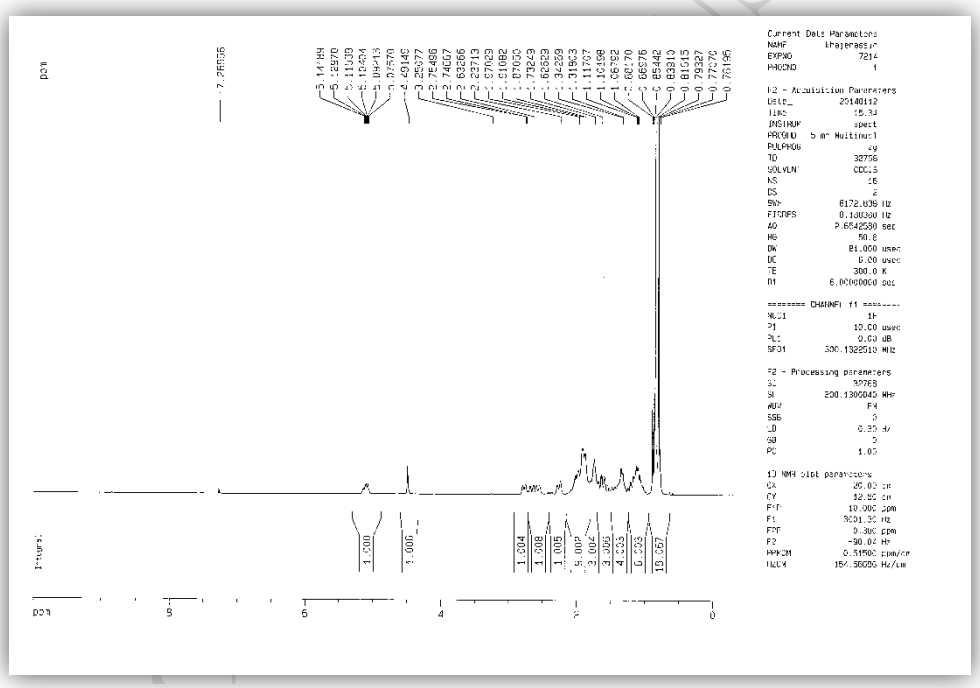
^{13}CNMR (75MHz, $CDCl_3$) **(5h)**



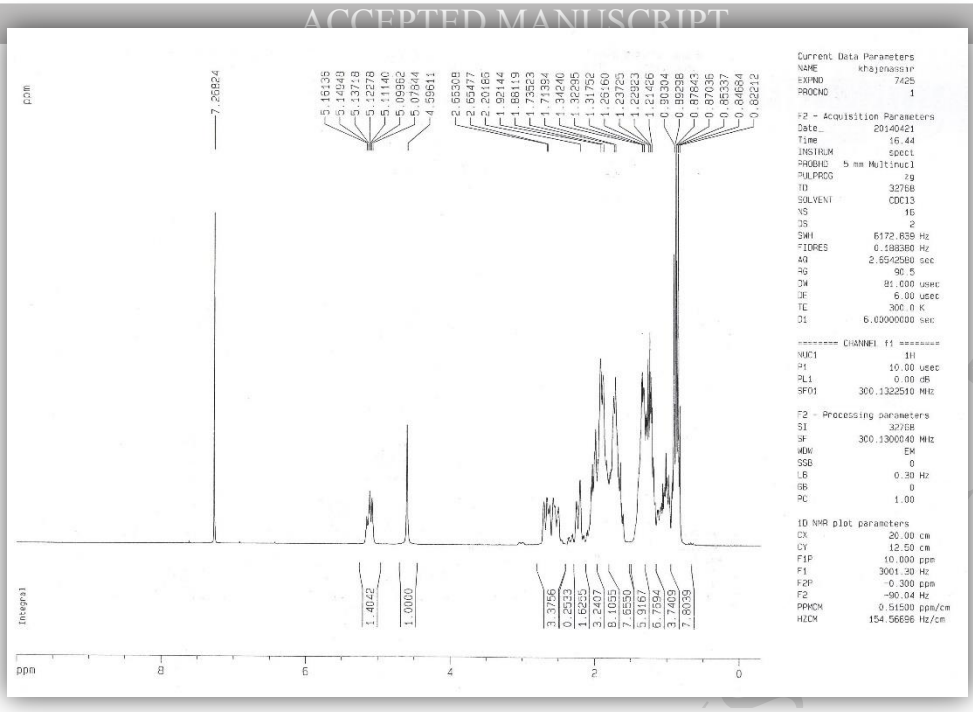
^{1}H-^{1}H COSY (300 MHz, $CDCl_3$) (**5d**)



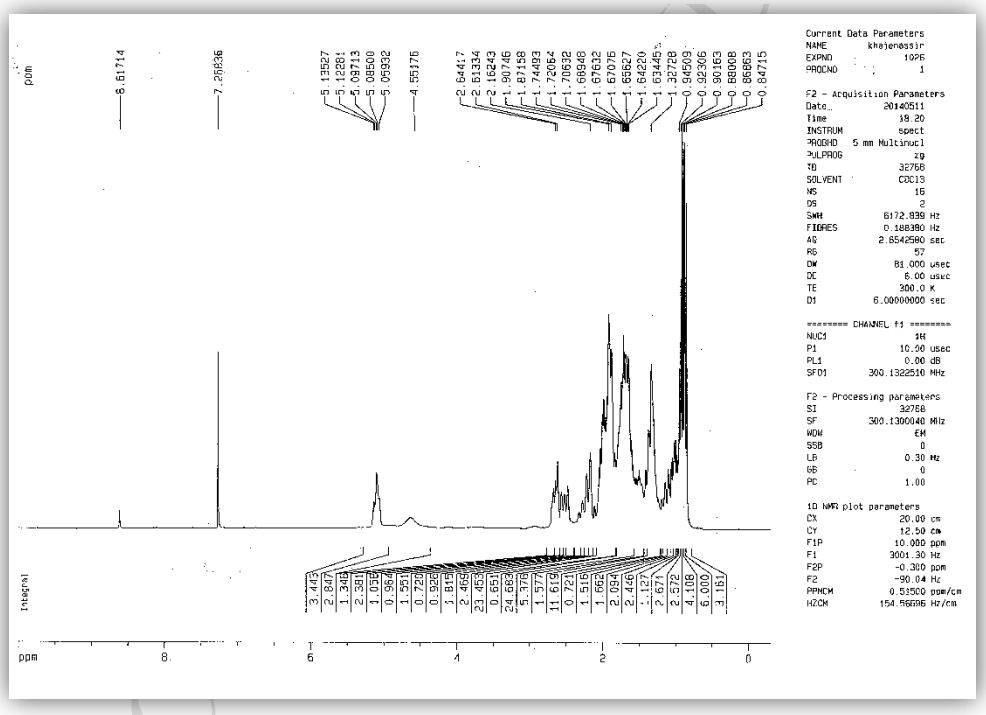
Crude ^{1}HNMR (300MHz, $CDCl_3$) **(5a)**



Crude ^{1}HNMR (300MHz, $CDCl_3$) **(5d)**



Crude ^{1}HNMR (300MHz, $CDCl_3$) **(5e)**



Crude ^{1}HNMR (300MHz, $CDCl_3$) **(5f)**

X-Ray Crystallographic Data 5a

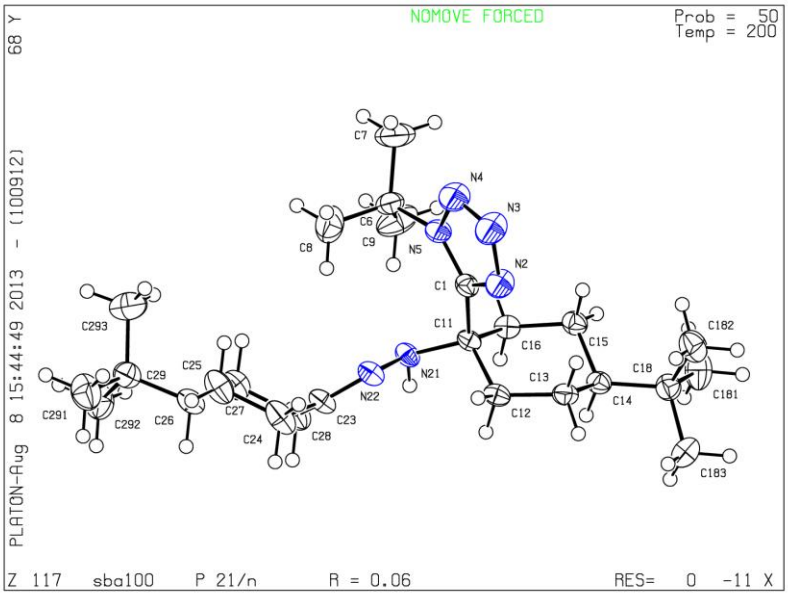


Table 1: Crystal data and structure refinement for **5a**.

Identification code	5a	
Empirical formula	$C_{25}H_{46}N_6$	
Formula weight	430.68	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Z	4	
Unit cell dimensions	a = 6.2048(7) Å	α = 90 deg.
	b = 29.336(3) Å	β = 98.484(4) deg.
	c = 14.4809(17) Å	γ = 90 deg.
Volume	2607.0(5) $Å^3$	
Density (calculated)	1.10 g/cm^3	
Absorption coefficient	0.07 mm^{-1}	
Crystal shape	plate	
Crystal size	0.370 x 0.230 x 0.030 mm^3	
Crystal colour	colourless	
Theta range for data collection	1.4 to 26.0 deg.	
Index ranges	-7≤h≤7, -35≤k≤36, -17≤l≤17	
Reflections collected	16371	
Independent reflections	5139 (R(int) = 0.0805)	
Observed reflections	3027 (I > 2σ(I))	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.96 and 0.90	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	5139 / 0 / 285	
Goodness-of-fit on F^2	0.99	
Final R indices (I>2sigma(I))	R1 = 0.057, wR2 = 0.109	

Tabelle 2: Atomkoordinaten und äquivalente isotrope Auslenkungsparameter (Å^2) für sba100. U_{eq} wird berechnet als ein Drittel der Spur des orthogonalen U_{ij} Tensors. (Atomic coordinates and equivalent isotropic displacement parameters (Å^2) for sba100. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.)

Atom	x	y	z	U_{eq}
C1	0.2043(3)	0.2416(1)	0.6006(1)	0.0278(5)
N2	0.3606(3)	0.2677(1)	0.6441(1)	0.0404(5)
N3	0.4119(4)	0.2508(1)	0.7314(1)	0.0545(6)
N4	0.2930(4)	0.2156(1)	0.7426(1)	0.0491(6)
N5	0.1569(3)	0.2088(1)	0.6616(1)	0.0329(4)
C6	0.0236(4)	0.1658(1)	0.6594(2)	0.0375(6)
C7	-0.0138(5)	0.1549(1)	0.7584(2)	0.0667(9)
H7A	0.1270	0.1513	0.7984	0.100
H7B	-0.0973	0.1265	0.7585	0.100
H7C	-0.0952	0.1798	0.7823	0.100
C8	0.1563(5)	0.1283(1)	0.6224(2)	0.0685(9)
H8A	0.1782	0.1355	0.5584	0.103
H8B	0.0783	0.0992	0.6229	0.103
H8C	0.2981	0.1258	0.6621	0.103
C9	-0.1968(4)	0.1711(1)	0.6005(2)	0.0622(8)
H9A	-0.1778	0.1777	0.5359	0.093
H9B	-0.2758	0.1962	0.6250	0.093
H9C	-0.2799	0.1428	0.6025	0.093
C11	0.1001(3)	0.2529(1)	0.5019(1)	0.0256(5)
C12	0.2422(3)	0.2866(1)	0.4557(1)	0.0295(5)
H12A	0.3949	0.2759	0.4677	0.035
H12B	0.1955	0.2855	0.3873	0.035
C13	0.2374(3)	0.3362(1)	0.4869(1)	0.0306(5)
H13A	0.3181	0.3551	0.4469	0.037
H13B	0.3126	0.3388	0.5519	0.037
C14	0.0055(3)	0.3547(1)	0.4818(1)	0.0290(5)
H14	-0.0647	0.3510	0.4155	0.035
C15	-0.1175(3)	0.3230(1)	0.5401(1)	0.0304(5)
H15A	-0.0442	0.3229	0.6056	0.036
H15B	-0.2677	0.3345	0.5394	0.036
C16	-0.1264(3)	0.2744(1)	0.5023(1)	0.0286(5)
H16A	-0.2054	0.2745	0.4377	0.034
H16B	-0.2100	0.2552	0.5407	0.034
C18	-0.0064(4)	0.4060(1)	0.5045(2)	0.0349(5)
N21	0.0689(3)	0.2105(1)	0.4468(1)	0.0284(4)
H21	-0.009(3)	0.2172(6)	0.3891(15)	0.033(6)
N22	0.2709(3)	0.1910(1)	0.4354(1)	0.0314(4)
C23	0.2688(3)	0.1611(1)	0.3711(1)	0.0333(5)
C24	0.4784(4)	0.1373(1)	0.3624(2)	0.0464(7)
H24A	0.5961	0.1496	0.4094	0.056
H24B	0.5182	0.1431	0.2997	0.056
C25	0.4573(4)	0.0860(1)	0.3768(2)	0.0437(6)
H25A	0.4361	0.0802	0.4422	0.052
H25B	0.5942	0.0709	0.3663	0.052
C26	0.2665(3)	0.0654(1)	0.3108(2)	0.0347(6)
H26	0.2962	0.0717	0.2460	0.042
C27	0.0595(3)	0.0917(1)	0.3220(2)	0.0385(6)
H27A	-0.0626	0.0796	0.2769	0.046
H27B	0.0242	0.0863	0.3856	0.046

C28	0.0776(4)	0.1427(1)	0.3067(2)	0.0387(6)
H28A	0.0964	0.1488	0.2411	0.046
H28B	-0.0575	0.1581	0.3190	0.046
C29	0.2449(4)	0.0131(1)	0.3182(2)	0.0395(6)
C181	-0.2429(4)	0.4208(1)	0.5047(2)	0.0540(7)
H18A	-0.3049	0.4039	0.5528	0.081
H18B	-0.3279	0.4146	0.4434	0.081
H18C	-0.2473	0.4536	0.5179	0.081
C182	0.1255(4)	0.4177(1)	0.5989(2)	0.0473(7)
H18D	0.0702	0.4002	0.6480	0.071
H18E	0.1120	0.4504	0.6111	0.071
H18F	0.2790	0.4101	0.5981	0.071
C183	0.0836(4)	0.4334(1)	0.4290(2)	0.0566(8)
H18G	0.0000	0.4263	0.3679	0.085
H18H	0.2370	0.4254	0.4289	0.085
H18I	0.0716	0.4660	0.4417	0.085
C291	0.4614(4)	-0.0099(1)	0.3094(2)	0.0582(8)
H29A	0.4452	-0.0431	0.3141	0.087
H29B	0.5734	0.0007	0.3596	0.087
H29C	0.5045	-0.0023	0.2488	0.087
C292	0.0766(4)	-0.0042(1)	0.2376(2)	0.0537(7)
H29D	0.0617	-0.0373	0.2425	0.081
H29E	0.1249	0.0034	0.1780	0.081
H29F	-0.0644	0.0103	0.2407	0.081
C293	0.1740(5)	-0.0013(1)	0.4105(2)	0.0675(8)
H29G	0.1606	-0.0346	0.4121	0.101
H29H	0.0329	0.0126	0.4161	0.101
H29I	0.2829	0.0088	0.4625	0.101

Tabelle 3: H-Atomkoordinaten und isotrope Auslenkungsparameter ($Å^2$) für sba100.
(Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for sba100.)

Atom	x	y	z	U_{eq}
H7A	0.1270	0.1513	0.7984	0.100
H7B	-0.0973	0.1265	0.7585	0.100
H7C	-0.0952	0.1798	0.7823	0.100
H8A	0.1782	0.1355	0.5584	0.103
H8B	0.0783	0.0992	0.6229	0.103
H8C	0.2981	0.1258	0.6621	0.103
H9A	-0.1778	0.1777	0.5359	0.093
H9B	-0.2758	0.1962	0.6250	0.093
H9C	-0.2799	0.1428	0.6025	0.093
H12A	0.3949	0.2759	0.4677	0.035
H12B	0.1955	0.2855	0.3873	0.035
H13A	0.3181	0.3551	0.4469	0.037
H13B	0.3126	0.3388	0.5519	0.037
H14	-0.0647	0.3510	0.4155	0.035
H15A	-0.0442	0.3229	0.6056	0.036
H15B	-0.2677	0.3345	0.5394	0.036
H16A	-0.2054	0.2745	0.4377	0.034
H16B	-0.2100	0.2552	0.5407	0.034
H21	-0.009(3)	0.2172(6)	0.3891(15)	0.033(6)
H24A	0.5961	0.1496	0.4094	0.056
H24B	0.5182	0.1431	0.2997	0.056
H25A	0.4361	0.0802	0.4422	0.052
H25B	0.5942	0.0709	0.3663	0.052
H26	0.2962	0.0717	0.2460	0.042

H27A	-0.0626	0.0796	0.2769	0.046
H27B	0.0242	0.0863	0.3856	0.046
H28A	0.0964	0.1488	0.2411	0.046
H28B	-0.0575	0.1581	0.3190	0.046
H18A	-0.3049	0.4039	0.5528	0.081
H18B	-0.3279	0.4146	0.4434	0.081
H18C	-0.2473	0.4536	0.5179	0.081
H18D	0.0702	0.4002	0.6480	0.071
H18E	0.1120	0.4504	0.6111	0.071
H18F	0.2790	0.4101	0.5981	0.071
H18G	0.0000	0.4263	0.3679	0.085
H18H	0.2370	0.4254	0.4289	0.085
H18I	0.0716	0.4660	0.4417	0.085
H29A	0.4452	-0.0431	0.3141	0.087
H29B	0.5734	0.0007	0.3596	0.087
H29C	0.5045	-0.0023	0.2488	0.087
H29D	0.0617	-0.0373	0.2425	0.081
H29E	0.1249	0.0034	0.1780	0.081
H29F	-0.0644	0.0103	0.2407	0.081
H29G	0.1606	-0.0346	0.4121	0.101
H29H	0.0329	0.0126	0.4161	0.101
H29I	0.2829	0.0088	0.4625	0.101

Tabelle 4: Anisotrope Auslenkungsparameter ($Å^2$) für sba100. Der Exponent für den anisotropen Auslenkungsparameter hat die Form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$
(Anisotropic displacement parameters ($Å^2$) for sba100. The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$)

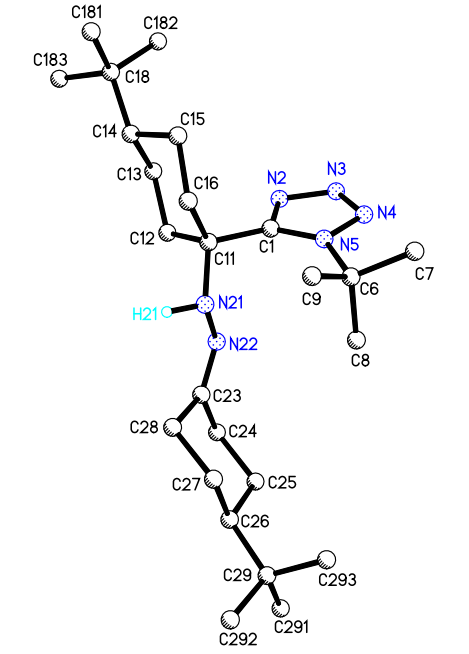
Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0299(12)	0.0303(12)	0.0224(11)	-0.0031(9)	0.0011(9)	0.0017(10)
N2	0.0469(12)	0.0390(11)	0.0300(11)	-0.0004(9)	-0.0119(9)	-0.0051(10)
N3	0.0758(16)	0.0453(13)	0.0337(12)	0.0014(10)	-0.0204(11)	-0.0093(12)
N4	0.0687(15)	0.0470(13)	0.0255(11)	0.0006(10)	-0.0134(10)	-0.0057(12)
N5	0.0423(11)	0.0356(11)	0.0186(9)	-0.0012(8)	-0.0030(8)	-0.0014(9)
C6	0.0479(15)	0.0359(13)	0.0293(12)	0.0056(10)	0.0081(11)	-0.0071(12)
C7	0.090(2)	0.075(2)	0.0375(16)	0.0127(14)	0.0172(15)	-0.0160(17)
C8	0.082(2)	0.0387(16)	0.093(2)	-0.0031(15)	0.0388(19)	-0.0057(15)
C9	0.0566(18)	0.0673(19)	0.0587(18)	0.0218(15)	-0.0053(14)	-0.0262(15)
C11	0.0256(11)	0.0316(12)	0.0191(11)	-0.0034(9)	0.0018(8)	-0.0013(10)
C12	0.0251(12)	0.0385(13)	0.0257(11)	-0.0006(10)	0.0061(9)	0.0009(10)
C13	0.0324(13)	0.0346(13)	0.0259(12)	0.0008(9)	0.0077(9)	-0.0035(10)
C14	0.0306(12)	0.0325(12)	0.0234(11)	0.0001(9)	0.0020(9)	0.0006(10)
C15	0.0270(12)	0.0348(13)	0.0299(12)	0.0001(10)	0.0063(9)	0.0038(10)
C16	0.0233(12)	0.0346(13)	0.0271(12)	0.0014(9)	0.0018(9)	-0.0009(10)
C18	0.0364(13)	0.0338(13)	0.0342(13)	-0.0019(10)	0.0040(10)	0.0007(11)
N21	0.0277(10)	0.0346(11)	0.0215(10)	-0.0058(8)	-0.0009(8)	0.0021(8)
N22	0.0279(10)	0.0394(11)	0.0273(10)	-0.0050(8)	0.0058(8)	0.0006(9)
C23	0.0330(13)	0.0422(14)	0.0257(12)	-0.0088(10)	0.0077(10)	-0.0015(11)
C24	0.0323(14)	0.0578(17)	0.0508(16)	-0.0251(13)	0.0122(12)	-0.0086(13)
C25	0.0270(13)	0.0552(16)	0.0481(15)	-0.0164(12)	0.0029(11)	0.0060(12)
C26	0.0323(13)	0.0437(14)	0.0294(12)	-0.0077(10)	0.0086(10)	-0.0017(11)
C27	0.0307(13)	0.0462(15)	0.0370(13)	-0.0097(11)	-0.0003(10)	-0.0029(11)
C28	0.0394(14)	0.0438(14)	0.0307(13)	-0.0097(11)	-0.0023(10)	0.0003(12)
C29	0.0437(15)	0.0413(15)	0.0342(13)	-0.0005(11)	0.0076(11)	0.0027(12)
C181	0.0453(16)	0.0416(15)	0.0728(19)	-0.0075(14)	0.0011(14)	0.0101(13)
C182	0.0506(16)	0.0454(15)	0.0455(15)	-0.0126(12)	0.0054(12)	-0.0046(12)
C183	0.082(2)	0.0344(15)	0.0569(18)	0.0092(13)	0.0200(15)	-0.0005(14)

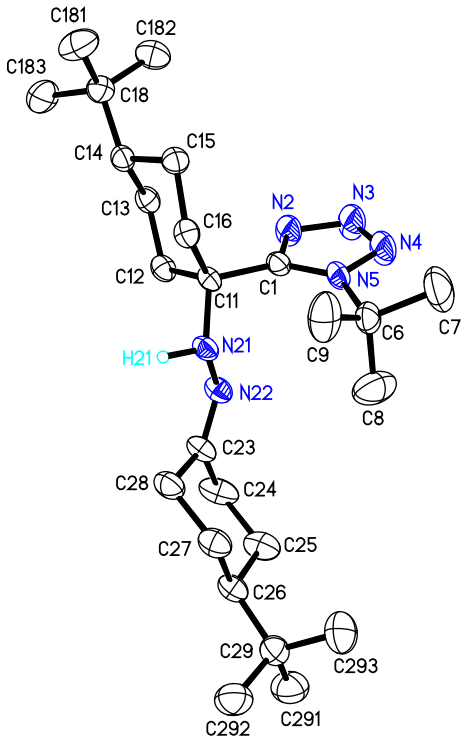
C291	0.0597(18)	0.0535(17)	0.0604(18)	-0.0085(14)	0.0057(14)	0.0115(14)
C292	0.0577(17)	0.0443(16)	0.0571(18)	-0.0078(13)	0.0017(14)	-0.0049(13)
C293	0.089(2)	0.067(2)	0.0489(18)	0.0122(15)	0.0196(16)	-0.0068(17)

Tabelle 5: Bindungslängen (Å) und -winkel (°) für sba100.
(Bond lengths (Å) and angles (deg) for sba100.)

C1-N2	1.320(2)	C27-H27A	0.9900
C1-N5	1.366(3)	C27-H27B	0.9900
C1-C11	1.516(3)	C28-H28A	0.9900
N2-N3	1.352(2)	C28-H28B	0.9900
N3-N4	1.292(3)	C29-C291	1.526(3)
N4-N5	1.356(2)	C29-C293	1.527(3)
N5-C6	1.507(3)	C29-C292	1.534(3)
C6-C9	1.508(3)	C181-H18A	0.9800
C6-C8	1.519(3)	C181-H18B	0.9800
C6-C7	1.521(3)	C181-H18C	0.9800
C7-H7A	0.9800	C182-H18D	0.9800
C7-H7B	0.9800	C182-H18E	0.9800
C7-H7C	0.9800	C182-H18F	0.9800
C8-H8A	0.9800	C183-H18G	0.9800
C8-H8B	0.9800	C183-H18H	0.9800
C8-H8C	0.9800	C183-H18I	0.9800
C9-H9A	0.9800	C291-H29A	0.9800
C9-H9B	0.9800	C291-H29B	0.9800
C9-H9C	0.9800	C291-H29C	0.9800
C11-N21	1.475(2)	C292-H29D	0.9800
C11-C16	1.541(3)	C292-H29E	0.9800
C11-C12	1.543(3)	C292-H29F	0.9800
C12-C13	1.525(3)	C293-H29G	0.9800
C12-H12A	0.9900	C293-H29H	0.9800
C12-H12B	0.9900	C293-H29I	0.9800
C13-C14	1.529(3)	N2-C1-N5	108.24(18)
C13-H13A	0.9900	N2-C1-C11	120.22(19)
C13-H13B	0.9900	N5-C1-C11	131.37(18)
C14-C15	1.533(3)	C1-N2-N3	106.74(18)
C14-C18	1.544(3)	N4-N3-N2	110.49(18)
C14-H14	1.0000	N3-N4-N5	107.69(17)
C15-C16	1.524(3)	N4-N5-C1	106.82(17)
C15-H15A	0.9900	N4-N5-C6	114.35(17)
C15-H15B	0.9900	C1-N5-C6	138.18(17)
C16-H16A	0.9900	N5-C6-C9	112.02(18)
C16-H16B	0.9900	N5-C6-C8	106.87(18)
C18-C182	1.525(3)	C9-C6-C8	111.7(2)
C18-C183	1.528(3)	N5-C6-C7	108.40(19)
C18-C181	1.531(3)	C9-C6-C7	107.6(2)
N21-N22	1.410(2)	C8-C6-C7	110.2(2)
N21-H21	0.92(2)	C6-C7-H7A	109.5
N22-C23	1.277(2)	C6-C7-H7B	109.5
C23-C28	1.497(3)	H7A-C7-H7B	109.5
C23-C24	1.498(3)	C6-C7-H7C	109.5
C24-C25	1.527(3)	H7A-C7-H7C	109.5
C24-H24A	0.9900	H7B-C7-H7C	109.5
C24-H24B	0.9900	C6-C8-H8A	109.5
C25-C26	1.532(3)	C6-C8-H8B	109.5
C25-H25A	0.9900	H8A-C8-H8B	109.5
C25-H25B	0.9900	C6-C8-H8C	109.5
C26-C27	1.527(3)	H8A-C8-H8C	109.5
C26-C29	1.545(3)	H8B-C8-H8C	109.5
C26-H26	1.0000	C6-C9-H9A	109.5
C27-C28	1.521(3)	C6-C9-H9B	109.5

H9A-C9-H9B	109.5
C6-C9-H9C	109.5
H9A-C9-H9C	109.5
H9B-C9-H9C	109.5
N21-C11-C1	109.20(17)
N21-C11-C16	107.43(16)
C1-C11-C16	110.44(16)
N21-C11-C12	110.09(16)
C1-C11-C12	111.02(16)
C16-C11-C12	108.59(16)
C13-C12-C11	116.45(17)
C13-C12-H12A	108.2
C11-C12-H12A	108.2
C13-C12-H12B	108.2
C11-C12-H12B	108.2
H12A-C12-H12B	107.3
C12-C13-C14	112.55(17)
C12-C13-H13A	109.1
C14-C13-H13A	109.1
C12-C13-H13B	109.1
C14-C13-H13B	109.1
H13A-C13-H13B	107.8
C13-C14-C15	107.43(16)
C13-C14-C18	114.18(17)
C15-C14-C18	115.58(17)
C13-C14-H14	106.3
C15-C14-H14	106.3
C18-C14-H14	106.3
C16-C15-C14	111.39(17)
C16-C15-H15A	109.3
C14-C15-H15A	109.3
C16-C15-H15B	109.3
C14-C15-H15B	109.3
H15A-C15-H15B	108.0
C15-C16-C11	113.52(16)
C15-C16-H16A	108.9
C11-C16-H16A	108.9
C15-C16-H16B	108.9
C11-C16-H16B	108.9
H16A-C16-H16B	107.7
C182-C18-C183	108.6(2)
C182-C18-C181	108.80(19)
C183-C18-C181	107.6(2)
C182-C18-C14	111.97(18)
C183-C18-C14	109.05(18)
C181-C18-C14	110.70(18)
N22-N21-C11	110.98(15)
N22-N21-H21	109.5(13)
C11-N21-H21	108.7(12)
C23-N22-N21	116.76(17)
N22-C23-C28	128.6(2)
N22-C23-C24	117.73(19)
C28-C23-C24	113.43(18)
C23-C24-C25	110.76(19)
C23-C24-H24A	109.5
C25-C24-H24A	109.5
C23-C24-H24B	109.5
C25-C24-H24B	109.5
H24A-C24-H24B	108.1
C24-C25-C26	112.2(2)
C24-C25-H25A	109.2
C26-C25-H25A	109.2
C24-C25-H25B	109.2
C26-C25-H25B	109.2
H25A-C25-H25B	107.9
C27-C26-C25	108.62(17)
C27-C26-C29	114.28(18)
C25-C26-C29	114.57(19)
C27-C26-H26	106.2
C25-C26-H26	106.2
C29-C26-H26	106.2
C28-C27-C26	113.61(19)
C28-C27-H27A	108.8
C26-C27-H27A	108.8
C28-C27-H27B	108.8
C26-C27-H27B	108.8
H27A-C27-H27B	107.7
C23-C28-C27	109.44(18)
C23-C28-H28A	109.8
C27-C28-H28A	109.8
C23-C28-H28B	109.8
C27-C28-H28B	109.8
H28A-C28-H28B	108.2
C291-C29-C293	108.8(2)
C291-C29-C292	107.4(2)
C293-C29-C292	108.8(2)
C291-C29-C26	110.4(2)
C293-C29-C26	112.0(2)
C292-C29-C26	109.37(19)
C18-C181-H18A	109.5
C18-C181-H18B	109.5
H18A-C181-H18B	109.5
C18-C181-H18C	109.5
H18A-C181-H18C	109.5
H18B-C181-H18C	109.5
C18-C182-H18D	109.5
C18-C182-H18E	109.5
H18D-C182-H18E	109.5
C18-C182-H18F	109.5
H18D-C182-H18F	109.5
H18E-C182-H18F	109.5
C18-C183-H18G	109.5
C18-C183-H18H	109.5
H18G-C183-H18H	109.5
C18-C183-H18I	109.5
H18G-C183-H18I	109.5
H18H-C183-H18I	109.5
C29-C291-H29A	109.5
C29-C291-H29B	109.5
H29A-C291-H29B	109.5
C29-C291-H29C	109.5
H29A-C291-H29C	109.5
H29B-C291-H29C	109.5
C29-C292-H29D	109.5
C29-C292-H29E	109.5
H29D-C292-H29E	109.5
C29-C292-H29F	109.5
H29D-C292-H29F	109.5
H29E-C292-H29F	109.5
C29-C293-H29G	109.5
C29-C293-H29H	109.5
H29G-C293-H29H	109.5
C29-C293-H29I	109.5
H29G-C293-H29I	109.5
H29H-C293-H29I	109.5





5a: colourless crystal (plate), dimensions 0.370 x 0.230 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.2048(7) Å, b=29.336(3) Å, c=14.4809(17) Å, alpha=90 deg, beta=98.484(4) deg, gamma=90 deg, V=2607.0(5) $Å^3$, rho=1.097 g/cm^3, T=200(2) K, $Theta_{max}$= 26.022 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.15and a completeness of 100.0% to a resolution of 0.81Å, 16371 reflections measured, 5139 unique (R(int)=0.0805), 3027 observed (I > 2σ(I)), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using

SADABS[1] based on the Laue symmetry of the reciprocal space, mu=0.07mm^{-1}, T_{min}=0.90, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package[2], 285 parameters refined, hydrogen atoms were treated using appropriate riding models, except H21 at N21, which was refined isotropically, goodness of fit 0.99 for observed reflections, final residual values R1(F)=0.057, wR(F^2)=0.109 for observed reflections, residual electron density -0.22 to 0.21 eÅ^{-3}. CCDC 1030930 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Lit. 1: (program SADABS 2012/1 for absorption correction) G. M. Sheldrick, Bruker Analytical X-ray-Division, Madison, Wisconsin 2012

Lit. 2: (software package SHELXTL 2013/3 for structure solution and refinement) Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Lit. APEX, APEX2, SMART, SAINT, SAINT-Plus: Bruker (2007). "Program name(s)". Bruker AXS Inc., Madison, Wisconsin, USA.

X-Ray Crystallographic Data 5d

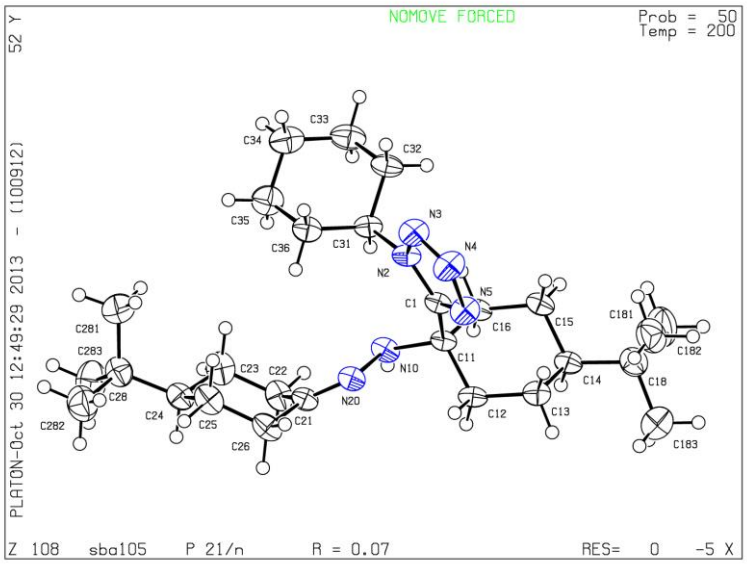


Table 1: Crystal data and structure refinement for sba105.

Identification code	sba105	
Empirical formula	$C_{27}H_{48}N_6$	
Formula weight	456.71	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Z	4	
Unit cell dimensions	a = 6.345(2) Å	α = 90 deg.
	b = 31.355(11) Å	β = 99.872(11) deg.
	c = 14.286(5) Å	γ = 90 deg.
Volume	2799.8(17) $Å^3$	
Density (calculated)	1.08 g/cm^3	
Absorption coefficient	0.06 mm^{-1}	
Crystal shape	needle	
Crystal size	0.420 x 0.050 x 0.030 mm^3	
Crystal colour	colourless	
Theta range for data collection	1.9 to 21.3 deg.	
Index ranges	$-6\le h\le 5$, $-31\le k\le 31$, $-14\le l\le 14$	
Reflections collected	11459	
Independent reflections	3120 (R(int) = 0.1049)	
Observed reflections	1898 ($I > 2\sigma(I)$)	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.96 and 0.82	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	3120 / 270 / 302	
Goodness-of-fit on F^2	1.01	
Final R indices (I>2sigma(I))	R1 = 0.074, wR2 = 0.159	
Largest diff. peak and hole	0.32 and -0.33 $eÅ^{-3}$	

Tabelle 2: Atomkoordinaten und äquivalente isotrope Auslenkungsparameter ($Å^2$) für sba105. U_{eq} wird berechnet als ein Drittel der Spur des orthogonalen U_{ij} Tensors. (Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for sba105. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.)

Atom	x	y	z	U_{eq}
C1	0.6738(7)	0.2531(1)	0.5585(3)	0.0265(11)
N2	0.6256(5)	0.2216(1)	0.6149(2)	0.0302(10)
N3	0.7585(6)	0.2247(1)	0.7003(3)	0.0376(11)
N4	0.8814(6)	0.2574(1)	0.6937(3)	0.0429(11)
N5	0.8334(6)	0.2759(1)	0.6057(3)	0.0367(10)
N10	0.5385(6)	0.2208(1)	0.4084(3)	0.0310(10)
H10	0.457(6)	0.2240(11)	0.357(3)	0.015(12)
C11	0.5596(7)	0.2621(1)	0.4580(3)	0.0254(11)
C12	0.6842(7)	0.2943(1)	0.4083(3)	0.0300(12)
H12A	0.8359	0.2853	0.4178	0.036
H12B	0.6285	0.2933	0.3392	0.036
C13	0.6753(7)	0.3404(1)	0.4412(3)	0.0347(12)
H13A	0.7440	0.3589	0.3991	0.042
H13B	0.7579	0.3430	0.5064	0.042
C14	0.4456(7)	0.3560(1)	0.4407(3)	0.0331(12)
H14	0.3696	0.3532	0.3737	0.040
C15	0.3355(7)	0.3244(1)	0.4995(3)	0.0331(12)
H15A	0.4099	0.3249	0.5663	0.040
H15B	0.1857	0.3336	0.4984	0.040
C16	0.3372(6)	0.2794(1)	0.4614(3)	0.0298(12)
H16A	0.2523	0.2788	0.3964	0.036
H16B	0.2660	0.2604	0.5017	0.036
C18	0.4303(8)	0.4035(2)	0.4673(4)	0.0474(14)
N20	0.7403(6)	0.2032(1)	0.4028(3)	0.0318(10)
C21	0.7457(7)	0.1723(2)	0.3461(3)	0.0321(12)
C22	0.5600(7)	0.1506(1)	0.2836(3)	0.0410(13)
H22A	0.5718	0.1547	0.2160	0.049
H22B	0.4238	0.1636	0.2944	0.049
C23	0.5606(7)	0.1034(2)	0.3064(4)	0.0454(14)
H23A	0.5280	0.0998	0.3713	0.054
H23B	0.4447	0.0894	0.2617	0.054
C24	0.7692(7)	0.0808(1)	0.3010(4)	0.0386(13)
H24	0.7949	0.0849	0.2344	0.046
C25	0.9511(7)	0.1044(2)	0.3647(4)	0.0430(13)
H25A	0.9348	0.1005	0.4319	0.052
H25B	1.0892	0.0914	0.3567	0.052
C26	0.9571(7)	0.1516(2)	0.3433(4)	0.0418(13)
H26A	1.0686	0.1654	0.3905	0.050
H26B	0.9955	0.1557	0.2796	0.050
C28	0.7668(8)	0.0319(2)	0.3169(4)	0.0476(14)
C31	0.4686(7)	0.1869(1)	0.6014(3)	0.0309(11)
H31	0.3768	0.1908	0.5378	0.037
C32	0.3247(7)	0.1890(2)	0.6757(3)	0.0403(13)
H32A	0.2532	0.2172	0.6728	0.048
H32B	0.4114	0.1856	0.7398	0.048
C33	0.1570(8)	0.1538(2)	0.6579(4)	0.0497(14)
H33A	0.0683	0.1545	0.7086	0.060
H33B	0.0619	0.1590	0.5964	0.060
C34	0.2598(8)	0.1105(2)	0.6562(4)	0.0542(15)
H34A	0.1467	0.0887	0.6400	0.065
H34B	0.3397	0.1039	0.7203	0.065

C35	0.4113(8)	0.1082(2)	0.5850(4)	0.0513(15)
H35A	0.3287	0.1106	0.5198	0.062
H35B	0.4850	0.0803	0.5906	0.062
C36	0.5770(7)	0.1438(1)	0.6018(3)	0.0396(13)
H36A	0.6717	0.1394	0.6637	0.048
H36B	0.6664	0.1431	0.5514	0.048
C181	0.5675(9)	0.4136(2)	0.5646(4)	0.0614(16)
H18A	0.5553	0.4440	0.5787	0.092
H18B	0.7174	0.4066	0.5629	0.092
H18C	0.5172	0.3966	0.6139	0.092
C182	0.1983(8)	0.4157(2)	0.4721(5)	0.0743(19)
H18D	0.1921	0.4458	0.4899	0.111
H18E	0.1455	0.3979	0.5196	0.111
H18F	0.1089	0.4111	0.4098	0.111
C183	0.5104(10)	0.4317(2)	0.3922(5)	0.078(2)
H18G	0.5037	0.4618	0.4104	0.117
H18H	0.4198	0.4272	0.3302	0.117
H18I	0.6585	0.4242	0.3883	0.117
C281	0.7092(10)	0.0202(2)	0.4128(4)	0.0759(19)
H28A	0.7093	-0.0109	0.4197	0.114
H28B	0.5667	0.0314	0.4168	0.114
H28C	0.8145	0.0327	0.4637	0.114
C282	0.9865(8)	0.0126(2)	0.3101(4)	0.0673(18)
H28D	0.9837	-0.0182	0.3218	0.101
H28E	1.0963	0.0259	0.3578	0.101
H28F	1.0195	0.0178	0.2466	0.101
C283	0.6047(9)	0.0111(2)	0.2366(4)	0.0719(19)
H28G	0.6001	-0.0197	0.2473	0.108
H28H	0.6489	0.0166	0.1752	0.108
H28I	0.4623	0.0233	0.2363	0.108

Tabelle 3: H-Atomkoordinaten und isotrope Auslenkungsparameter ($Å^2$) für sba105.
(Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for sba105.)

Atom	x	y	z	U_{eq}
H10	0.457(6)	0.2240(11)	0.357(3)	0.015(12)
H12A	0.8359	0.2853	0.4178	0.036
H12B	0.6285	0.2933	0.3392	0.036
H13A	0.7440	0.3589	0.3991	0.042
H13B	0.7579	0.3430	0.5064	0.042
H14	0.3696	0.3532	0.3737	0.040
H15A	0.4099	0.3249	0.5663	0.040
H15B	0.1857	0.3336	0.4984	0.040
H16A	0.2523	0.2788	0.3964	0.036
H16B	0.2660	0.2604	0.5017	0.036
H22A	0.5718	0.1547	0.2160	0.049
H22B	0.4238	0.1636	0.2944	0.049
H23A	0.5280	0.0998	0.3713	0.054
H23B	0.4447	0.0894	0.2617	0.054
H24	0.7949	0.0849	0.2344	0.046
H25A	0.9348	0.1005	0.4319	0.052
H25B	1.0892	0.0914	0.3567	0.052
H26A	1.0686	0.1654	0.3905	0.050
H26B	0.9955	0.1557	0.2796	0.050
H31	0.3768	0.1908	0.5378	0.037
H32A	0.2532	0.2172	0.6728	0.048
H32B	0.4114	0.1856	0.7398	0.048

H33A	0.0683	0.1545	0.7086	0.060
H33B	0.0619	0.1590	0.5964	0.060
H34A	0.1467	0.0887	0.6400	0.065
H34B	0.3397	0.1039	0.7203	0.065
H35A	0.3287	0.1106	0.5198	0.062
H35B	0.4850	0.0803	0.5906	0.062
H36A	0.6717	0.1394	0.6637	0.048
H36B	0.6664	0.1431	0.5514	0.048
H18A	0.5553	0.4440	0.5787	0.092
H18B	0.7174	0.4066	0.5629	0.092
H18C	0.5172	0.3966	0.6139	0.092
H18D	0.1921	0.4458	0.4899	0.111
H18E	0.1455	0.3979	0.5196	0.111
H18F	0.1089	0.4111	0.4098	0.111
H18G	0.5037	0.4618	0.4104	0.117
H18H	0.4198	0.4272	0.3302	0.117
H18I	0.6585	0.4242	0.3883	0.117
H28A	0.7093	-0.0109	0.4197	0.114
H28B	0.5667	0.0314	0.4168	0.114
H28C	0.8145	0.0327	0.4637	0.114
H28D	0.9837	-0.0182	0.3218	0.101
H28E	1.0963	0.0259	0.3578	0.101
H28F	1.0195	0.0178	0.2466	0.101
H28G	0.6001	-0.0197	0.2473	0.108
H28H	0.6489	0.0166	0.1752	0.108
H28I	0.4623	0.0233	0.2363	0.108

Tabelle 4: Anisotrope Auslenkungsparameter ($Å^2$) für sba105. Der Exponent für den anisotropen Auslenkungsparameter hat die Form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$
(Anisotropic displacement parameters ($Å^2$) for sba105. The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.020(3)	0.043(3)	0.013(2)	-0.003(2)	-0.0052(19)	-0.001(2)
N2	0.029(2)	0.048(2)	0.010(2)	0.0003(18)	-0.0068(17)	-0.0038(18)
N3	0.041(3)	0.051(3)	0.014(2)	0.0009(19)	-0.0152(19)	-0.004(2)
N4	0.049(3)	0.050(3)	0.021(2)	0.005(2)	-0.019(2)	-0.009(2)
N5	0.035(2)	0.048(2)	0.019(2)	0.0013(19)	-0.0165(19)	-0.0037(19)
N10	0.022(2)	0.049(2)	0.018(2)	-0.003(2)	-0.009(2)	0.0021(18)
C11	0.024(2)	0.040(2)	0.009(2)	-0.0004(19)	-0.0065(19)	-0.0010(19)
C12	0.023(3)	0.050(3)	0.014(3)	0.003(2)	-0.004(2)	-0.001(2)
C13	0.031(3)	0.045(3)	0.026(3)	-0.001(2)	-0.002(2)	-0.004(2)
C14	0.034(3)	0.047(3)	0.016(3)	0.000(2)	-0.002(2)	0.001(2)
C15	0.026(3)	0.049(3)	0.024(3)	-0.002(2)	0.002(2)	0.003(2)
C16	0.021(2)	0.050(3)	0.017(3)	0.000(2)	-0.003(2)	0.002(2)
C18	0.046(3)	0.048(3)	0.049(3)	-0.002(2)	0.010(3)	0.005(2)
N20	0.026(2)	0.044(2)	0.024(2)	-0.0013(19)	-0.0026(19)	0.0004(18)
C21	0.030(3)	0.043(3)	0.023(3)	-0.004(2)	0.001(2)	-0.001(2)
C22	0.038(3)	0.048(3)	0.031(3)	-0.010(2)	-0.009(2)	0.002(2)
C23	0.033(3)	0.051(3)	0.046(4)	-0.009(3)	-0.011(3)	-0.001(2)
C24	0.037(3)	0.047(3)	0.029(3)	-0.005(2)	-0.001(2)	0.003(2)
C25	0.027(3)	0.055(3)	0.045(3)	-0.009(3)	-0.001(2)	0.003(2)
C26	0.030(3)	0.056(3)	0.040(3)	-0.012(3)	0.005(2)	0.000(2)
C28	0.050(3)	0.047(3)	0.045(3)	-0.005(2)	0.006(3)	0.004(2)
C31	0.028(3)	0.046(3)	0.016(3)	0.001(2)	-0.005(2)	-0.006(2)
C32	0.035(3)	0.062(3)	0.025(3)	0.001(2)	0.005(2)	-0.001(2)
C33	0.038(3)	0.074(4)	0.039(3)	-0.002(3)	0.009(3)	-0.010(3)

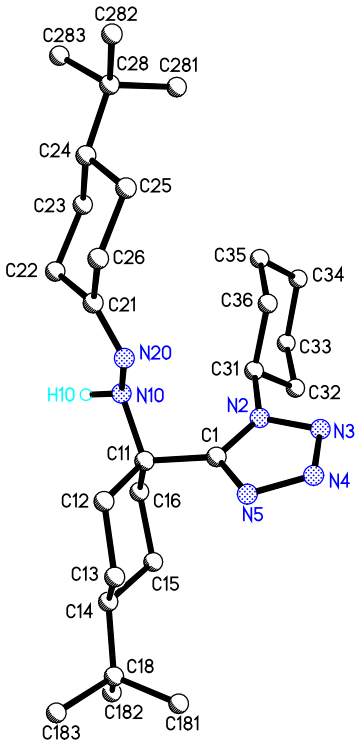
C34	0.052(3)	0.067(3)	0.044(4)	0.003(3)	0.008(3)	-0.014(3)
C35	0.042(3)	0.059(3)	0.052(4)	-0.003(3)	0.006(3)	-0.008(3)
C36	0.036(3)	0.051(3)	0.031(3)	-0.002(2)	0.001(3)	-0.003(2)
C181	0.068(4)	0.056(4)	0.060(4)	-0.017(3)	0.010(3)	-0.006(3)
C182	0.061(4)	0.059(4)	0.105(5)	-0.005(4)	0.019(3)	0.017(3)
C183	0.107(5)	0.056(4)	0.077(5)	0.009(3)	0.033(4)	0.002(3)
C281	0.113(5)	0.060(4)	0.061(4)	0.009(3)	0.034(4)	0.001(3)
C282	0.072(4)	0.059(4)	0.072(5)	-0.003(3)	0.014(3)	0.013(3)
C283	0.075(4)	0.054(4)	0.079(4)	-0.011(3)	-0.009(3)	-0.003(3)

Tabelle 5: Bindungslängen (Å) und -winkel (°) für sba105.
(Bond lengths (Å) and angles (deg) for sba105.)

C1-N5	1.325(5)	C28-C283	1.548(7)
C1-N2	1.342(5)	C31-C36	1.515(6)
C1-C11	1.519(6)	C31-C32	1.515(6)
N2-N3	1.362(5)	C31-H31	1.0000
N2-C31	1.466(5)	C32-C33	1.524(6)
N3-N4	1.301(5)	C32-H32A	0.9900
N4-N5	1.372(5)	C32-H32B	0.9900
N10-N20	1.409(5)	C33-C34	1.508(6)
N10-C11	1.473(6)	C33-H33A	0.9900
N10-H10	0.83(4)	C33-H33B	0.9900
C11-C16	1.521(5)	C34-C35	1.516(6)
C11-C12	1.531(6)	C34-H34A	0.9900
C12-C13	1.522(6)	C34-H34B	0.9900
C12-H12A	0.9900	C35-C36	1.525(6)
C12-H12B	0.9900	C35-H35A	0.9900
C13-C14	1.536(6)	C35-H35B	0.9900
C13-H13A	0.9900	C36-H36A	0.9900
C13-H13B	0.9900	C36-H36B	0.9900
C14-C15	1.539(6)	C181-H18A	0.9800
C14-C18	1.544(6)	C181-H18B	0.9800
C14-H14	1.0000	C181-H18C	0.9800
C15-C16	1.513(6)	C182-H18D	0.9800
C15-H15A	0.9900	C182-H18E	0.9800
C15-H15B	0.9900	C182-H18F	0.9800
C16-H16A	0.9900	C183-H18G	0.9800
C16-H16B	0.9900	C183-H18H	0.9800
C18-C182	1.534(7)	C183-H18I	0.9800
C18-C181	1.541(7)	C281-H28A	0.9800
C18-C183	1.543(7)	C281-H28B	0.9800
N20-C21	1.266(5)	C281-H28C	0.9800
C21-C26	1.496(6)	C282-H28D	0.9800
C21-C22	1.511(6)	C282-H28E	0.9800
C22-C23	1.517(6)	C282-H28F	0.9800
C22-H22A	0.9900	C283-H28G	0.9800
C22-H22B	0.9900	C283-H28H	0.9800
C23-C24	1.515(6)	C283-H28I	0.9800
C23-H23A	0.9900	N5-C1-N2	108.9(4)
C23-H23B	0.9900	N5-C1-C11	125.4(4)
C24-C25	1.532(6)	N2-C1-C11	125.7(4)
C24-C28	1.550(6)	C1-N2-N3	108.4(3)
C24-H24	1.0000	C1-N2-C31	133.4(4)
C25-C26	1.514(6)	N3-N2-C31	118.2(4)
C25-H25A	0.9900	N4-N3-N2	106.2(3)
C25-H25B	0.9900	N3-N4-N5	111.0(3)
C26-H26A	0.9900	C1-N5-N4	105.6(4)
C26-H26B	0.9900	N20-N10-C11	111.3(3)
C28-C281	1.522(7)	N20-N10-H10	115(3)
C28-C282	1.537(6)	C11-N10-H10	108(3)

N10-C11-C1	106.3(4)	C26-C25-C24	113.2(4)
N10-C11-C16	108.7(3)	C26-C25-H25A	108.9
C1-C11-C16	109.3(4)	C24-C25-H25A	108.9
N10-C11-C12	111.7(4)	C26-C25-H25B	108.9
C1-C11-C12	111.1(3)	C24-C25-H25B	108.9
C16-C11-C12	109.5(4)	H25A-C25-H25B	107.7
C13-C12-C11	115.7(4)	C21-C26-C25	111.4(4)
C13-C12-H12A	108.4	C21-C26-H26A	109.3
C11-C12-H12A	108.4	C25-C26-H26A	109.3
C13-C12-H12B	108.4	C21-C26-H26B	109.3
C11-C12-H12B	108.4	C25-C26-H26B	109.3
H12A-C12-H12B	107.4	H26A-C26-H26B	108.0
C12-C13-C14	112.7(4)	C281-C28-C282	108.8(5)
C12-C13-H13A	109.1	C281-C28-C283	109.6(5)
C14-C13-H13A	109.1	C282-C28-C283	106.4(4)
C12-C13-H13B	109.1	C281-C28-C24	112.3(4)
C14-C13-H13B	109.1	C282-C28-C24	110.3(4)
H13A-C13-H13B	107.8	C283-C28-C24	109.3(4)
C13-C14-C15	108.2(4)	N2-C31-C36	111.3(3)
C13-C14-C18	113.9(4)	N2-C31-C32	110.7(4)
C15-C14-C18	115.6(4)	C36-C31-C32	111.4(4)
C13-C14-H14	106.1	N2-C31-H31	107.8
C15-C14-H14	106.1	C36-C31-H31	107.8
C18-C14-H14	106.1	C32-C31-H31	107.8
C16-C15-C14	111.7(4)	C31-C32-C33	109.8(4)
C16-C15-H15A	109.3	C31-C32-H32A	109.7
C14-C15-H15A	109.3	C33-C32-H32A	109.7
C16-C15-H15B	109.3	C31-C32-H32B	109.7
C14-C15-H15B	109.3	C33-C32-H32B	109.7
H15A-C15-H15B	107.9	H32A-C32-H32B	108.2
C15-C16-C11	114.1(4)	C34-C33-C32	111.3(4)
C15-C16-H16A	108.7	C34-C33-H33A	109.4
C11-C16-H16A	108.7	C32-C33-H33A	109.4
C15-C16-H16B	108.7	C34-C33-H33B	109.4
C11-C16-H16B	108.7	C32-C33-H33B	109.4
H16A-C16-H16B	107.6	H33A-C33-H33B	108.0
C182-C18-C181	107.8(5)	C33-C34-C35	112.3(4)
C182-C18-C183	108.7(5)	C33-C34-H34A	109.1
C181-C18-C183	107.8(5)	C35-C34-H34A	109.1
C182-C18-C14	110.7(4)	C33-C34-H34B	109.1
C181-C18-C14	111.8(4)	C35-C34-H34B	109.1
C183-C18-C14	110.0(4)	H34A-C34-H34B	107.9
C21-N20-N10	117.4(4)	C34-C35-C36	111.0(4)
N20-C21-C26	118.1(4)	C34-C35-H35A	109.4
N20-C21-C22	128.1(4)	C36-C35-H35A	109.4
C26-C21-C22	113.6(4)	C34-C35-H35B	109.4
C21-C22-C23	109.8(4)	C36-C35-H35B	109.4
C21-C22-H22A	109.7	H35A-C35-H35B	108.0
C23-C22-H22A	109.7	C31-C36-C35	110.6(4)
C21-C22-H22B	109.7	C31-C36-H36A	109.5
C23-C22-H22B	109.7	C35-C36-H36A	109.5
H22A-C22-H22B	108.2	C31-C36-H36B	109.5
C24-C23-C22	114.6(4)	C35-C36-H36B	109.5
C24-C23-H23A	108.6	H36A-C36-H36B	108.1
C22-C23-H23A	108.6	C18-C181-H18A	109.5
C24-C23-H23B	108.6	C18-C181-H18B	109.5
C22-C23-H23B	108.6	H18A-C181-H18B	109.5
H23A-C23-H23B	107.6	C18-C181-H18C	109.5
C23-C24-C25	108.6(4)	H18A-C181-H18C	109.5
C23-C24-C28	115.1(4)	H18B-C181-H18C	109.5
C25-C24-C28	114.6(4)	C18-C182-H18D	109.5
C23-C24-H24	105.9	C18-C182-H18E	109.5
C25-C24-H24	105.9	H18D-C182-H18E	109.5
C28-C24-H24	105.9	C18-C182-H18F	109.5

H18D-C182-H18F	109.5
H18E-C182-H18F	109.5
C18-C183-H18G	109.5
C18-C183-H18H	109.5
H18G-C183-H18H	109.5
C18-C183-H18I	109.5
H18G-C183-H18I	109.5
H18H-C183-H18I	109.5
C28-C281-H28A	109.5
C28-C281-H28B	109.5
H28A-C281-H28B	109.5
C28-C281-H28C	109.5
H28A-C281-H28C	109.5
H28B-C281-H28C	109.5
C28-C282-H28D	109.5
C28-C282-H28E	109.5
H28D-C282-H28E	109.5
C28-C282-H28F	109.5
H28D-C282-H28F	109.5
H28E-C282-H28F	109.5
C28-C283-H28G	109.5
C28-C283-H28H	109.5
H28G-C283-H28H	109.5
C28-C283-H28I	109.5
H28G-C283-H28I	109.5
H28H-C283-H28I	109.5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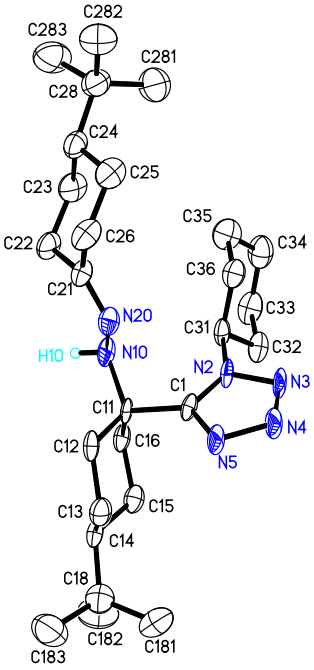
C18
C182
C183
C181


C282
C283
C281
C28
C24
C25
C23
C22
C26
C35
C34
C36
C33
C21
C31
C32
N20
H10
N10
N2
N3
C1
C11
N4
C12
C16
N5
C13
C15
C14
C18
C182
C183
C181

5d: colourless crystal (needle), dimensions 0.420 x 0.050 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.345(2) Å, b=31.355(11) Å, c=14.286(5) Å, alpha=90 deg, beta=99.872(11) deg, gamma=90 deg, V=2799.8(17) $Å^3$, rho=1.083 g/cm^3, T=200(2) K, $Theta_{max}$= 21.260 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.63and a completeness of 61.5% to a resolution of 0.98Å, 11459 reflections measured, 3120 unique (R(int)=0.1049), 1898 observed ($I > 2\sigma(I)$), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using SADABS[1] based on the Laue symmetry of the reciprocal space, mu=0.06mm^{-1}, T_{min}=0.82, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package[2], 302 parameters refined, hydrogen atoms were treated using appropriate riding models, except H10 at N10, which was refined isotropically, goodness of fit 1.01 for observed reflections, final residual values R1(F)=0.074, $wR(F^2)$=0.159 for observed reflections, residual electron density -0.33 to 0.32 $eÅ^{-3}$. CCDC 1030931 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Lit. 1: (program SADABS 2012/1 for absorption correction) G. M. Sheldrick, Bruker Analytical X-ray-Division, Madison, Wisconsin 2012

Lit. 2: (software package SHELXTL 2013/4 for structure solution and refinement) Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Lit. APEX, APEX2, SMART, SAINT, SAINT-Plus: Bruker (2007). "Program name(s)". Bruker AXS Inc., Madison, Wisconsin, USA.

Supporting information

Efficient and Stereoselective Synthesis of α-Hydrazino Tetrazoles through a Pseudo Five-component Domino Reaction

Ali Nikbakht, [a] Sorour Ramezanpour, [a] Saeed Balalaie,* [a,b] Frank Rominger [c]

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[c]Organisch-Chemisches Institut der Universitaet Heidelberg, Im Neuenheimer Feld 270, 69120 Heidelberg, Germany

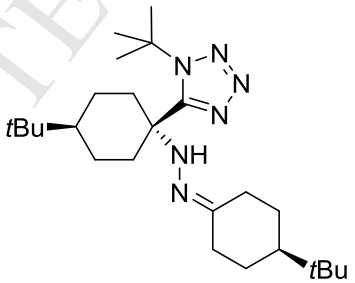
General information

High resolution mass spectra were recorded on Mass-ESI-POS (FT-ICR) spectrometer.

General procedure for the synthesis of α-hydrazino tetrazoles 5a-h:

To a solution of hydrazine hydrate **1** (1 mmol), cyclic ketone **2a-f** (2 mmol) in 5 mL ethanol was added. The mixture was stirred for 30 min then isocyanide **3a-b** (1.2 mmol) and trimethylsilyl azide **4** (1 mmol) was added to the mixture. The mixture was stirred for 24h at ambient temperature. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 1:3), the precipitate was filtered off and then washed with methanol.

Spectroscopic data for compounds **5a-h:**

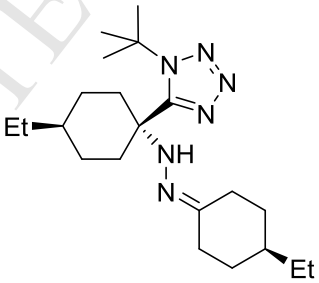


5a (89%)

1-(*tert*-butyl)-5- ((1r, 4S)-4- (*tert*-butyl)-1-(2 -((S,*E*)-4 -(*tert*-butyl)cyclohexylidene) hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5a**): Colorless powder; 383 mg(89%), mp: 209-211°C, IR υ_{max} (KBr, cm^{-1}): 3258, 1639; ^{1}H NMR (300 MHz, $CDCl_3$): 0.82 (*s*, 9H, $3CH_3$, *t*-bu), 0.86(*s*, 9H, $3CH_3$, *t*-bu), 1.07-1.72 (*m*, 8H, $4CH_2$, $H_{Cyclohexyl}$), 1.78 (*s*, 9H, $3CH_3$, *t*-bu), 1.82-2.01 (*m*, 4H, $H_{Cyclohexyl}$), 2.28-2.50 (*m*, 2H, $H_{Cyclohexyl}$), 2.60-2.81 (*m*, 4H, $H_{Cyclohexyl}$), 4.36(*s*, 1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): 23.9, 24.2, 25.1, 26.3, 27.4, 27.5, 27.7, 31.2, 32.3, 32.4, 34.8, 37.9, 38.5, 47.4, 47.6, 59.3, 64.4, 152.6, 157.8; HR-MS (ESI): calc. for $C_{25}H_{47}N_6$ $[M+H]^+$ 431.38567, found 431.38567; calc. for

$C_{25}H_{46}N_6Na$ $[M+Na]^+$ 453.36767, found 453.36762; calc. for $C_{25}H_{46}N_6K$ $[M+K]^+$ 469.34161, found 469.34155.

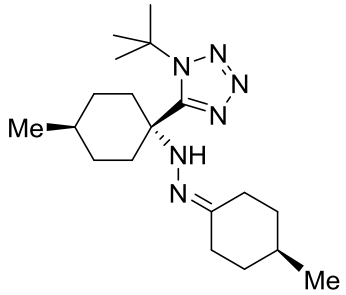
colourless crystal (plate), dimensions 0.370 x 0.230 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.2048(7) Å, b=29.336(3) Å, c=14.4809(17) Å, alpha=90 deg, beta=98.484(4) deg, gamma=90 deg, V=2607.0(5) $Å^3$, rho=1.097 g/cm^3, T=200(2) K, $Theta_{max}$= 26.022 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.15and a completeness of 100.0% to a resolution of 0.81Å, 16371 reflections measured, 5139 unique (R(int)=0.0805), 3027 observed (I > 2σ(I)), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using SADABS based on the Laue symmetry of the reciprocal space, mu=0.07mm^{-1}, T_{min}=0.90, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package, 285 parameters refined, hydrogen atoms were treated using appropriate riding models, except H21 at N21, which was refined isotropically, goodness of fit 0.99 for observed reflections, final residual values R1(F)=0.057, $wR(F^2)$=0.109 for observed reflections, residual electron density -0.22 to 0.21 $eÅ^{-3}$. CCDC 103930 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



5b (54%)

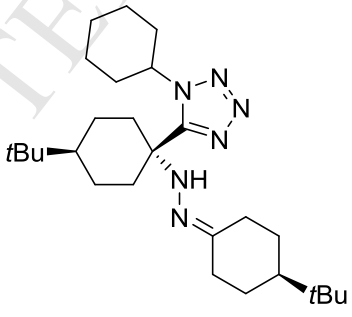
1-(*tert*-butyl)-5-((1*r*,4S)-4-ethyl-1-(2- ((S,*E*)-4-ethyl cyclohexylidene) hydrazinyl) cyclohexyl) -1*H*-tetrazole (**5b**): Waxy liquid; 202 mg(54%), IR υ_{max} (KBr, cm^{-1}): 3362, 1713; ^1H NMR (300 MHz, $CDCl_3$): 0.85 (*t*, J= 7.0 Hz, 3H, CH_3), 0.91 (*t*, *J*=7.0 Hz, 3H, CH_3), 0.94-1.03 (*m*, 2H, $H_{Cyclohexyl}$), 1.22-1.37 (*m*, 7H, $H_{Cyclohexyl}$), 1.61-1.88 (*m*, 8H, HCyclohexyl), 1.78 (*s*, 9H, $3CH_3$, *t*-bu), 2.00-2.23(*m*, 2H, $H_{Cyclohexyl}$), 2.53-2.67 (*m*, 3H, $H_{Cyclohexyl}$), 4.54 (*brs*, 1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): 11.4, 11.6, 11.7, 24.4, 28.1, 28.2, 28.9, 31.2, 31.3, 31.5, 32.8, 34.4, 35.6, 35.9, 37.5, 38.6, 59.4, 64.3, 152.7, 158.4; HR-MS (ESI): calc. for $C_{21}H_{39}N_6$ $[M+H]^+$

375.33606, found 375.33565; calc. for $C_{21}H_{38}N_6Na$ $[M+Na]^+$ 397.30385, found 397.30340; calc. for $C_{21}H_{38}N_6K$ $[M+K]^+$ 413.27867, found 413.27853.



5c (62%)

1-(*tert*-butyl)-5-((1*r*,4S)- 4-methyl-1- (2 -((S, *E*)-4-methylcyclohexylidene) hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5c**): Colorless solid; 215 mg(62%), mp: 120-123 ^oC, IR υ_{max} (KBr, cm^{-1}): 3255, 1632; ^1H NMR (300 MHz, $CDCl_3$): 0.89 (*d*, 6H, *J* = 6.3 Hz, $2CH_3$), 0.92-1.70 (*m*, 10H, $H_{cyclohexyl}$), 1.74 (*s*, 9H, *t*-bu), 1.76-2.66 (*m*, 8H, $H_{cyclohexyl}$), 4.62 (*s*,1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): 20.9, 21.6, 24.3, 30.3, 30.5, 30.6, 31.0, 31.2, 32.0, 33.8, 34.5, 35.2, 35.6, 59.1, 64.3, 76.6, 152.2, 158.5; HR-MS (ESI): HR-MS (ESI): calc. for $C_{19}H_{35}N_6$ $[M+H]^+$ 347.24282, found 347.24282.

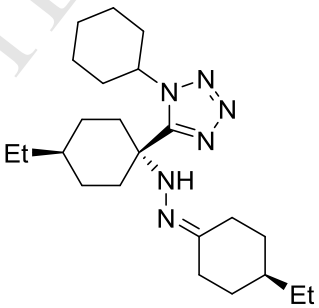


5d (92%)

5-((1r,4S)-4-(*tert*-butyl)-1-(2-((S,*E*)-4-(*tert*-butyl)cyclohexylidene)hydrazinyl) cyclohexyl)-1-cyclohexyl-1*H*-tetrazole (**5d**): Colorless solid; 420 mg(92%), mp: 239-241 ^oC, IR υ_{max} (KBr, cm^{-1}): 3253, 1642; ^1H NMR (300 MHz, $CDCl_3$): 0.77 (*s*, 9H, $3CH_3$, *t*-bu), 0.84(*s*, 9H, 3CH*3*, *t*-bu), 0.88-1.50 (*m*, 10H, $H_{Cyclohexyl}$), 1.53-1.82 (*m*, 6H, $H_{Cyclohexyl}$), 1.83-2.24 (*m*, 9H, $H_{Cyclohexyl}$), 2.28-2.79 (*m*, 3H, $H_{Cyclohexyl}$), 4.49 (*s*, 1H, NH), 5.11 (*m*, 1H, CNH); ^{13}C NMR (75 MHz, $CDCl_3$): 21.6, 22.0, 24.8, 25.0, 25.7, 26.5, 27.3, 27.7, 32.3, 32.5, 33.1, 33.2, 34.2, 34.8, 46.4, 47.4, 56.8,

58.9, 153.4, 158.5; HR-MS (ESI): calc for $C_{27}H_{49}N_6$ $[M+H]^+$ 457.40144, found 457.40132; calc for $C_{27}H_{49}N_6Na$ $[M+Na]^+$ 479.38340, found 479.38327; calc. for $C_{27}H_{48}KN_6$ $[M+K]^+$ 495.35734, found 495.35720.

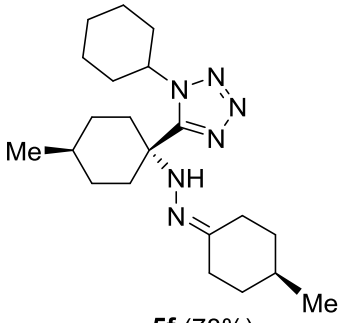
colourless crystal (needle), dimensions 0.420 x 0.050 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.345(2) Å, b=31.355(11) Å, c=14.286(5) Å, alpha=90 deg, beta=99.872(11) deg, gamma=90 deg, V=2799.8(17) $Å^3$, rho=1.083 g/cm^3, T=200(2) K, $Theta_{max}$= 21.260 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.63and a completeness of 61.5% to a resolution of 0.98Å, 11459 reflections measured, 3120 unique (R(int)=0.1049), 1898 observed ($I > 2\sigma(I)$), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using SADABS[1] based on the Laue symmetry of the reciprocal space, mu=0.06mm^{-1}, T_{min}=0.82, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package[2], 302 parameters refined, hydrogen atoms were treated using appropriate riding models, except H10 at N10, which was refined isotropically, goodness of fit 1.01 for observed reflections, final residual values R1(F)=0.074, $wR(F^2)$=0.159 for observed reflections, residual electron density -0.33 to 0.32 $eÅ^{-3}$. CCDC 103931 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



5e (71%)

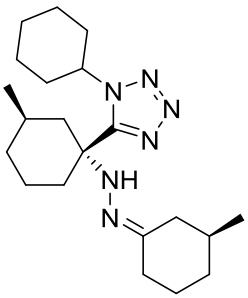
1-cyclohexyl-5- ((1*r*,4S)-4-ethyl-1 -(2 -((S,*E*)-4-ethylcyclohexylidene)hydrazinyl) cyclohexyl)-1H-tetrazole (**5e**): Colorless solid; 216 mg(71%), mp: 129-130 ^oC, IR υ_{max} (KBr, cm^{-1}): 3244, 1739, 1635; ^1H NMR (300 MHz, $CDCl_3$): 0.84 (*t*, 3H, *J* = 7.2 Hz, CH_3), 0.97-1.02 (*m*, 3H, $H_{Cyclohexyl}$), 1.78-1.38 (*m*, 10H, $H_{Cyclohexyl}$), 1.63-2.24 (*m*, 16H, $H_{Cyclohexyl}$), 2.53-2.70 (*m*, 3H, $H_{Cyclohexyl}$), 4.59 (*s*,1H, NH), 5.07-5.14 (*m*, 1H, CNH); ^{13}C NMR (75 MHz, $CDCl_3$): 11.5, 11.6, 24.3, 25.0, 25.6, 28.5, 28.6, 28.8, 31.3, 32.6, 33.2, 34.4, 35.3, 35.7, 38.5, 58.3, 59.1, 153.6.2; HR-MS (ESI): calc. for C23H41N6 $[M+H]^+$ 401.33876, found 401.33872; calc. for $C_{23}H_{40}N_6$

$[M+Na]^+$ 423.31971, found 432.31933; calc. for $C_{23}H_{40}KN_6$ $[M+K]^+$ 439.29467, found 439.29460



5f (78%)

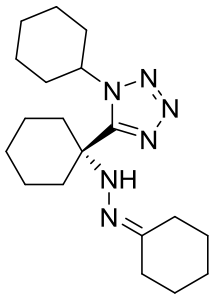
1-cyclohexyl-5 -((1*r*,4S)-4-methyl-1-(2 -((S, *E*)-4-methylcyclohexylidene) hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5f**) Colorless solid; 291 mg(78%), mp: 167-169 ^oC, IR υ_{max} (KBr, cm^{-1}): 3240, 1739, 1631; ^1H NMR (300 MHz, $CDCl_3$): 0.85 (*d*, 3H, *J* = 6.4Hz, CH_3), 0.88(*d*, 3H, *J* = 6.5 Hz, CH_3), 0.93-1.56 (*m*, 10H, $H_{Cyclohexyl}$), 1.58-1.78 (*m*, 8H, $H_{Cyclohexyl}$), 1.79-2.20 (*m*, 8H, $H_{Cyclohexyl}$), 2.45-2.66 (*m*, 2H, $H_{Cyclohexyl}$), 4.6 (*s*, 1H, NH), 5.10 (*m*, 1H, NCH); ^{13}C NMR (75 MHz, $CDCl_3$): 21.4, 21.6, 24.3, 25.0, 25.6, 30.8, 30.9, 31.7, 31.8, 33.1, 33.2, 33.7, 34.5, 35.1, 35.3, 35.8, 56.9, 58.0, 59.1, 153.3, 156.2, HR-MS (ESI): calc. for $C_{21}H_{37}N_6$ $[M+H]^+$ 373.30743, found 373.30742; calc. for $C_{21}H_{36}N_6Na$ $[M+Na]^+$ 395.28939, found 395.28937; calc. for $C_{21}H_{36}KN_6$ $[M+K]^+$ 411.26332, found 411.26330.



5g (65%)

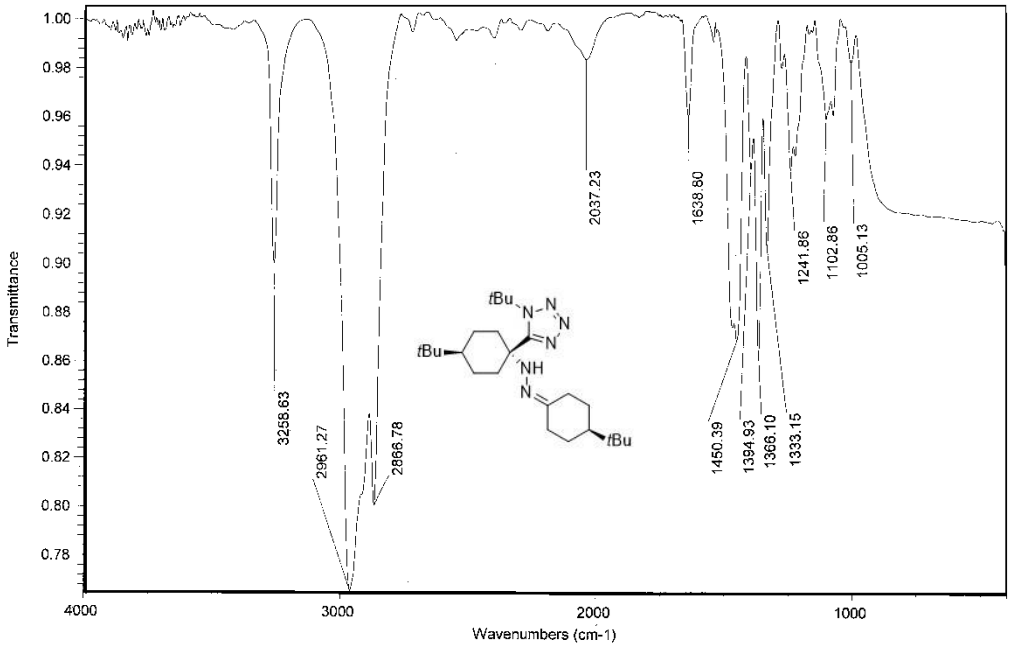
1-cyclohexyl-5- ((1R, 3R)-3-methyl-1- (2-((S, *Z*)-3-methylcyclohexylidene)hydrazinyl) cyclohexyl)-1*H*-tetrazole (**5g**): Colorless solid; 231 mg(65%), mp: 145-147 ^oC, IR υ_{max} (KBr, cm^{-1}): 3238, 1740, 1632; ^1H NMR (300 MHz, $CDCl_3$) 0.85-0.95 (*m*, 6H, CH_3), 1.00-1.55 (*m*, 10H, $H_{Cyclohexyl}$), 1.63-1.79 (*m*, 6H, $H_{Cyclohexyl}$), 1.87-2.21 (*m*, 9H, $H_{Cyclohexyl}$), 2.48-2.65 (*m*,3H,

$H_{Cyclohexyl}$), 4.56(*s*, 1H, NH), 5.05-5.13 (*m*,1H, CHN); ^{13}C NMR (75 MHz, $CDCl_3$): 22.2, 22.4, 22.5, 25.0, 25.6, 25.7, 29.0, 32.3, 33.2, 33.3, 34.1, 34.4, 34.7, 35.9, 44.4, 58.9, 59.1, 59.2, 153.3, 156.1; 156.1; HR-MS (ESI): calc. for $C_{21}H_{37}N_6$ $[M+H]^+$ 373.30745, found 373.30742; calc. for $C_{21}H_{36}N_6Na$ $[M+Na]^+$ 395.28940, found 395.28937; calc. for $C_{21}H_{36}KN_6$ $[M+K]^+$ 411.26334, found 411.26330.

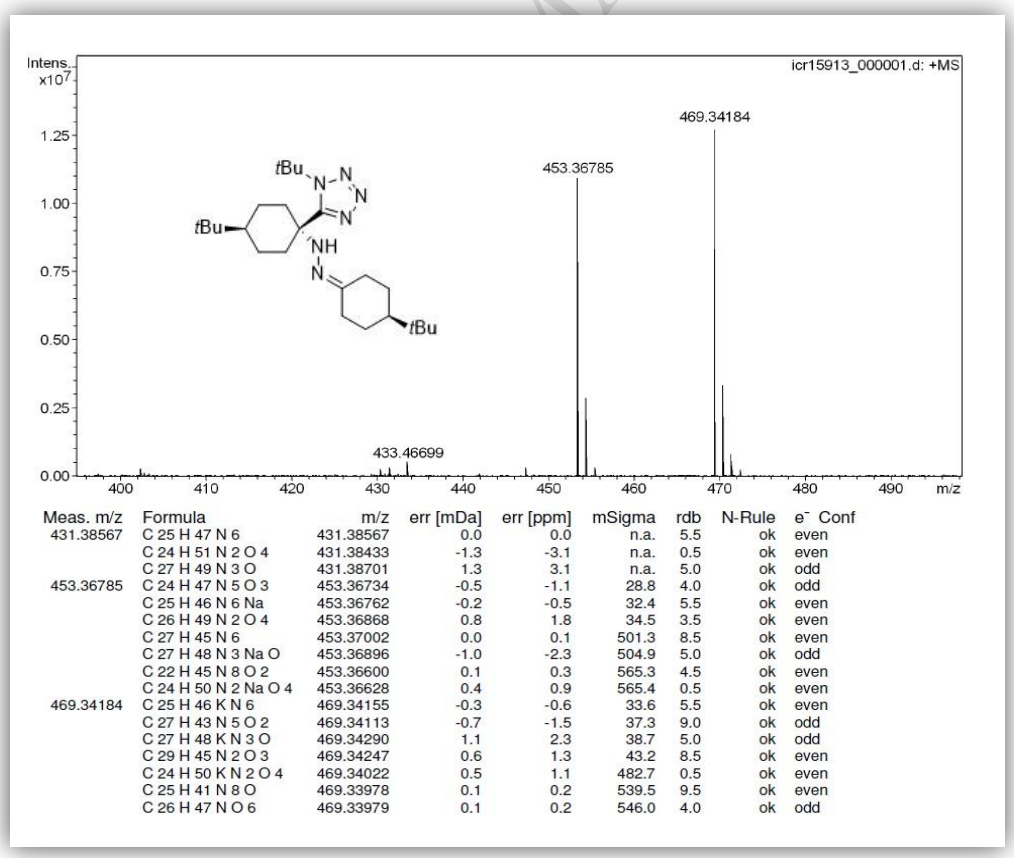


5h (75%)

1-cyclohexyl-5-(1-(2-cyclohexylidenehydrazinyl)cyclohexyl)-1H-tetrazole(**5h**): Colorless solid; 258 mg(75%), mp: 147-150 ^{o}C, IR υ_{max} (KBr, cm^{-1}): 3239, 1631; ^{1}H NMR (300 MHz, $CDCl_3$): 1.29-1.50 (*m*, 5H, $H_{Cyclohexyl}$), 1.50-1.63 (*m*, 9H, $H_{Cyclohexyl}$), 1.67-1.95 (*m*,7H, $H_{Cyclohexyl}$), 1.97-2.19 (*m*, 8H, $4CH_2$, $H_{Cyclohexyl}$), 4.96 (*s*, 1H, NH), 5.11(*m*, 1H, CNH); ^{13}C NMR (75 MHz, CDCl3): 21.6, 24.9, 25.0, 25.1, 25.1, 25.4, 25.7, 25.8, 25.9, 27.0, 33.2, 34.4, 35.2, 57.6, 59.0, 153.4, 157.8; HR-MS (ESI): calc. for $C_{19}H_{33}N_6[M+1]^+$ 345.27618, found 345.27613; calc. for C19H33N6Na$[M+Na]^+$ 367.25813, found 367.25807; calc. for $C_{19}H_{33}KN_6[M+1]^+$ 383.23207, found 383.23200.

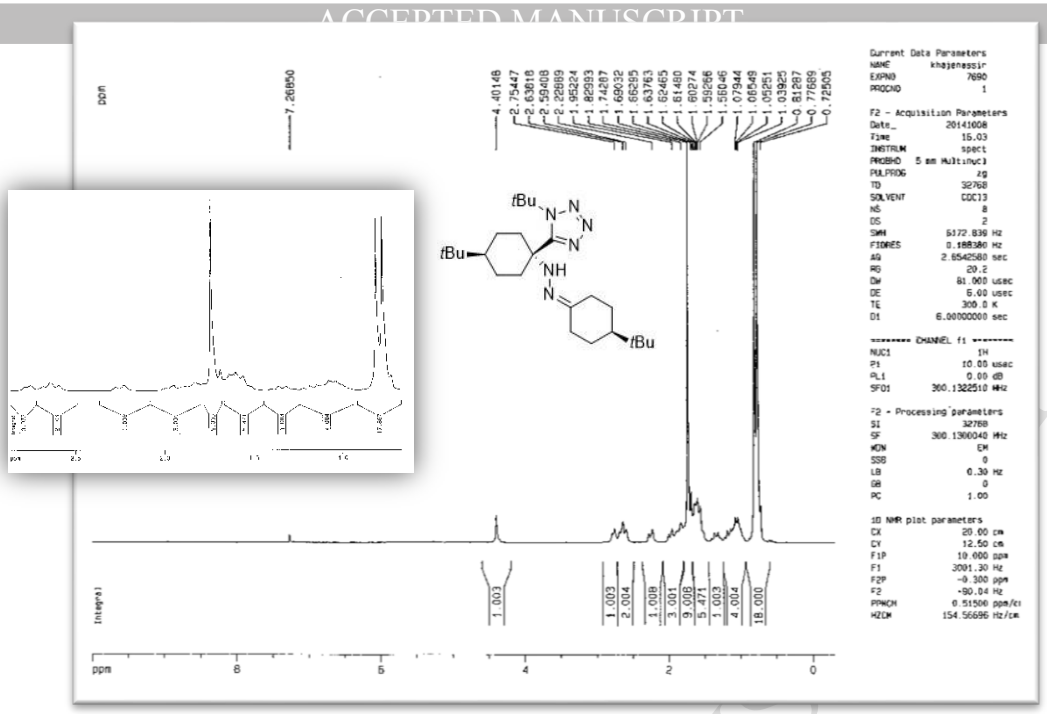


IR (KBr) **(5a)**

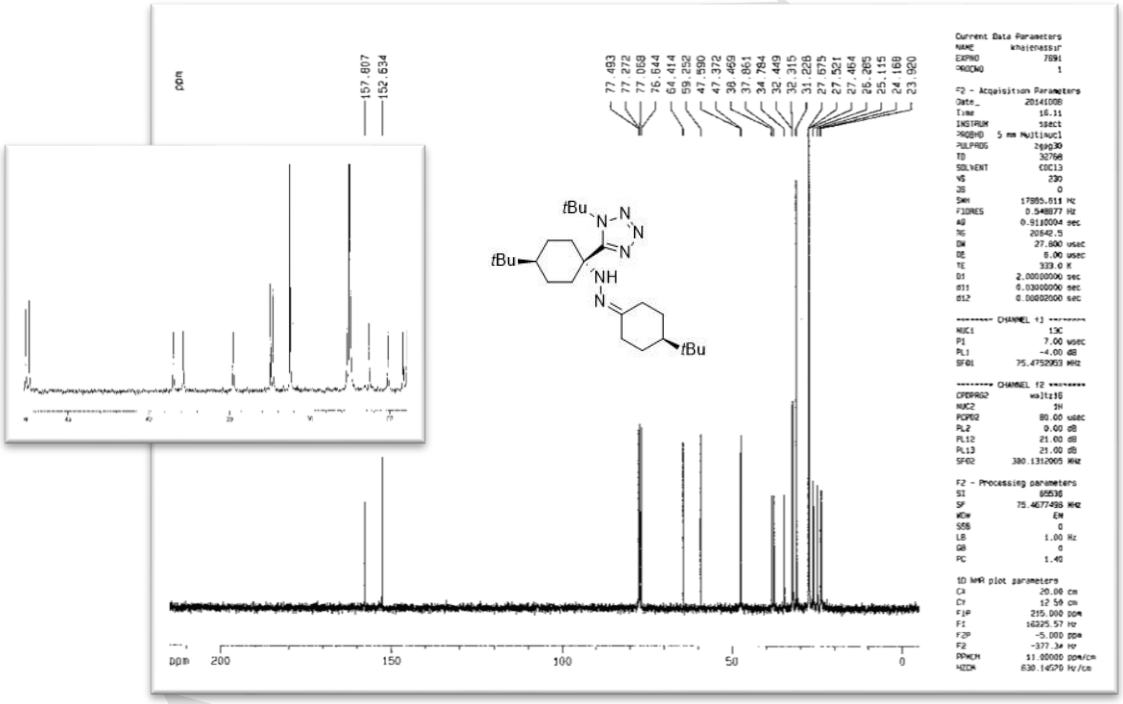


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
431.38567	C 25 H 47 N 6	431.38567	0.0	0.0	n.a.	5.5	ok	even
	C 24 H 51 N 2 O 4	431.38433	-1.3	-3.1	n.a.	0.5	ok	even
	C 27 H 49 N 3 O	431.38701	1.3	3.1	n.a.	5.0	ok	odd
453.36785	C 24 H 47 N 5 O 3	453.36734	-0.5	-1.1	28.8	4.0	ok	odd
	C 25 H 46 N 6 Na	453.36762	-0.2	-0.5	32.4	5.5	ok	even
	C 26 H 49 N 2 O 4	453.36868	0.8	1.8	34.5	3.5	ok	even
	C 27 H 45 N 6	453.37002	0.0	0.1	501.3	8.5	ok	even
	C 27 H 48 N 3 Na O	453.36896	-1.0	-2.3	504.9	5.0	ok	odd
	C 22 H 45 N 8 O 2	453.36600	0.1	0.3	565.3	4.5	ok	even
	C 24 H 50 N 2 Na O 4	453.36628	0.4	0.9	565.4	0.5	ok	even
469.34184	C 25 H 46 K N 6	469.34155	-0.3	-0.6	33.6	5.5	ok	even
	C 27 H 43 N 5 O 2	469.34113	-0.7	-1.5	37.3	9.0	ok	odd
	C 27 H 48 K N 3 O	469.34290	1.1	2.3	38.7	5.0	ok	odd
	C 29 H 45 N 2 O 3	469.34247	0.6	1.3	43.2	8.5	ok	even
	C 24 H 50 K N 2 O 4	469.34022	0.5	1.1	482.7	0.5	ok	even
	C 25 H 41 N 8 O	469.33978	0.1	0.2	539.5	9.5	ok	even
	C 26 H 47 N O 6	469.33979	0.1	0.2	546.0	4.0	ok	odd

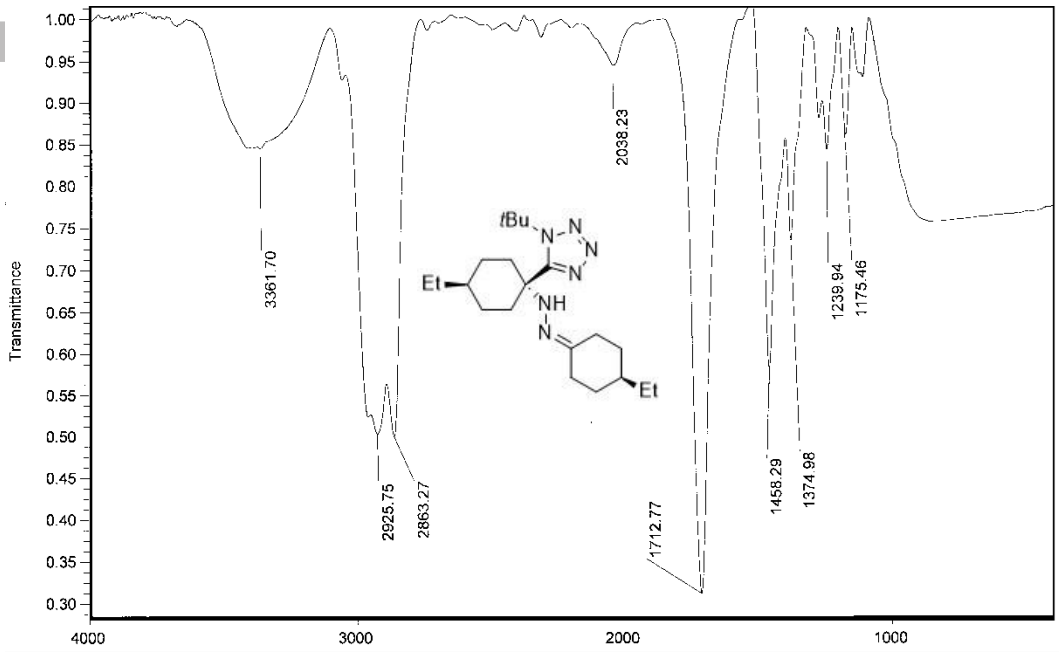
ESI-HRMS **(5a)**



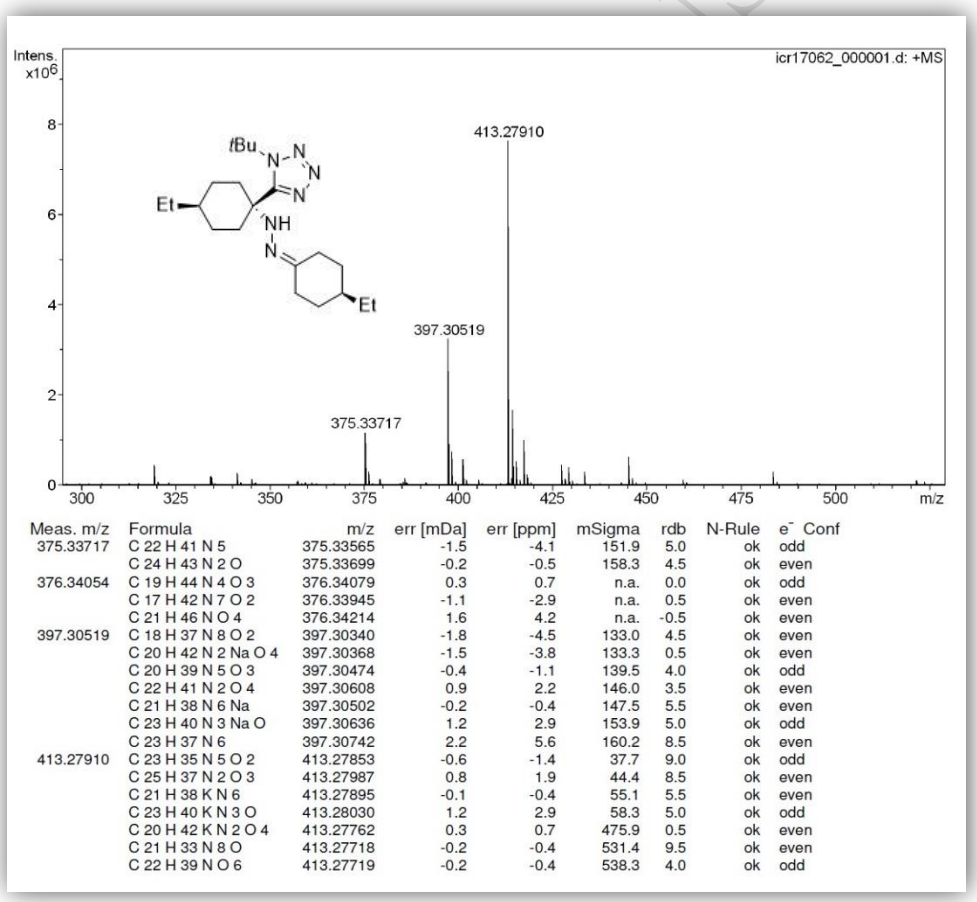
^{1}HNMR (300MHz, $CDCl_3$) **(5a)**



^{13}CNMR (75MHz, $CDCl_3$) **(5a)**

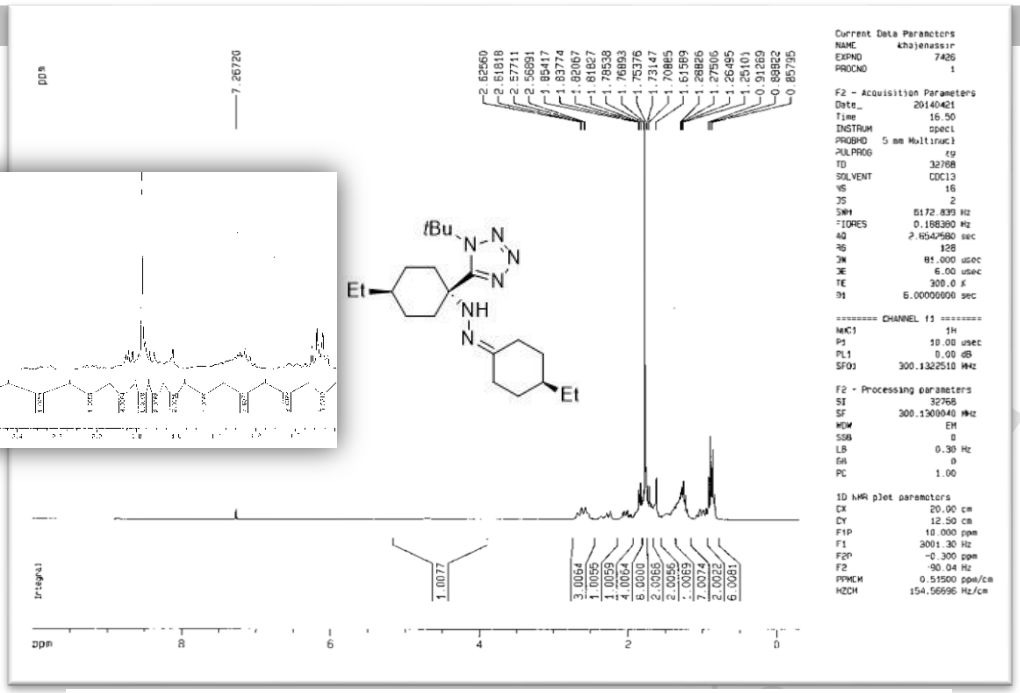


IR (KBr) **(5b)**

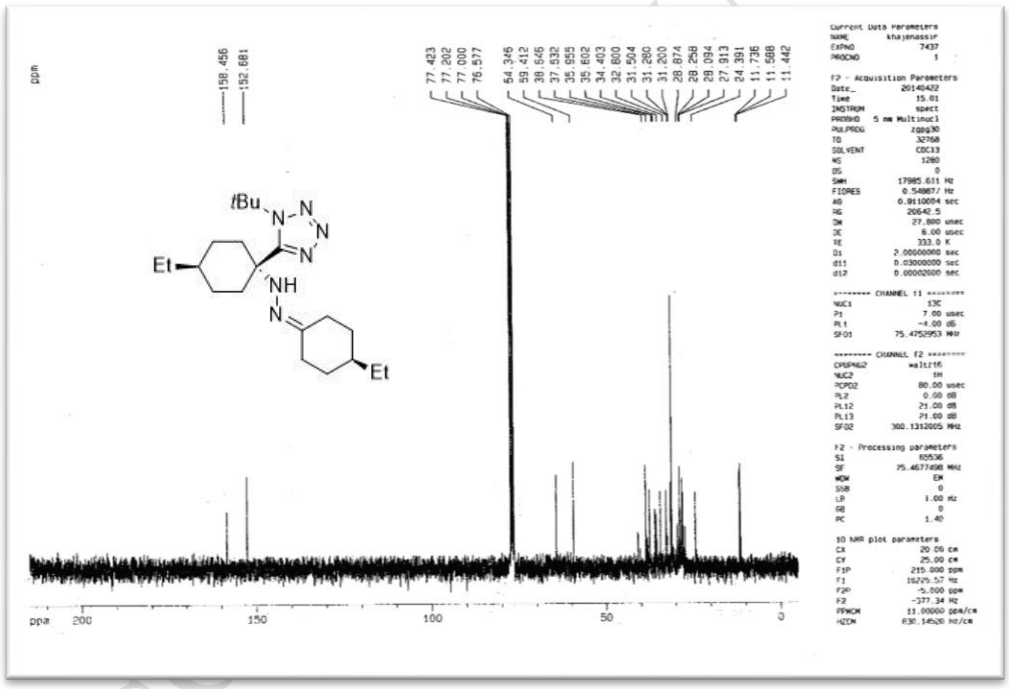


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
375.33717	C 22 H 41 N 5	375.33565	-1.5	-4.1	151.9	5.0	ok	odd
	C 24 H 43 N 2 O	375.33699	-0.2	-0.5	158.3	4.5	ok	even
376.34054	C 19 H 44 N 4 O 3	376.34079	0.3	0.7	n.a.	0.0	ok	odd
	C 17 H 42 N 7 O 2	376.33945	-1.1	-2.9	n.a.	0.5	ok	even
	C 21 H 46 N O 4	376.34214	1.6	4.2	n.a.	-0.5	ok	even
397.30519	C 18 H 37 N 8 O 2	397.30340	-1.8	-4.5	133.0	4.5	ok	even
	C 20 H 42 N 2 Na O 4	397.30368	-1.5	-3.8	133.3	0.5	ok	even
	C 20 H 39 N 5 O 3	397.30474	-0.4	-1.1	139.5	4.0	ok	odd
	C 22 H 41 N 2 O 4	397.30608	0.9	2.2	146.0	3.5	ok	even
	C 21 H 38 N 6 Na	397.30502	-0.2	-0.4	147.5	5.5	ok	even
	C 23 H 40 N 3 Na O	397.30636	1.2	2.9	153.9	5.0	ok	odd
	C 23 H 37 N 6	397.30742	2.2	5.6	160.2	8.5	ok	even
413.27910	C 23 H 35 N 5 O 2	413.27853	-0.6	-1.4	37.7	9.0	ok	odd
	C 25 H 37 N 2 O 3	413.27987	0.8	1.9	44.4	8.5	ok	even
	C 21 H 38 K N 6	413.27895	-0.1	-0.4	55.1	5.5	ok	even
	C 23 H 40 K N 3 O	413.28030	1.2	2.9	58.3	5.0	ok	odd
	C 20 H 42 K N 2 O 4	413.27762	0.3	0.7	475.9	0.5	ok	even
	C 21 H 33 N 8 O	413.27718	-0.2	-0.4	531.4	9.5	ok	even
	C 22 H 39 N O 6	413.27719	-0.2	-0.4	538.3	4.0	ok	odd

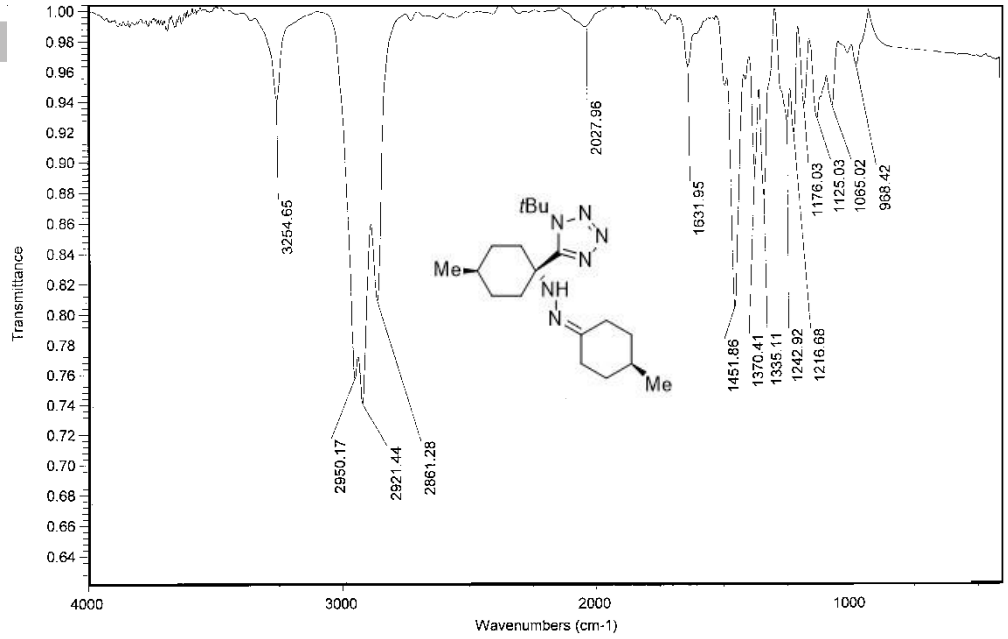
ESI-HRMS **(5b)**



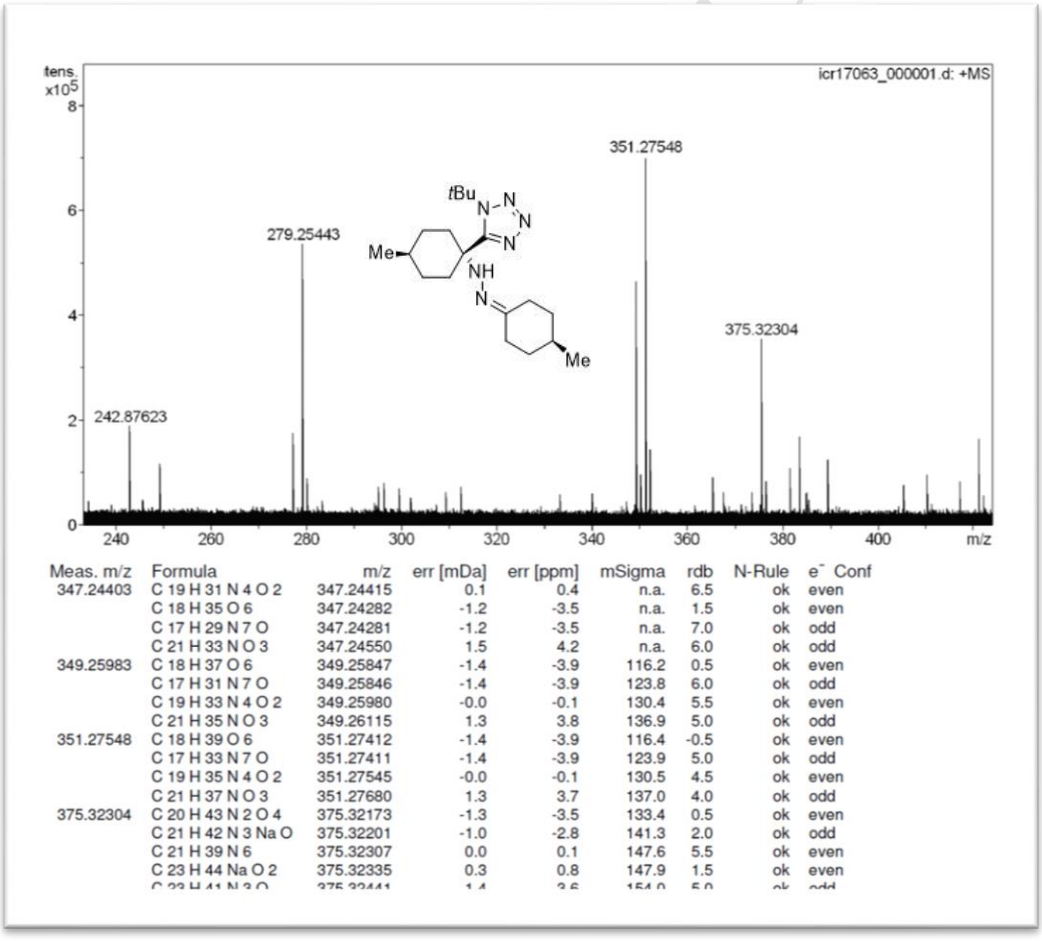
^{1}HNMR (300MHz, $CDCl_3$) **(5b)**



^{13}CNMR (75MHz, $CDCl_3$) **(5b)**

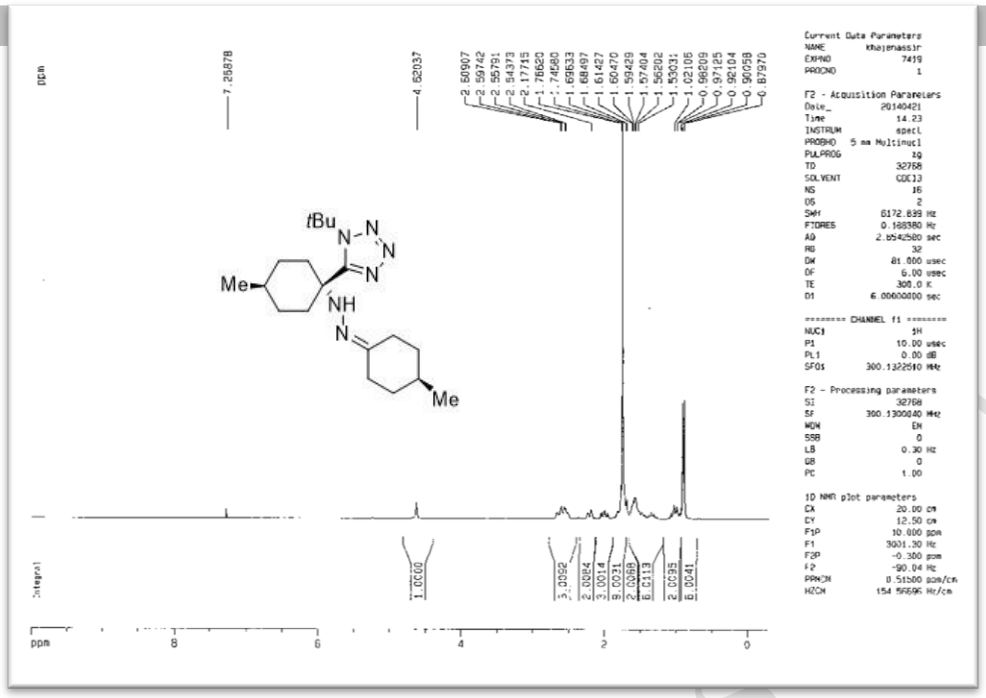


IR (KBr) **(5c)**

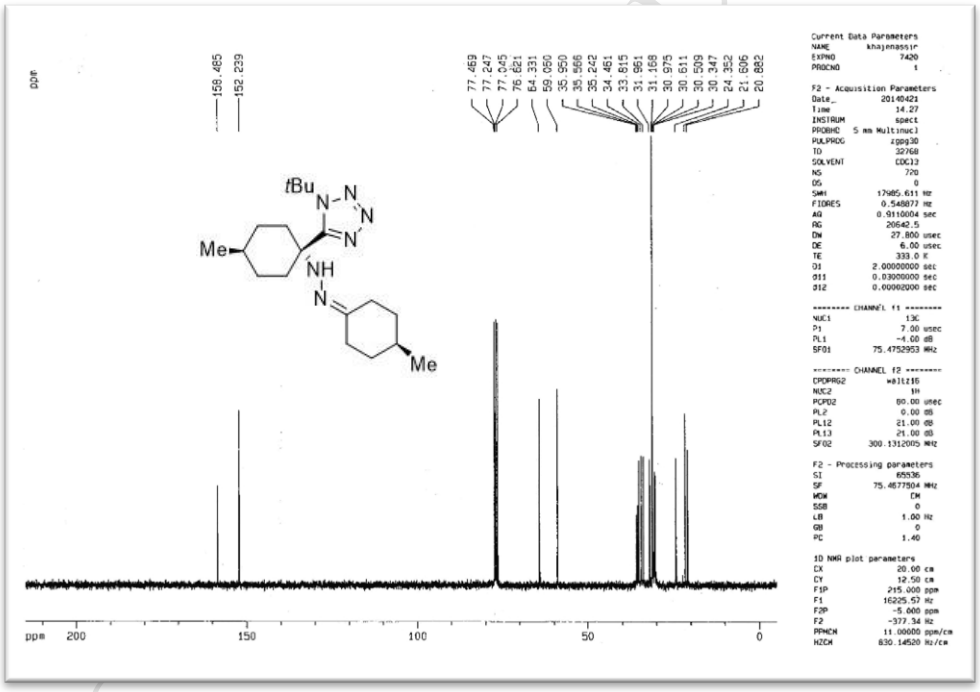


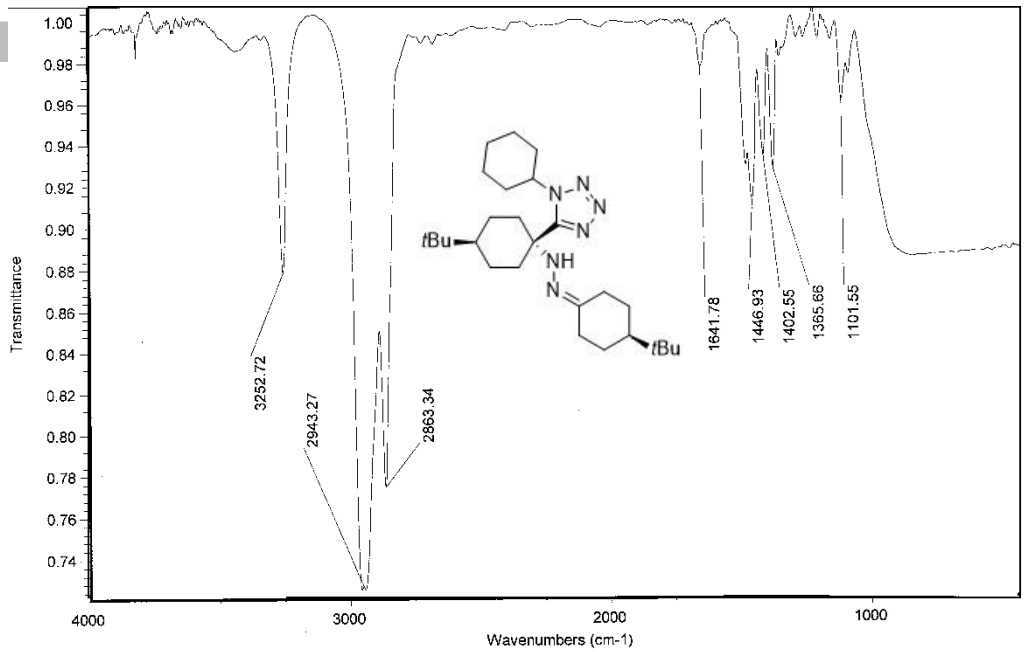
Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
347.24403	C 19 H 31 N 4 O 2	347.24415	0.1	0.4	n.a.	6.5	ok	even
	C 18 H 35 O 6	347.24282	-1.2	-3.5	n.a.	1.5	ok	even
	C 17 H 29 N 7 O	347.24281	-1.2	-3.5	n.a.	7.0	ok	odd
	C 21 H 33 N O 3	347.24550	1.5	4.2	n.a.	6.0	ok	odd
349.25983	C 18 H 37 O 6	349.25847	-1.4	-3.9	116.2	0.5	ok	even
	C 17 H 31 N 7 O	349.25846	-1.4	-3.9	123.8	6.0	ok	odd
	C 19 H 33 N 4 O 2	349.25980	-0.0	-0.1	130.4	5.5	ok	even
	C 21 H 35 N O 3	349.26115	1.3	3.8	136.9	5.0	ok	odd
351.27548	C 18 H 39 O 6	351.27412	-1.4	-3.9	116.4	-0.5	ok	even
	C 17 H 33 N 7 O	351.27411	-1.4	-3.9	123.9	5.0	ok	odd
	C 19 H 35 N 4 O 2	351.27545	-0.0	-0.1	130.5	4.5	ok	even
	C 21 H 37 N O 3	351.27680	1.3	3.7	137.0	4.0	ok	odd
375.32304	C 20 H 43 N 2 O 4	375.32173	-1.3	-3.5	133.4	0.5	ok	even
	C 21 H 42 N 3 Na O	375.32201	-1.0	-2.8	141.3	2.0	ok	odd
	C 21 H 39 N 6	375.32307	0.0	0.1	147.6	5.5	ok	even
	C 23 H 44 Na O 2	375.32335	0.3	0.8	147.9	1.5	ok	even

ESI-HRMS **(5c)**

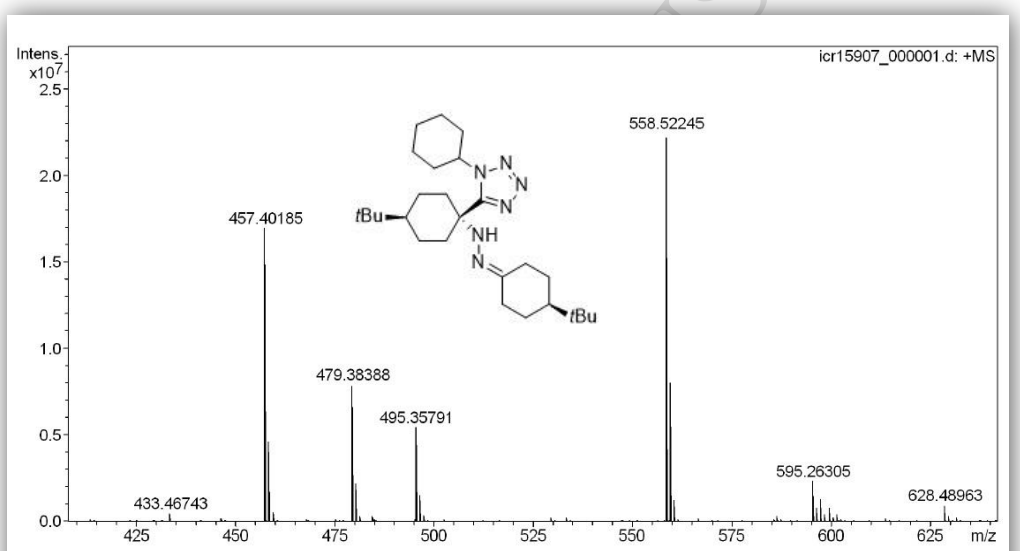


^{1}HNMR (300MHz, $CDCl_3$) **(5c)**



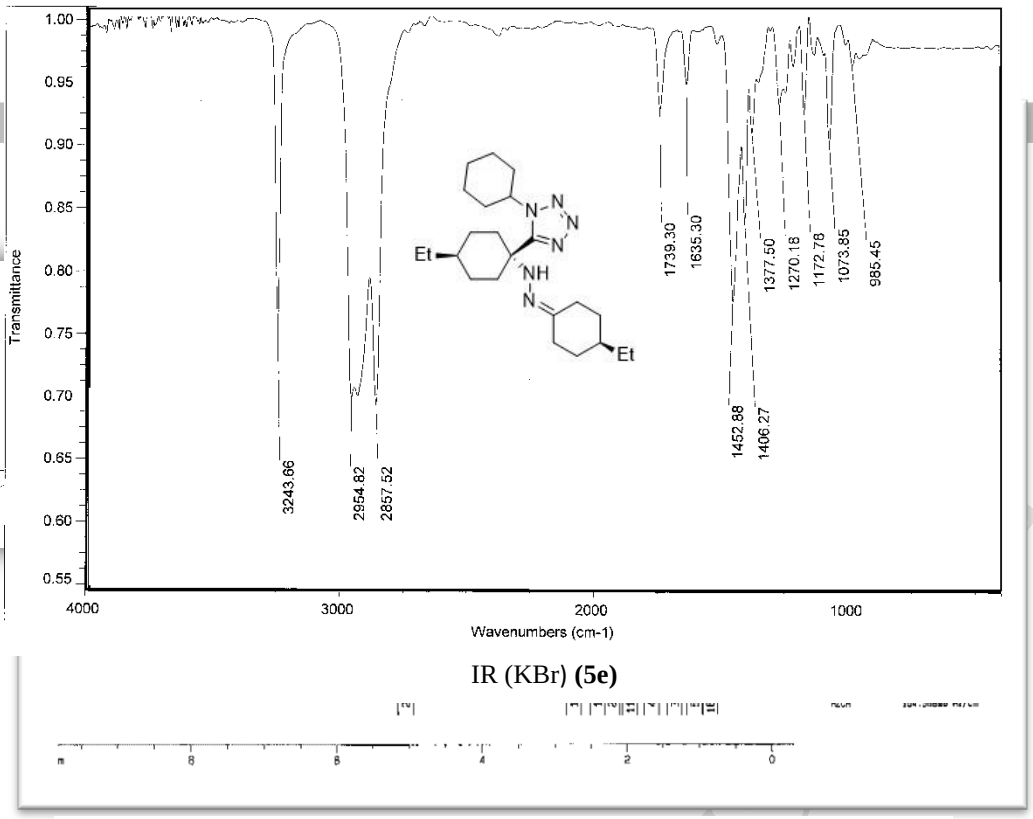


IR (KBr) **(5d)**



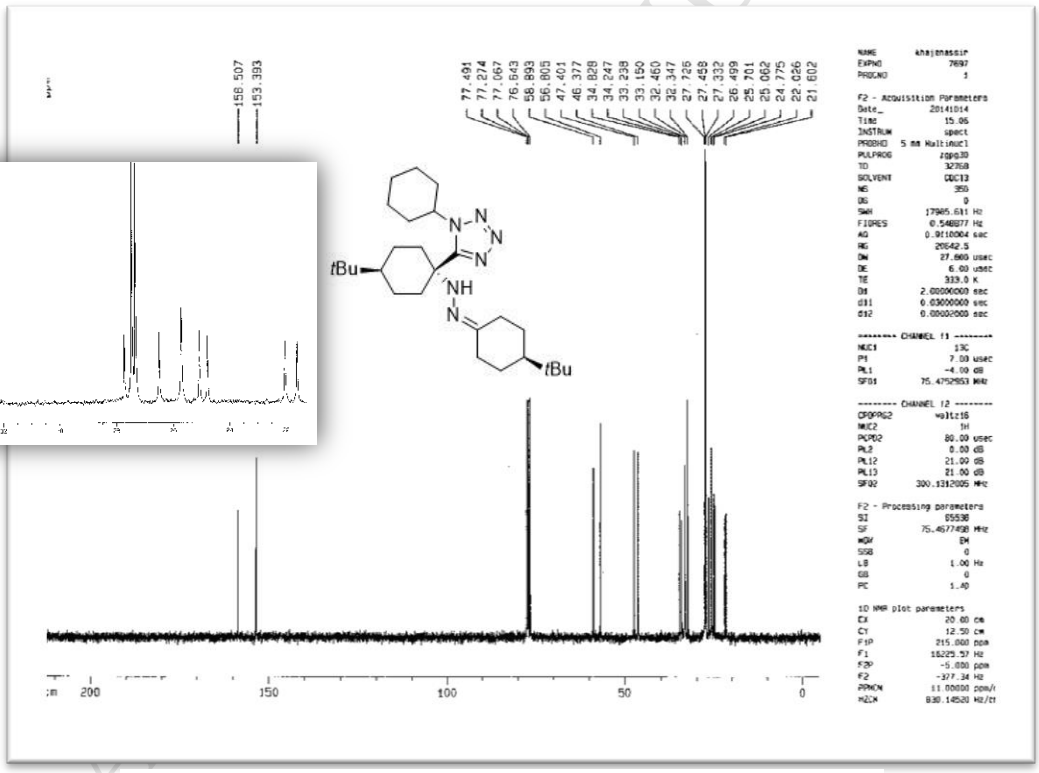
Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
457.40185	C 27 H 49 N 6	457.40132	-0.5	-1.2	23.9	6.5	ok	even
	C 29 H 51 N 3 O	457.40266	0.8	1.8	29.6	6.0	ok	odd
	C 24 H 51 N 5 O 3	457.39864	0.2	0.5	166.0	2.0	ok	odd
	C 31 H 53 O 2	457.40401	-0.1	-0.2	492.4	5.5	ok	even
	C 26 H 53 N 2 O 4	457.39998	0.1	0.3	547.5	1.5	ok	even
479.38388	C 27 H 48 N 6 Na	479.38327	-0.6	-1.3	32.4	6.5	ok	even
	C 26 H 49 N 5 O 3	479.38299	-0.9	-1.9	33.5	5.0	ok	odd
	C 28 H 51 N 2 O 4	479.38433	0.5	0.9	34.3	4.5	ok	even
	C 29 H 50 N 3 Na O	479.38461	0.7	1.5	37.4	6.0	ok	odd
	C 23 H 51 N 4 O 6	479.38031	-1.2	-2.5	157.9	0.5	ok	even
	C 22 H 45 N 11 O	479.38031	-1.2	-2.6	165.3	6.0	ok	odd
	C 24 H 50 N 5 Na O 3	479.38059	-0.9	-2.0	165.9	2.0	ok	odd
	C 31 H 49 N 3 O	479.38701	0.8	1.7	477.3	9.0	ok	odd
	C 29 H 47 N 6	479.38567	-0.5	-1.1	480.8	9.5	ok	even
	C 31 H 52 Na O 2	479.38595	-0.2	-0.5	481.0	5.5	ok	even
	C 25 H 53 N O 7	479.38165	0.1	0.3	483.9	0.0	ok	odd
	C 24 H 47 N 8 O 2	479.38165	0.1	0.2	552.4	5.5	ok	even
	C 26 H 52 N 2 Na O 4	479.38193	0.4	0.8	552.5	1.5	ok	even
495.35791	C 18 H 45 N 11 O 5	495.35997	2.1	4.1	28.3	2.0	ok	odd

ESI-HRMS **(5d)**

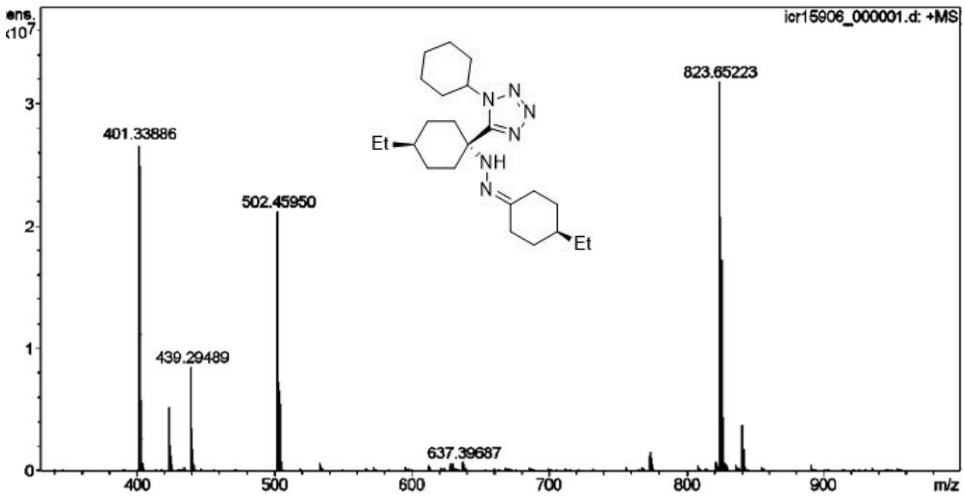


IR (KBr) **(5e)**

^{1}HNMR (300MHz, $CDCl_3$) **(5d)**

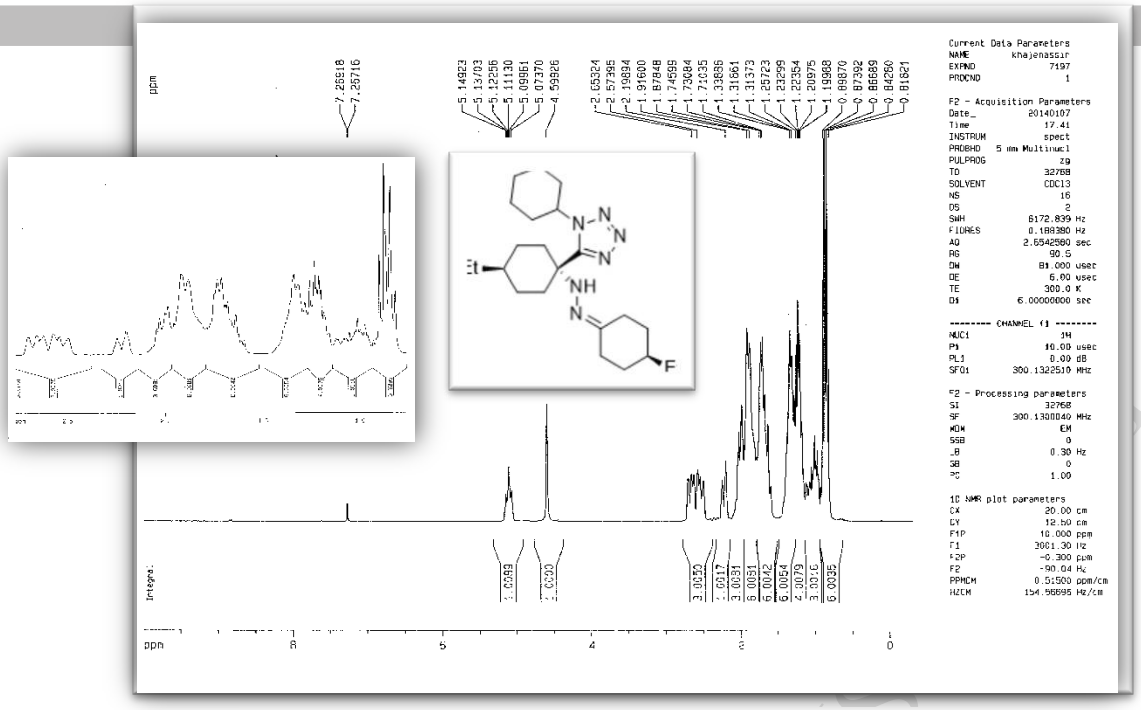


^{13}CNMR (75MHz, $CDCl_3$) **(5d)**

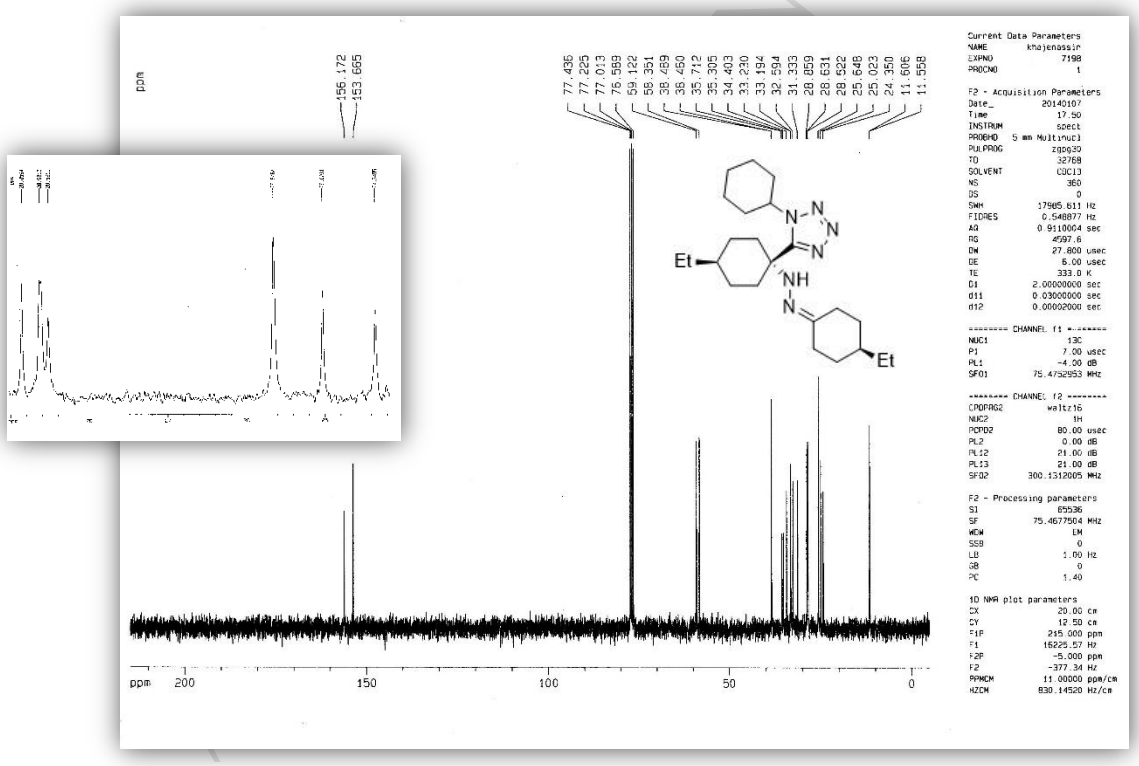


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
401.33886	C 20 H 43 N 5 O 3	401.33604	-0.2	-0.5	139.8	2.0	ok	odd
	C 27 H 45 O 2	401.34141	0.0	0.0	611.6	5.5	ok	even
	C 23 H 41 N 6	401.33872	-0.1	-0.4	648.8	6.5	ok	even
	C 22 H 45 N 2 O 4	401.33738	1.1	2.8	658.3	1.5	ok	even
	C 25 H 43 N 3 O	401.34006	0.0	0.0	709.6	6.0	ok	odd
423.32095	C 20 H 39 N 8 O 2	423.31905	-1.9	-4.5	20.4	5.5	ok	even
	C 22 H 44 N 2 Na O 4	423.31933	-1.6	-3.8	22.9	1.5	ok	even
	C 21 H 45 N O 7	423.31905	-1.9	-4.5	23.8	0.0	ok	odd
	C 22 H 41 N 5 O 3	423.32039	-0.6	-1.3	25.5	5.0	ok	odd
	C 23 H 40 N 6 Na	423.32067	-0.3	-0.7	29.4	6.5	ok	even
	C 24 H 43 N 2 O 4	423.32173	0.8	1.8	31.3	4.5	ok	even
	C 25 H 42 N 3 Na O	423.32201	1.1	2.5	35.1	6.0	ok	odd
	C 27 H 44 Na O 2	423.32335	2.4	5.7	37.0	5.5	ok	even
	C 25 H 39 N 6	423.32307	2.1	5.0	41.0	9.5	ok	even
	C 20 H 42 N 5 Na O 3	423.31799	-3.0	-7.0	139.7	2.0	ok	odd

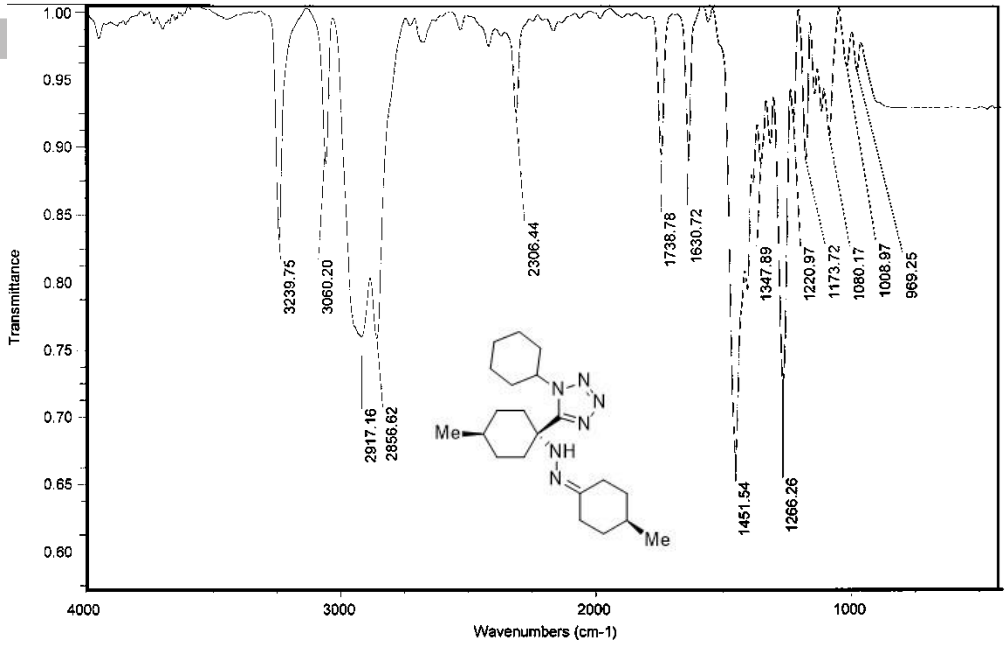
ESI-HRMS **(5e)**



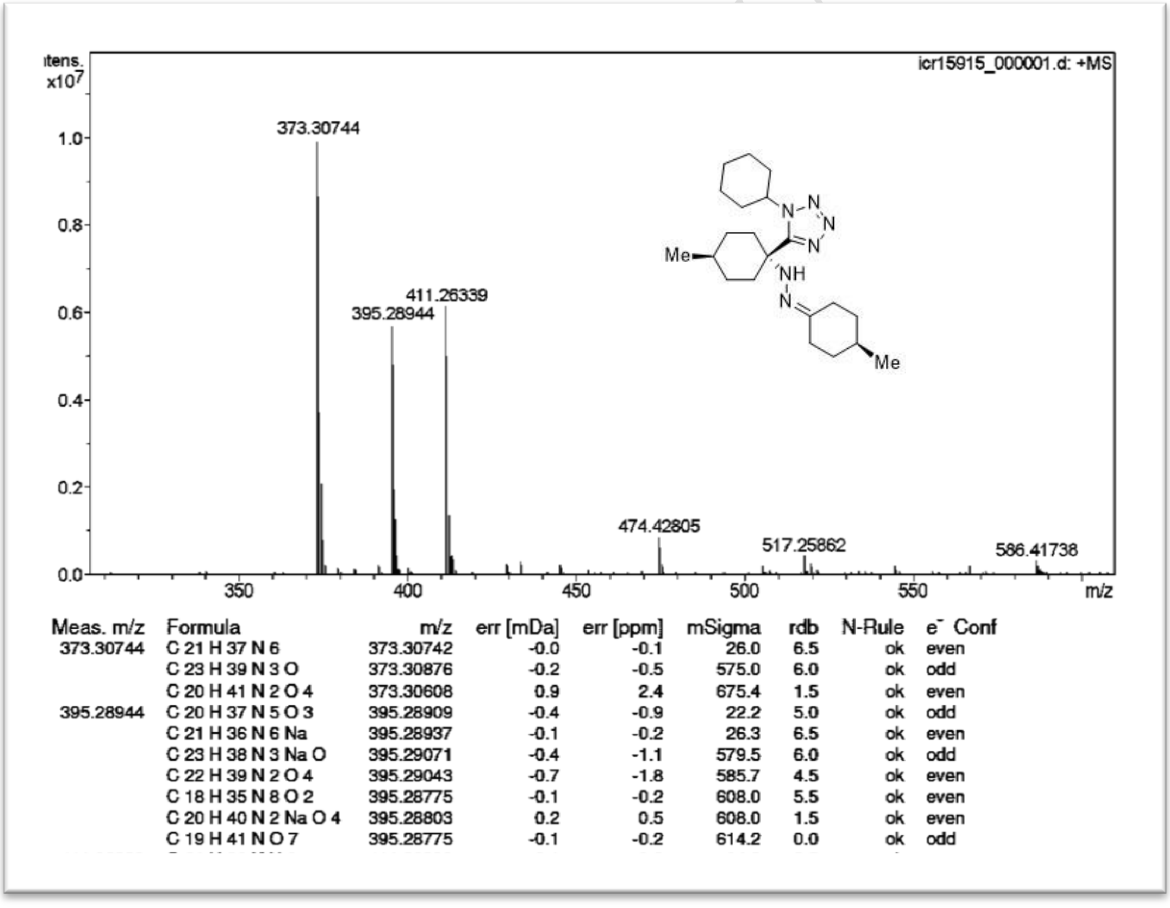
^{1}HNMR (300MHz, $CDCl_3$) **(5e)**



^{13}CNMR (75MHz, $CDCl_3$) **(5e)**

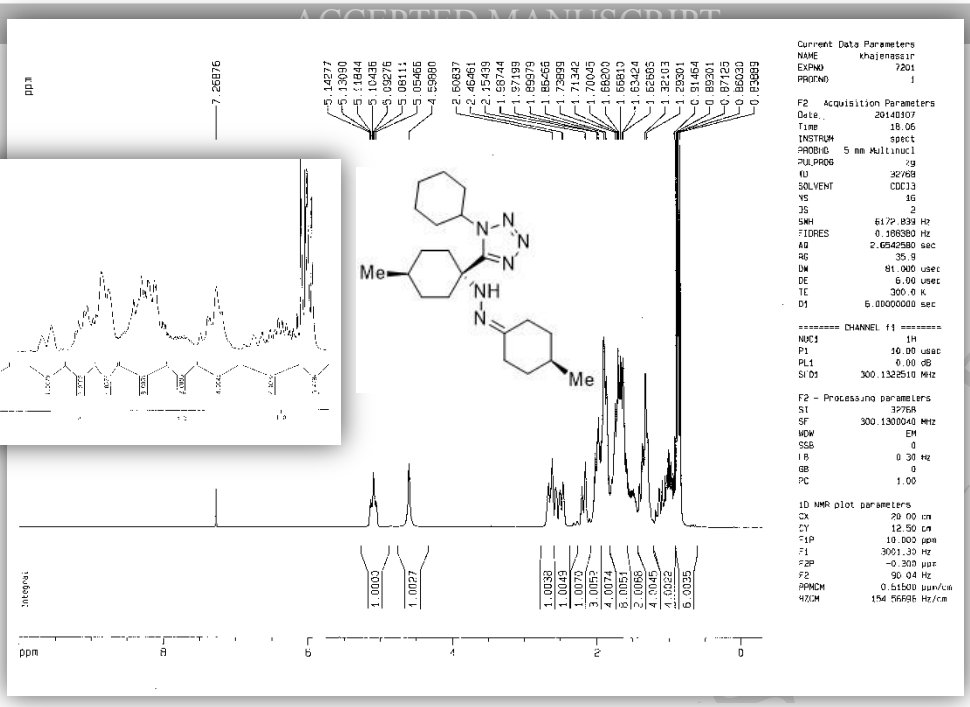


IR (KBr) **(5f)**

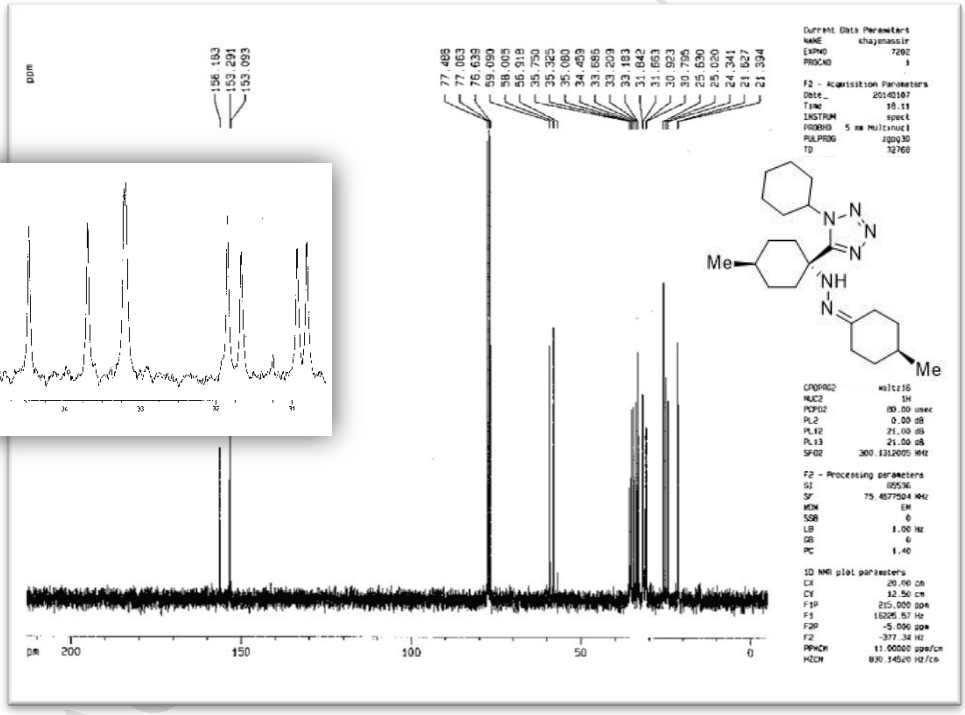


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
373.30744	C 21 H 37 N 6	373.30742	-0.0	-0.1	26.0	6.5	ok	even
	C 23 H 39 N 3 O	373.30876	-0.2	-0.5	575.0	6.0	ok	odd
	C 20 H 41 N 2 O 4	373.30608	0.9	2.4	675.4	1.5	ok	even
395.28944	C 20 H 37 N 5 O 3	395.28909	-0.4	-0.9	22.2	5.0	ok	odd
	C 21 H 36 N 6 Na	395.28937	-0.1	-0.2	26.3	6.5	ok	even
	C 23 H 38 N 3 Na O	395.29071	-0.4	-1.1	579.5	6.0	ok	odd
	C 22 H 39 N 2 O 4	395.29043	-0.7	-1.8	585.7	4.5	ok	even
	C 18 H 35 N 8 O 2	395.28775	-0.1	-0.2	608.0	5.5	ok	even
	C 20 H 40 N 2 Na O 4	395.28803	0.2	0.5	608.0	1.5	ok	even
	C 19 H 41 N O 7	395.28775	-0.1	-0.2	614.2	0.0	ok	odd

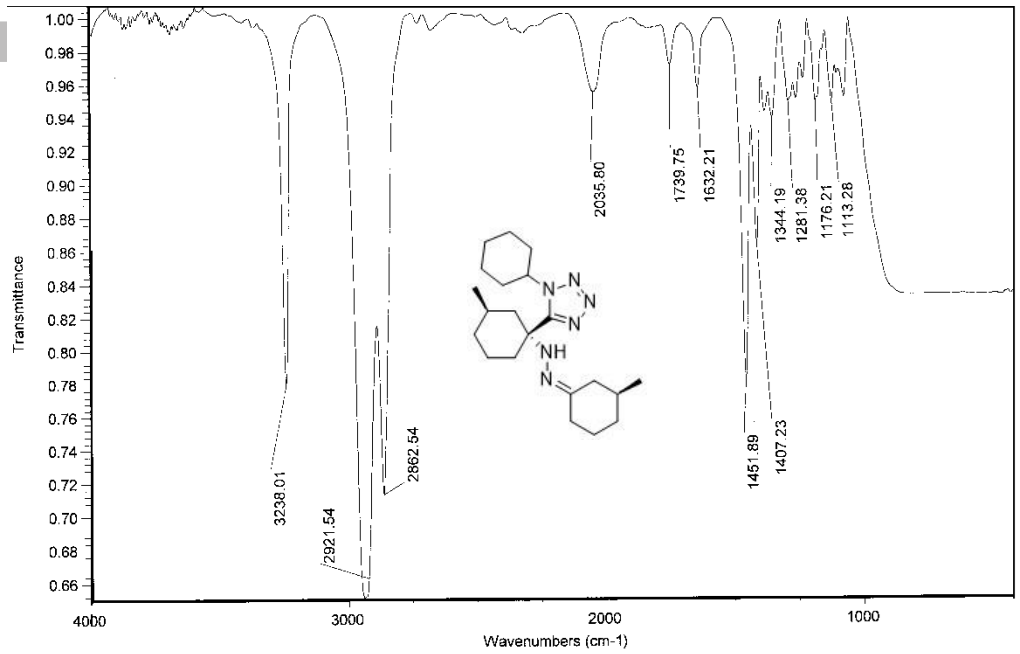
ESI-HRMS **(5f)**



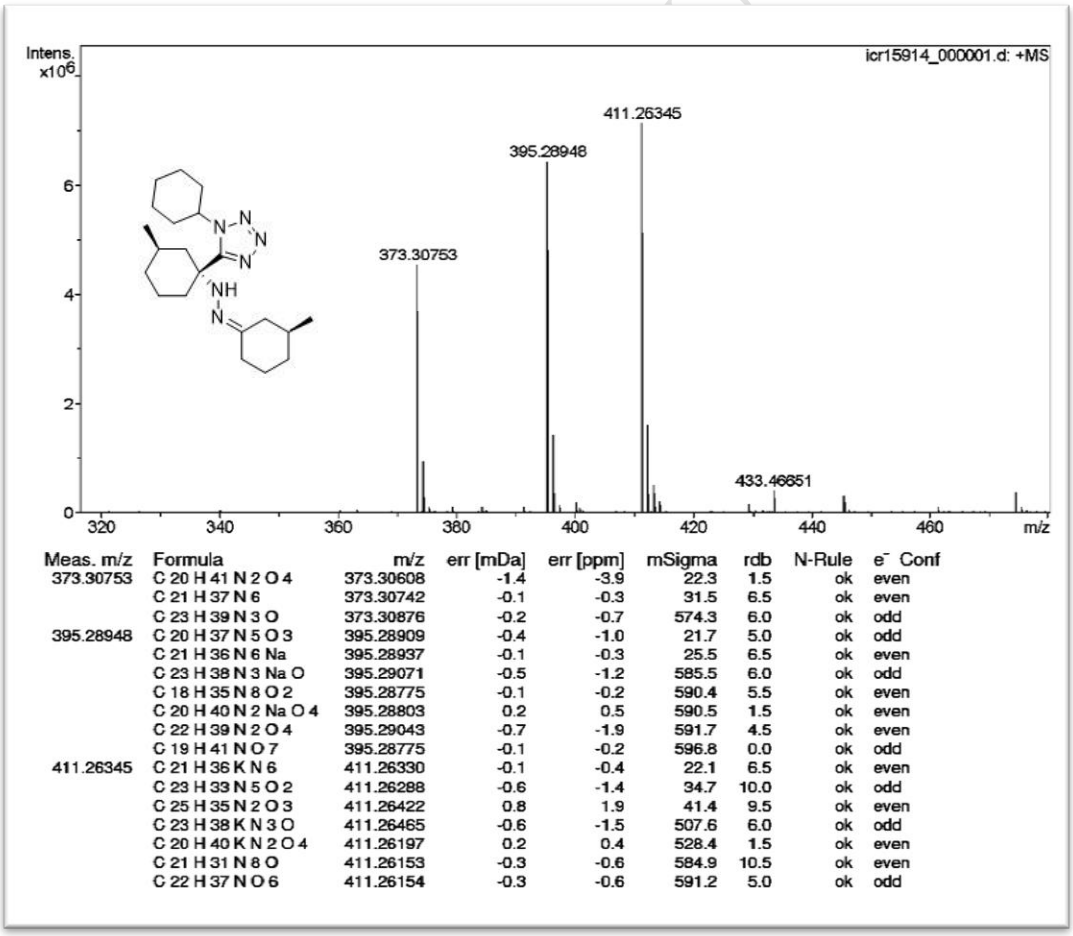
^{1}HNMR (300MHz, $CDCl_3$) **(5f)**



^{13}CNMR (75MHz, $CDCl_3$) **(5f)**

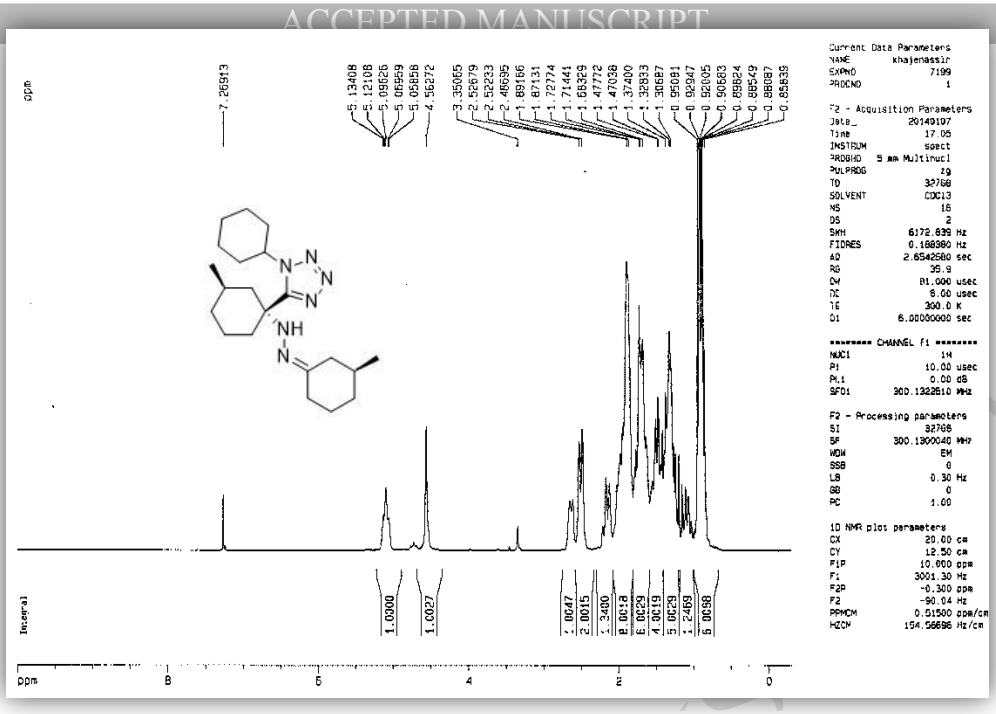


IR (KBr) **(5g)**

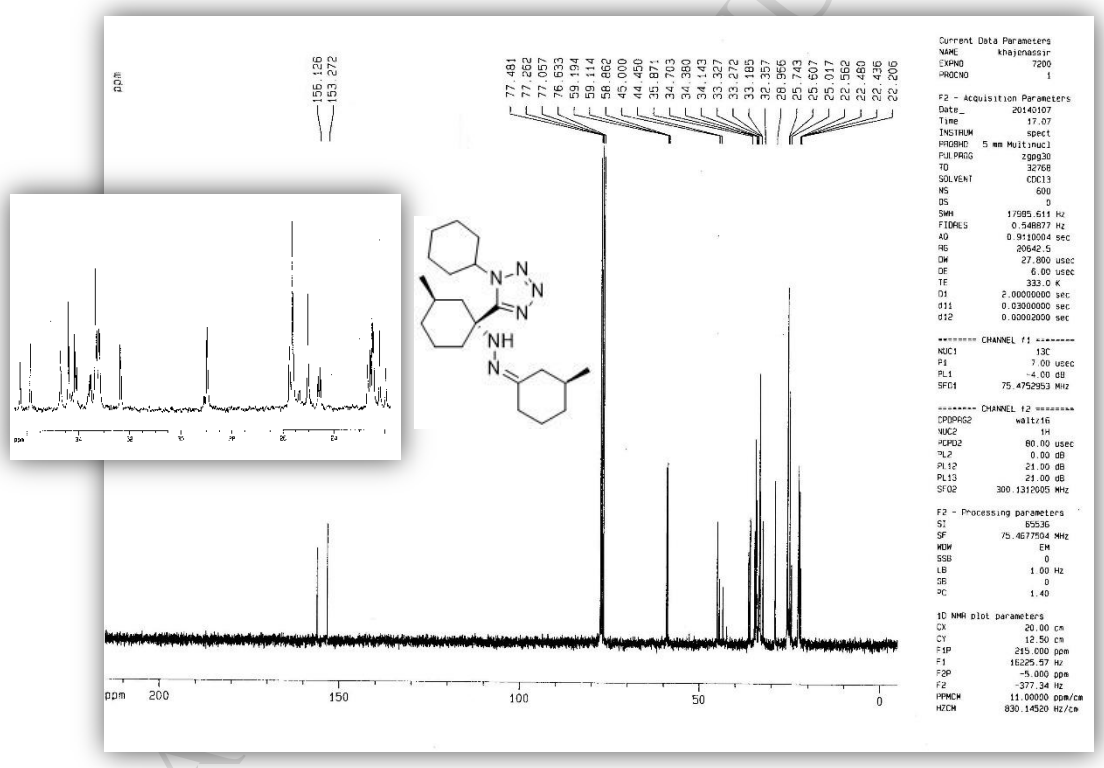


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
373.30753	C 20 H 41 N 2 O 4	373.30608	-1.4	-3.9	22.3	1.5	ok	even
	C 21 H 37 N 6	373.30742	-0.1	-0.3	31.5	6.5	ok	even
	C 23 H 39 N 3 O	373.30876	-0.2	-0.7	574.3	6.0	ok	odd
395.28948	C 20 H 37 N 5 O 3	395.28909	-0.4	-1.0	21.7	5.0	ok	odd
	C 21 H 36 N 6 Na	395.28937	-0.1	-0.3	25.5	6.5	ok	even
	C 23 H 38 N 3 Na O	395.29071	-0.5	-1.2	585.5	6.0	ok	odd
	C 18 H 35 N 8 O 2	395.28775	-0.1	-0.2	590.4	5.5	ok	even
	C 20 H 40 N 2 Na O 4	395.28803	0.2	0.5	590.5	1.5	ok	even
	C 22 H 39 N 2 O 4	395.29043	-0.7	-1.9	591.7	4.5	ok	even
	C 19 H 41 N O 7	395.28775	-0.1	-0.2	596.8	0.0	ok	odd
411.26345	C 21 H 36 K N 6	411.26330	-0.1	-0.4	22.1	6.5	ok	even
	C 23 H 33 N 5 O 2	411.26288	-0.6	-1.4	34.7	10.0	ok	odd
	C 25 H 35 N 2 O 3	411.26422	0.8	1.9	41.4	9.5	ok	even
	C 23 H 38 K N 3 O	411.26465	-0.6	-1.5	507.6	6.0	ok	odd
	C 20 H 40 K N 2 O 4	411.26197	0.2	0.4	528.4	1.5	ok	even
	C 21 H 31 N 8 O	411.26153	-0.3	-0.6	584.9	10.5	ok	even
	C 22 H 37 N O 6	411.26154	-0.3	-0.6	591.2	5.0	ok	odd

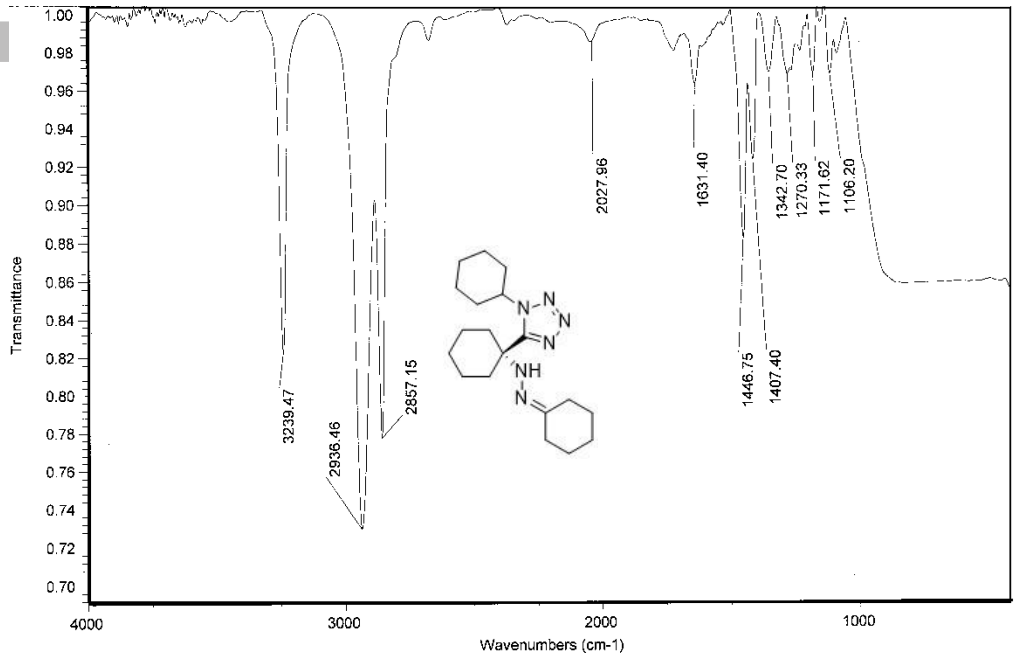
ESI-HRMS **(5g)**



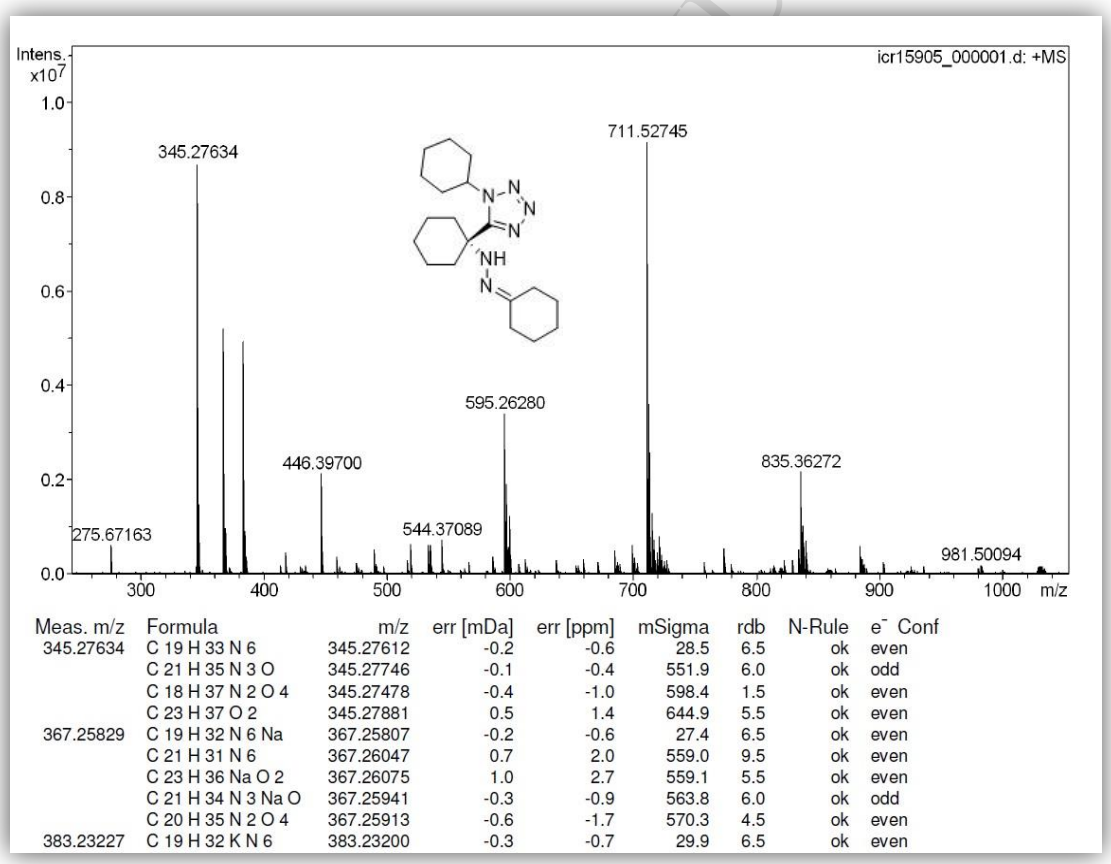
^{1}HNMR (300MHz, $CDCl_3$) **(5g)**



^{13}CNMR (75MHz, $CDCl_3$) **(5g)**

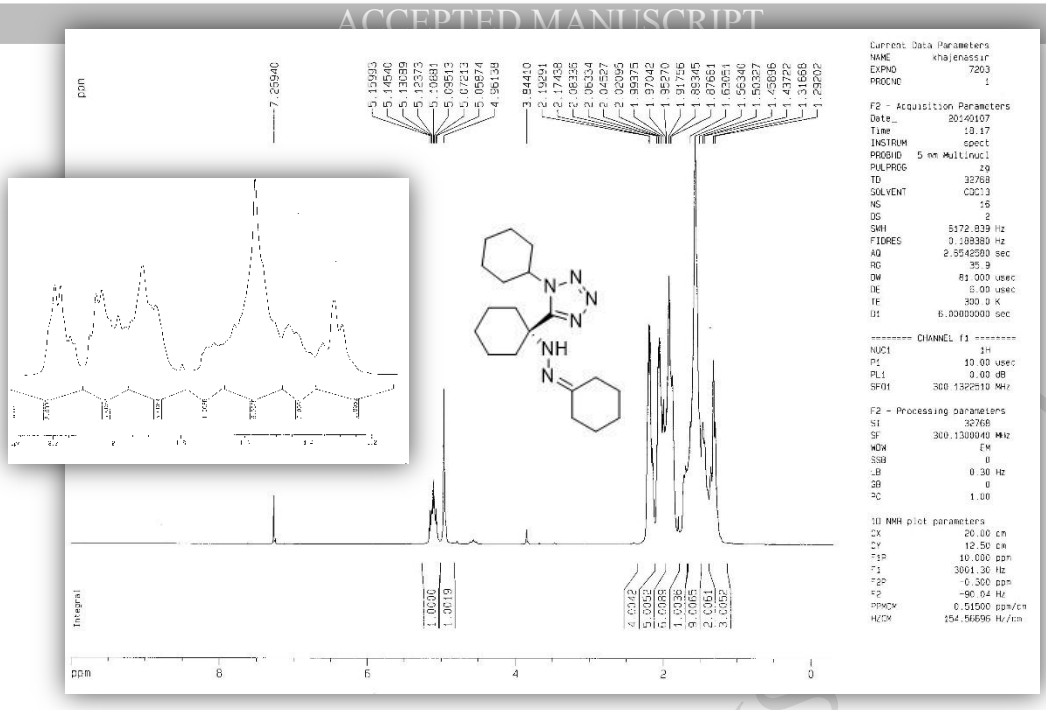


IR (KBr) **(5h)**

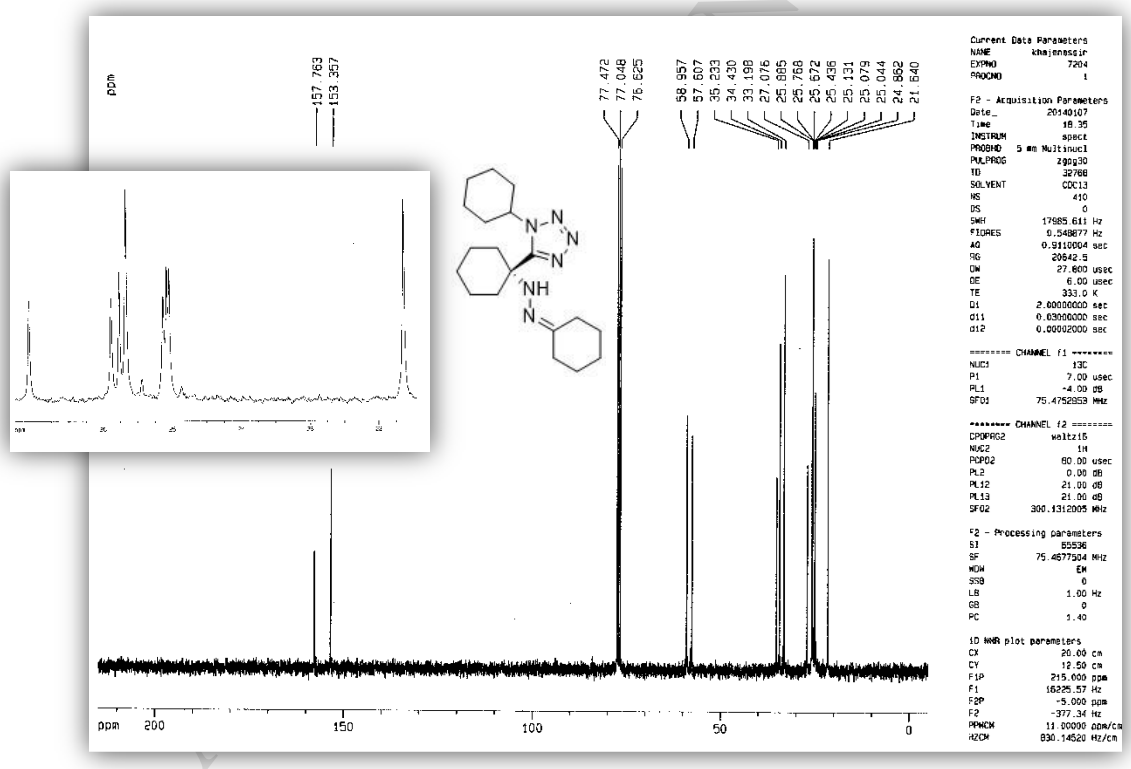


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
345.27634	C 19 H 33 N 6	345.27612	-0.2	-0.6	28.5	6.5	ok	even
	C 21 H 35 N 3 O	345.27746	-0.1	-0.4	551.9	6.0	ok	odd
	C 18 H 37 N 2 O 4	345.27478	-0.4	-1.0	598.4	1.5	ok	even
	C 23 H 37 O 2	345.27881	0.5	1.4	644.9	5.5	ok	even
367.25829	C 19 H 32 N 6 Na	367.25807	-0.2	-0.6	27.4	6.5	ok	even
	C 21 H 31 N 6	367.26047	0.7	2.0	559.0	9.5	ok	even
	C 23 H 36 Na O 2	367.26075	1.0	2.7	559.1	5.5	ok	even
	C 21 H 34 N 3 Na O	367.25941	-0.3	-0.9	563.8	6.0	ok	odd
	C 20 H 35 N 2 O 4	367.25913	-0.6	-1.7	570.3	4.5	ok	even
383.23227	C 19 H 32 K N 6	383.23200	-0.3	-0.7	29.9	6.5	ok	even

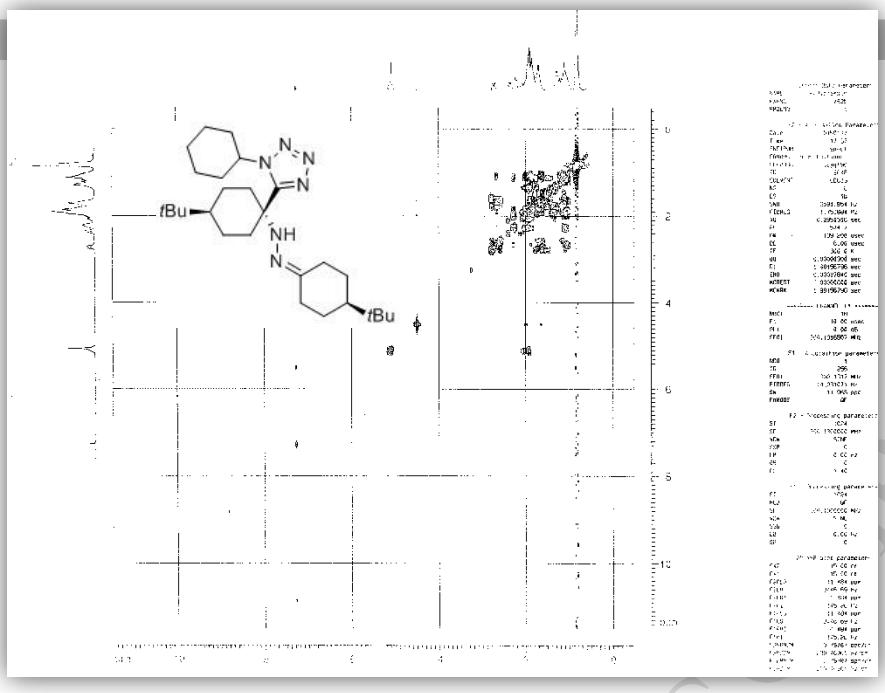
ESI-HRMS **(5h)**



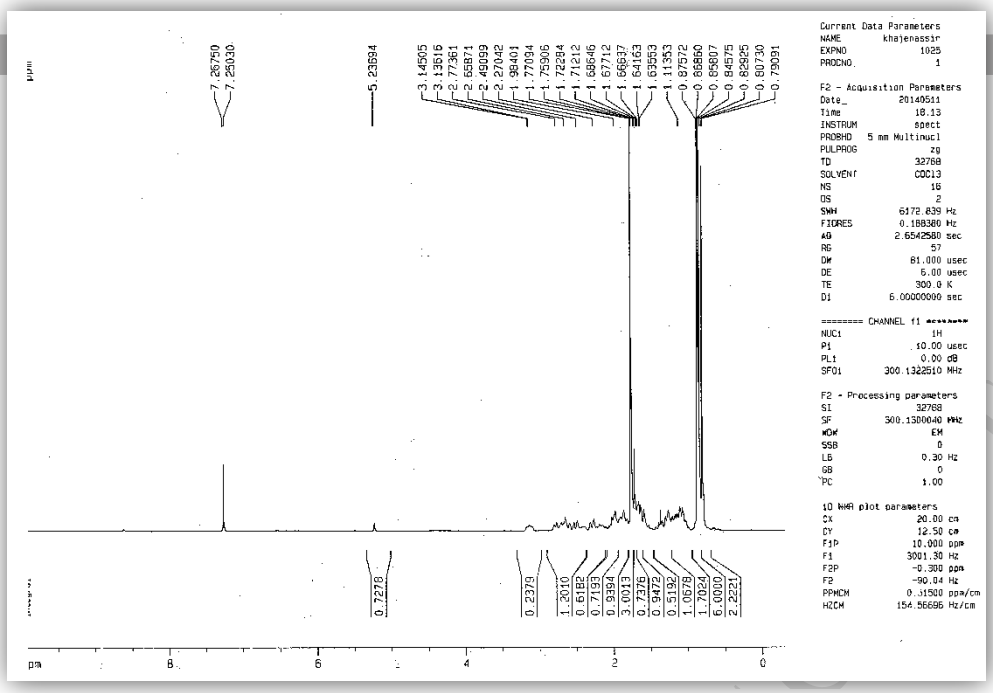
^{1}HNMR (300MHz, $CDCl_3$) **(5h)**



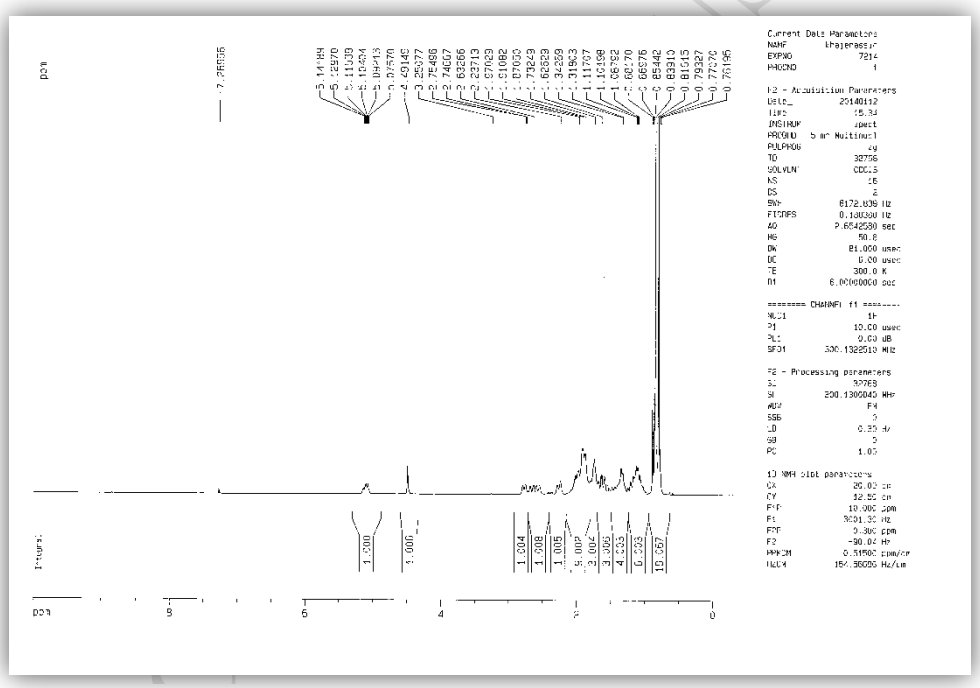
^{13}CNMR (75MHz, $CDCl_3$) **(5h)**



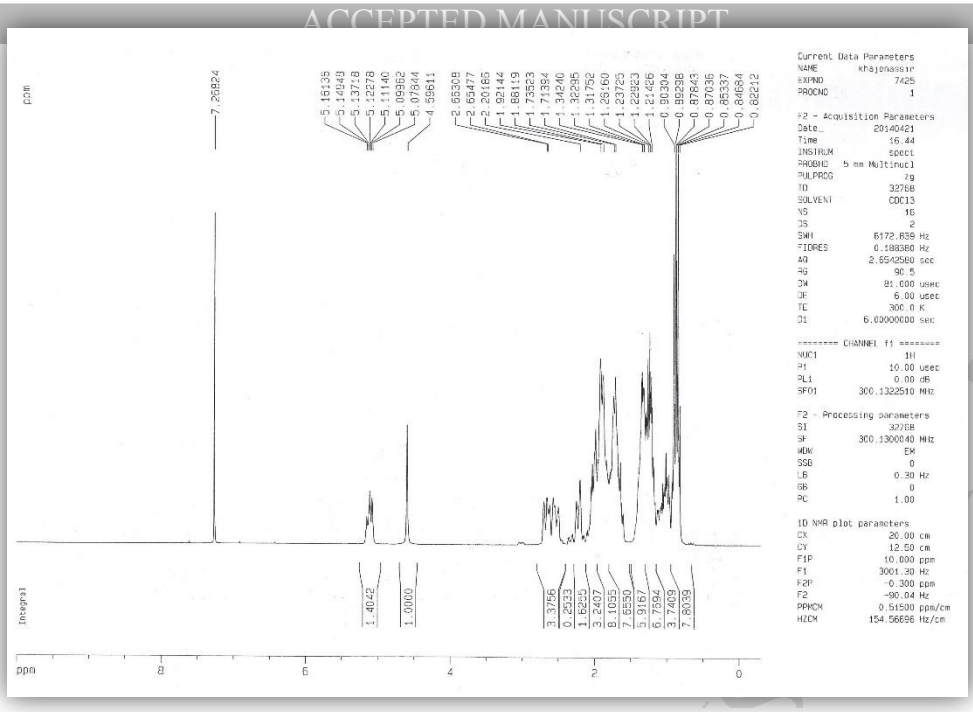
^{1}H-^{1}H COSY (300 MHz, $CDCl_3$) (**5d**)



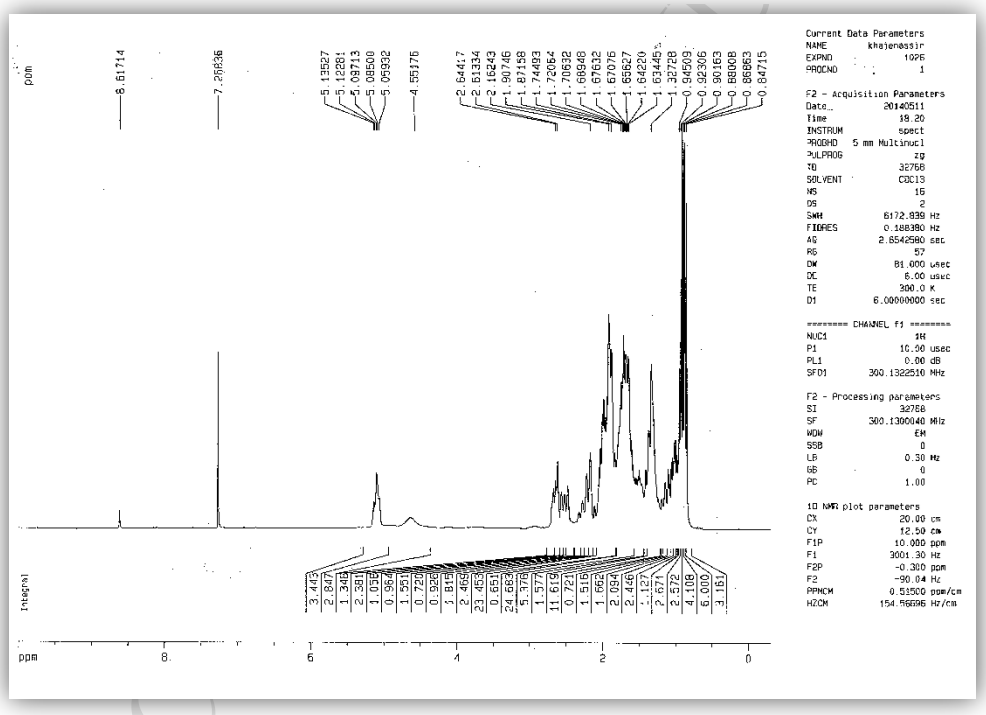
Crude ^{1}HNMR (300MHz, $CDCl_3$) **(5a)**



Crude ^{1}HNMR (300MHz, $CDCl_3$) **(5d)**



Crude ^{1}HNMR (300MHz, $CDCl_3$) **(5e)**



Crude ^{1}HNMR (300MHz, $CDCl_3$) **(5f)**

X-Ray Crystallographic Data 5a

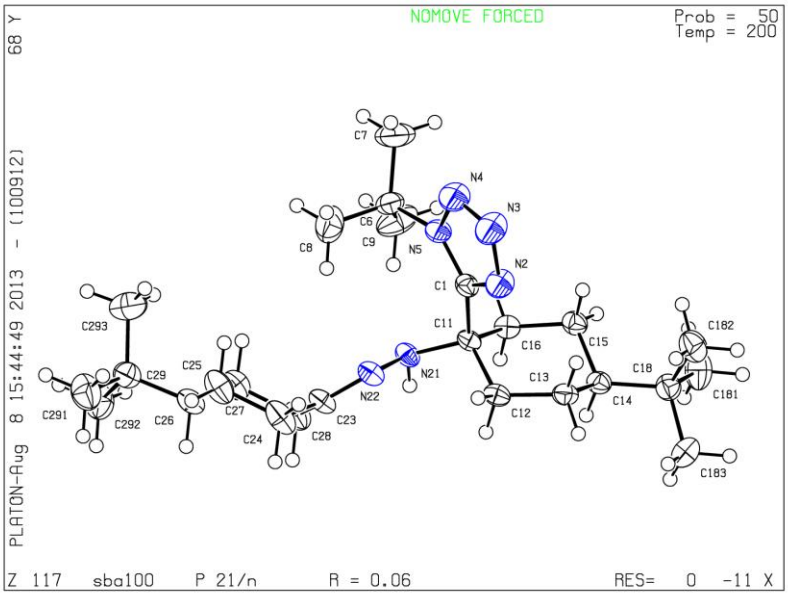


Table 1: Crystal data and structure refinement for **5a**.

Identification code	5a	
Empirical formula	$C_{25}H_{46}N_6$	
Formula weight	430.68	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Z	4	
Unit cell dimensions	a = 6.2048(7) Å	α = 90 deg.
	b = 29.336(3) Å	β = 98.484(4) deg.
	c = 14.4809(17) Å	γ = 90 deg.
Volume	2607.0(5) $Å^3$	
Density (calculated)	1.10 g/cm^3	
Absorption coefficient	0.07 mm^{-1}	
Crystal shape	plate	
Crystal size	0.370 x 0.230 x 0.030 mm^3	
Crystal colour	colourless	
Theta range for data collection	1.4 to 26.0 deg.	
Index ranges	$-7 \le h \le 7$, $-35 \le k \le 36$, $-17 \le l \le 17$	
Reflections collected	16371	
Independent reflections	5139 (R(int) = 0.0805)	
Observed reflections	3027 ($I > 2\sigma(I)$)	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.96 and 0.90	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	5139 / 0 / 285	
Goodness-of-fit on F^2	0.99	
Final R indices (I>2sigma(I))	R1 = 0.057, wR2 = 0.109	

Largest diff. peak and hole	0.21 and -0.22 eÅ^{-3}

Tabelle 2: Atomkoordinaten und äquivalente isotrope Auslenkungsparameter (Å^2) für sba100. U_{eq} wird berechnet als ein Drittel der Spur des orthogonalen U_{ij} Tensors. (Atomic coordinates and equivalent isotropic displacement parameters (Å^2) for sba100. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.)

Atom	x	y	z	U_{eq}
C1	0.2043(3)	0.2416(1)	0.6006(1)	0.0278(5)
N2	0.3606(3)	0.2677(1)	0.6441(1)	0.0404(5)
N3	0.4119(4)	0.2508(1)	0.7314(1)	0.0545(6)
N4	0.2930(4)	0.2156(1)	0.7426(1)	0.0491(6)
N5	0.1569(3)	0.2088(1)	0.6616(1)	0.0329(4)
C6	0.0236(4)	0.1658(1)	0.6594(2)	0.0375(6)
C7	-0.0138(5)	0.1549(1)	0.7584(2)	0.0667(9)
H7A	0.1270	0.1513	0.7984	0.100
H7B	-0.0973	0.1265	0.7585	0.100
H7C	-0.0952	0.1798	0.7823	0.100
C8	0.1563(5)	0.1283(1)	0.6224(2)	0.0685(9)
H8A	0.1782	0.1355	0.5584	0.103
H8B	0.0783	0.0992	0.6229	0.103
H8C	0.2981	0.1258	0.6621	0.103
C9	-0.1968(4)	0.1711(1)	0.6005(2)	0.0622(8)
H9A	-0.1778	0.1777	0.5359	0.093
H9B	-0.2758	0.1962	0.6250	0.093
H9C	-0.2799	0.1428	0.6025	0.093
C11	0.1001(3)	0.2529(1)	0.5019(1)	0.0256(5)
C12	0.2422(3)	0.2866(1)	0.4557(1)	0.0295(5)
H12A	0.3949	0.2759	0.4677	0.035
H12B	0.1955	0.2855	0.3873	0.035
C13	0.2374(3)	0.3362(1)	0.4869(1)	0.0306(5)
H13A	0.3181	0.3551	0.4469	0.037
H13B	0.3126	0.3388	0.5519	0.037
C14	0.0055(3)	0.3547(1)	0.4818(1)	0.0290(5)
H14	-0.0647	0.3510	0.4155	0.035
C15	-0.1175(3)	0.3230(1)	0.5401(1)	0.0304(5)
H15A	-0.0442	0.3229	0.6056	0.036
H15B	-0.2677	0.3345	0.5394	0.036
C16	-0.1264(3)	0.2744(1)	0.5023(1)	0.0286(5)
H16A	-0.2054	0.2745	0.4377	0.034
H16B	-0.2100	0.2552	0.5407	0.034
C18	-0.0064(4)	0.4060(1)	0.5045(2)	0.0349(5)
N21	0.0689(3)	0.2105(1)	0.4468(1)	0.0284(4)
H21	-0.009(3)	0.2172(6)	0.3891(15)	0.033(6)
N22	0.2709(3)	0.1910(1)	0.4354(1)	0.0314(4)
C23	0.2688(3)	0.1611(1)	0.3711(1)	0.0333(5)
C24	0.4784(4)	0.1373(1)	0.3624(2)	0.0464(7)
H24A	0.5961	0.1496	0.4094	0.056
H24B	0.5182	0.1431	0.2997	0.056
C25	0.4573(4)	0.0860(1)	0.3768(2)	0.0437(6)
H25A	0.4361	0.0802	0.4422	0.052
H25B	0.5942	0.0709	0.3663	0.052
C26	0.2665(3)	0.0654(1)	0.3108(2)	0.0347(6)
H26	0.2962	0.0717	0.2460	0.042
C27	0.0595(3)	0.0917(1)	0.3220(2)	0.0385(6)
H27A	-0.0626	0.0796	0.2769	0.046
H27B	0.0242	0.0863	0.3856	0.046

C28	0.0776(4)	0.1427(1)	0.3067(2)	0.0387(6)
H28A	0.0964	0.1488	0.2411	0.046
H28B	-0.0575	0.1581	0.3190	0.046
C29	0.2449(4)	0.0131(1)	0.3182(2)	0.0395(6)
C181	-0.2429(4)	0.4208(1)	0.5047(2)	0.0540(7)
H18A	-0.3049	0.4039	0.5528	0.081
H18B	-0.3279	0.4146	0.4434	0.081
H18C	-0.2473	0.4536	0.5179	0.081
C182	0.1255(4)	0.4177(1)	0.5989(2)	0.0473(7)
H18D	0.0702	0.4002	0.6480	0.071
H18E	0.1120	0.4504	0.6111	0.071
H18F	0.2790	0.4101	0.5981	0.071
C183	0.0836(4)	0.4334(1)	0.4290(2)	0.0566(8)
H18G	0.0000	0.4263	0.3679	0.085
H18H	0.2370	0.4254	0.4289	0.085
H18I	0.0716	0.4660	0.4417	0.085
C291	0.4614(4)	-0.0099(1)	0.3094(2)	0.0582(8)
H29A	0.4452	-0.0431	0.3141	0.087
H29B	0.5734	0.0007	0.3596	0.087
H29C	0.5045	-0.0023	0.2488	0.087
C292	0.0766(4)	-0.0042(1)	0.2376(2)	0.0537(7)
H29D	0.0617	-0.0373	0.2425	0.081
H29E	0.1249	0.0034	0.1780	0.081
H29F	-0.0644	0.0103	0.2407	0.081
C293	0.1740(5)	-0.0013(1)	0.4105(2)	0.0675(8)
H29G	0.1606	-0.0346	0.4121	0.101
H29H	0.0329	0.0126	0.4161	0.101
H29I	0.2829	0.0088	0.4625	0.101

Tabelle 3: H-Atomkoordinaten und isotrope Auslenkungsparameter ($Å^2$) für sba100.
(Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for sba100.)

Atom	x	y	z	U_{eq}
H7A	0.1270	0.1513	0.7984	0.100
H7B	-0.0973	0.1265	0.7585	0.100
H7C	-0.0952	0.1798	0.7823	0.100
H8A	0.1782	0.1355	0.5584	0.103
H8B	0.0783	0.0992	0.6229	0.103
H8C	0.2981	0.1258	0.6621	0.103
H9A	-0.1778	0.1777	0.5359	0.093
H9B	-0.2758	0.1962	0.6250	0.093
H9C	-0.2799	0.1428	0.6025	0.093
H12A	0.3949	0.2759	0.4677	0.035
H12B	0.1955	0.2855	0.3873	0.035
H13A	0.3181	0.3551	0.4469	0.037
H13B	0.3126	0.3388	0.5519	0.037
H14	-0.0647	0.3510	0.4155	0.035
H15A	-0.0442	0.3229	0.6056	0.036
H15B	-0.2677	0.3345	0.5394	0.036
H16A	-0.2054	0.2745	0.4377	0.034
H16B	-0.2100	0.2552	0.5407	0.034
H21	-0.009(3)	0.2172(6)	0.3891(15)	0.033(6)
H24A	0.5961	0.1496	0.4094	0.056
H24B	0.5182	0.1431	0.2997	0.056
H25A	0.4361	0.0802	0.4422	0.052
H25B	0.5942	0.0709	0.3663	0.052
H26	0.2962	0.0717	0.2460	0.042

H27A	-0.0626	0.0796	0.2769	0.046
H27B	0.0242	0.0863	0.3856	0.046
H28A	0.0964	0.1488	0.2411	0.046
H28B	-0.0575	0.1581	0.3190	0.046
H18A	-0.3049	0.4039	0.5528	0.081
H18B	-0.3279	0.4146	0.4434	0.081
H18C	-0.2473	0.4536	0.5179	0.081
H18D	0.0702	0.4002	0.6480	0.071
H18E	0.1120	0.4504	0.6111	0.071
H18F	0.2790	0.4101	0.5981	0.071
H18G	0.0000	0.4263	0.3679	0.085
H18H	0.2370	0.4254	0.4289	0.085
H18I	0.0716	0.4660	0.4417	0.085
H29A	0.4452	-0.0431	0.3141	0.087
H29B	0.5734	0.0007	0.3596	0.087
H29C	0.5045	-0.0023	0.2488	0.087
H29D	0.0617	-0.0373	0.2425	0.081
H29E	0.1249	0.0034	0.1780	0.081
H29F	-0.0644	0.0103	0.2407	0.081
H29G	0.1606	-0.0346	0.4121	0.101
H29H	0.0329	0.0126	0.4161	0.101
H29I	0.2829	0.0088	0.4625	0.101

Tabelle 4: Anisotrope Auslenkungsparameter ($Å^2$) für sba100. Der Exponent für den anisotropen Auslenkungsparameter hat die Form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$
(Anisotropic displacement parameters ($Å^2$) for sba100. The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$)

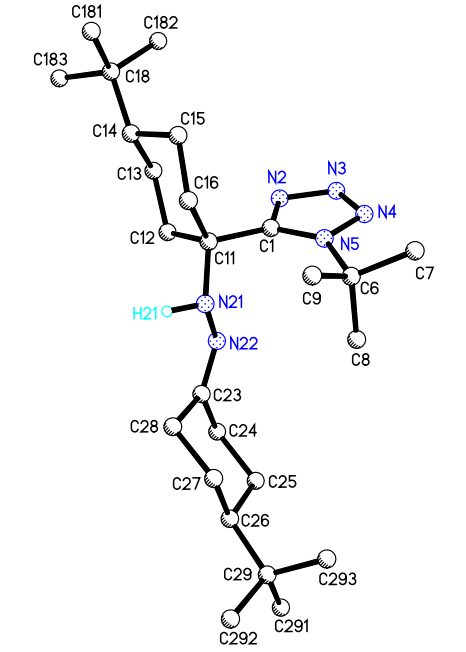
Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0299(12)	0.0303(12)	0.0224(11)	-0.0031(9)	0.0011(9)	0.0017(10)
N2	0.0469(12)	0.0390(11)	0.0300(11)	-0.0004(9)	-0.0119(9)	-0.0051(10)
N3	0.0758(16)	0.0453(13)	0.0337(12)	0.0014(10)	-0.0204(11)	-0.0093(12)
N4	0.0687(15)	0.0470(13)	0.0255(11)	0.0006(10)	-0.0134(10)	-0.0057(12)
N5	0.0423(11)	0.0356(11)	0.0186(9)	-0.0012(8)	-0.0030(8)	-0.0014(9)
C6	0.0479(15)	0.0359(13)	0.0293(12)	0.0056(10)	0.0081(11)	-0.0071(12)
C7	0.090(2)	0.075(2)	0.0375(16)	0.0127(14)	0.0172(15)	-0.0160(17)
C8	0.082(2)	0.0387(16)	0.093(2)	-0.0031(15)	0.0388(19)	-0.0057(15)
C9	0.0566(18)	0.0673(19)	0.0587(18)	0.0218(15)	-0.0053(14)	-0.0262(15)
C11	0.0256(11)	0.0316(12)	0.0191(11)	-0.0034(9)	0.0018(8)	-0.0013(10)
C12	0.0251(12)	0.0385(13)	0.0257(11)	-0.0006(10)	0.0061(9)	0.0009(10)
C13	0.0324(13)	0.0346(13)	0.0259(12)	0.0008(9)	0.0077(9)	-0.0035(10)
C14	0.0306(12)	0.0325(12)	0.0234(11)	0.0001(9)	0.0020(9)	0.0006(10)
C15	0.0270(12)	0.0348(13)	0.0299(12)	0.0001(10)	0.0063(9)	0.0038(10)
C16	0.0233(12)	0.0346(13)	0.0271(12)	0.0014(9)	0.0018(9)	-0.0009(10)
C18	0.0364(13)	0.0338(13)	0.0342(13)	-0.0019(10)	0.0040(10)	0.0007(11)
N21	0.0277(10)	0.0346(11)	0.0215(10)	-0.0058(8)	-0.0009(8)	0.0021(8)
N22	0.0279(10)	0.0394(11)	0.0273(10)	-0.0050(8)	0.0058(8)	0.0006(9)
C23	0.0330(13)	0.0422(14)	0.0257(12)	-0.0088(10)	0.0077(10)	-0.0015(11)
C24	0.0323(14)	0.0578(17)	0.0508(16)	-0.0251(13)	0.0122(12)	-0.0086(13)
C25	0.0270(13)	0.0552(16)	0.0481(15)	-0.0164(12)	0.0029(11)	0.0060(12)
C26	0.0323(13)	0.0437(14)	0.0294(12)	-0.0077(10)	0.0086(10)	-0.0017(11)
C27	0.0307(13)	0.0462(15)	0.0370(13)	-0.0097(11)	-0.0003(10)	-0.0029(11)
C28	0.0394(14)	0.0438(14)	0.0307(13)	-0.0097(11)	-0.0023(10)	0.0003(12)
C29	0.0437(15)	0.0413(15)	0.0342(13)	-0.0005(11)	0.0076(11)	0.0027(12)
C181	0.0453(16)	0.0416(15)	0.0728(19)	-0.0075(14)	0.0011(14)	0.0101(13)
C182	0.0506(16)	0.0454(15)	0.0455(15)	-0.0126(12)	0.0054(12)	-0.0046(12)
C183	0.082(2)	0.0344(15)	0.0569(18)	0.0092(13)	0.0200(15)	-0.0005(14)

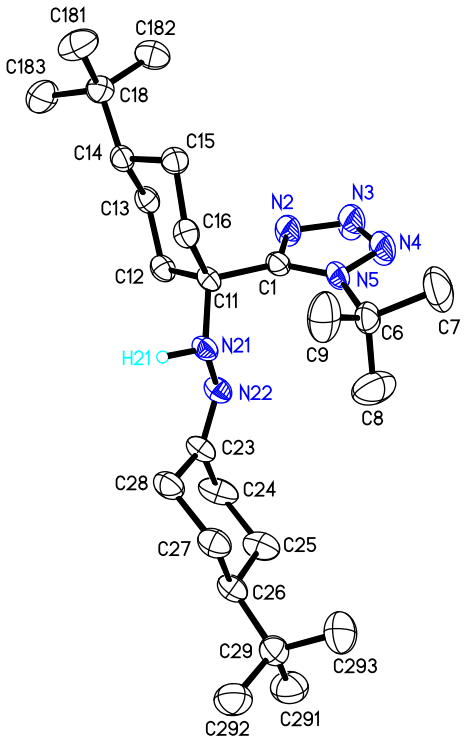
C291	0.0597(18)	0.0535(17)	0.0604(18)	-0.0085(14)	0.0057(14)	0.0115(14)
C292	0.0577(17)	0.0443(16)	0.0571(18)	-0.0078(13)	0.0017(14)	-0.0049(13)
C293	0.089(2)	0.067(2)	0.0489(18)	0.0122(15)	0.0196(16)	-0.0068(17)

Tabelle 5: Bindungslängen (Å) und -winkel (°) für sba100.
(Bond lengths (Å) and angles (deg) for sba100.)

C1-N2	1.320(2)	C27-H27A	0.9900
C1-N5	1.366(3)	C27-H27B	0.9900
C1-C11	1.516(3)	C28-H28A	0.9900
N2-N3	1.352(2)	C28-H28B	0.9900
N3-N4	1.292(3)	C29-C291	1.526(3)
N4-N5	1.356(2)	C29-C293	1.527(3)
N5-C6	1.507(3)	C29-C292	1.534(3)
C6-C9	1.508(3)	C181-H18A	0.9800
C6-C8	1.519(3)	C181-H18B	0.9800
C6-C7	1.521(3)	C181-H18C	0.9800
C7-H7A	0.9800	C182-H18D	0.9800
C7-H7B	0.9800	C182-H18E	0.9800
C7-H7C	0.9800	C182-H18F	0.9800
C8-H8A	0.9800	C183-H18G	0.9800
C8-H8B	0.9800	C183-H18H	0.9800
C8-H8C	0.9800	C183-H18I	0.9800
C9-H9A	0.9800	C291-H29A	0.9800
C9-H9B	0.9800	C291-H29B	0.9800
C9-H9C	0.9800	C291-H29C	0.9800
C11-N21	1.475(2)	C292-H29D	0.9800
C11-C16	1.541(3)	C292-H29E	0.9800
C11-C12	1.543(3)	C292-H29F	0.9800
C12-C13	1.525(3)	C293-H29G	0.9800
C12-H12A	0.9900	C293-H29H	0.9800
C12-H12B	0.9900	C293-H29I	0.9800
C13-C14	1.529(3)	N2-C1-N5	108.24(18)
C13-H13A	0.9900	N2-C1-C11	120.22(19)
C13-H13B	0.9900	N5-C1-C11	131.37(18)
C14-C15	1.533(3)	C1-N2-N3	106.74(18)
C14-C18	1.544(3)	N4-N3-N2	110.49(18)
C14-H14	1.0000	N3-N4-N5	107.69(17)
C15-C16	1.524(3)	N4-N5-C1	106.82(17)
C15-H15A	0.9900	N4-N5-C6	114.35(17)
C15-H15B	0.9900	C1-N5-C6	138.18(17)
C16-H16A	0.9900	N5-C6-C9	112.02(18)
C16-H16B	0.9900	N5-C6-C8	106.87(18)
C18-C182	1.525(3)	C9-C6-C8	111.7(2)
C18-C183	1.528(3)	N5-C6-C7	108.40(19)
C18-C181	1.531(3)	C9-C6-C7	107.6(2)
N21-N22	1.410(2)	C8-C6-C7	110.2(2)
N21-H21	0.92(2)	C6-C7-H7A	109.5
N22-C23	1.277(2)	C6-C7-H7B	109.5
C23-C28	1.497(3)	H7A-C7-H7B	109.5
C23-C24	1.498(3)	C6-C7-H7C	109.5
C24-C25	1.527(3)	H7A-C7-H7C	109.5
C24-H24A	0.9900	H7B-C7-H7C	109.5
C24-H24B	0.9900	C6-C8-H8A	109.5
C25-C26	1.532(3)	C6-C8-H8B	109.5
C25-H25A	0.9900	H8A-C8-H8B	109.5
C25-H25B	0.9900	C6-C8-H8C	109.5
C26-C27	1.527(3)	H8A-C8-H8C	109.5
C26-C29	1.545(3)	H8B-C8-H8C	109.5
C26-H26	1.0000	C6-C9-H9A	109.5
C27-C28	1.521(3)	C6-C9-H9B	109.5

H9A-C9-H9B	109.5	H25A-C25-H25B	107.9
C6-C9-H9C	109.5	C27-C26-C25	108.62(17)
H9A-C9-H9C	109.5	C27-C26-C29	114.28(18)
H9B-C9-H9C	109.5	C25-C26-C29	114.57(19)
N21-C11-C1	109.20(17)	C27-C26-H26	106.2
N21-C11-C16	107.43(16)	C25-C26-H26	106.2
C1-C11-C16	110.44(16)	C29-C26-H26	106.2
N21-C11-C12	110.09(16)	C28-C27-C26	113.61(19)
C1-C11-C12	111.02(16)	C28-C27-H27A	108.8
C16-C11-C12	108.59(16)	C26-C27-H27A	108.8
C13-C12-C11	116.45(17)	C28-C27-H27B	108.8
C13-C12-H12A	108.2	C26-C27-H27B	108.8
C11-C12-H12A	108.2	H27A-C27-H27B	107.7
C13-C12-H12B	108.2	C23-C28-C27	109.44(18)
C11-C12-H12B	108.2	C23-C28-H28A	109.8
H12A-C12-H12B	107.3	C27-C28-H28A	109.8
C12-C13-C14	112.55(17)	C23-C28-H28B	109.8
C12-C13-H13A	109.1	C27-C28-H28B	109.8
C14-C13-H13A	109.1	H28A-C28-H28B	108.2
C12-C13-H13B	109.1	C291-C29-C293	108.8(2)
C14-C13-H13B	109.1	C291-C29-C292	107.4(2)
H13A-C13-H13B	107.8	C293-C29-C292	108.8(2)
C13-C14-C15	107.43(16)	C291-C29-C26	110.4(2)
C13-C14-C18	114.18(17)	C293-C29-C26	112.0(2)
C15-C14-C18	115.58(17)	C292-C29-C26	109.37(19)
C13-C14-H14	106.3	C18-C181-H18A	109.5
C15-C14-H14	106.3	C18-C181-H18B	109.5
C18-C14-H14	106.3	H18A-C181-H18B	109.5
C16-C15-C14	111.39(17)	C18-C181-H18C	109.5
C16-C15-H15A	109.3	H18A-C181-H18C	109.5
C14-C15-H15A	109.3	H18B-C181-H18C	109.5
C16-C15-H15B	109.3	C18-C182-H18D	109.5
C14-C15-H15B	109.3	C18-C182-H18E	109.5
H15A-C15-H15B	108.0	H18D-C182-H18E	109.5
C15-C16-C11	113.52(16)	C18-C182-H18F	109.5
C15-C16-H16A	108.9	H18D-C182-H18F	109.5
C11-C16-H16A	108.9	H18E-C182-H18F	109.5
C15-C16-H16B	108.9	C18-C183-H18G	109.5
C11-C16-H16B	108.9	C18-C183-H18H	109.5
H16A-C16-H16B	107.7	H18G-C183-H18H	109.5
C182-C18-C183	108.6(2)	C18-C183-H18I	109.5
C182-C18-C181	108.80(19)	H18G-C183-H18I	109.5
C183-C18-C181	107.6(2)	H18H-C183-H18I	109.5
C182-C18-C14	111.97(18)	C29-C291-H29A	109.5
C183-C18-C14	109.05(18)	C29-C291-H29B	109.5
C181-C18-C14	110.70(18)	H29A-C291-H29B	109.5
N22-N21-C11	110.98(15)	C29-C291-H29C	109.5
N22-N21-H21	109.5(13)	H29A-C291-H29C	109.5
C11-N21-H21	108.7(12)	H29B-C291-H29C	109.5
C23-N22-N21	116.76(17)	C29-C292-H29D	109.5
N22-C23-C28	128.6(2)	C29-C292-H29E	109.5
N22-C23-C24	117.73(19)	H29D-C292-H29E	109.5
C28-C23-C24	113.43(18)	C29-C292-H29F	109.5
C23-C24-C25	110.76(19)	H29D-C292-H29F	109.5
C23-C24-H24A	109.5	H29E-C292-H29F	109.5
C25-C24-H24A	109.5	C29-C293-H29G	109.5
C23-C24-H24B	109.5	C29-C293-H29H	109.5
C25-C24-H24B	109.5	H29G-C293-H29H	109.5
H24A-C24-H24B	108.1	C29-C293-H29I	109.5
C24-C25-C26	112.2(2)	H29G-C293-H29I	109.5
C24-C25-H25A	109.2	H29H-C293-H29I	109.5
C26-C25-H25A	109.2		
C24-C25-H25B	109.2		
C26-C25-H25B	109.2		





5a: colourless crystal (plate), dimensions 0.370 x 0.230 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.2048(7) Å, b=29.336(3) Å, c=14.4809(17) Å, alpha=90 deg, beta=98.484(4) deg, gamma=90 deg, V=2607.0(5) $Å^3$, rho=1.097 g/cm^3, T=200(2) K, $Theta_{max}$= 26.022 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.15and a completeness of 100.0% to a resolution of 0.81Å, 16371 reflections measured, 5139 unique (R(int)=0.0805), 3027 observed (I > 2σ(I)), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using

SADABS[1] based on the Laue symmetry of the reciprocal space, mu=0.07mm^{-1}, T_{min}=0.90, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package[2], 285 parameters refined, hydrogen atoms were treated using appropriate riding models, except H21 at N21, which was refined isotropically, goodness of fit 0.99 for observed reflections, final residual values R1(F)=0.057, wR(F^2)=0.109 for observed reflections, residual electron density -0.22 to 0.21 eÅ^{-3}. CCDC 1030930 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Lit. 1: (program SADABS 2012/1 for absorption correction) G. M. Sheldrick, Bruker Analytical X-ray-Division, Madison, Wisconsin 2012

Lit. 2: (software package SHELXTL 2013/3 for structure solution and refinement) Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Lit. APEX, APEX2, SMART, SAINT, SAINT-Plus: Bruker (2007). "Program name(s)". Bruker AXS Inc., Madison, Wisconsin, USA.

X-Ray Crystallographic Data 5d

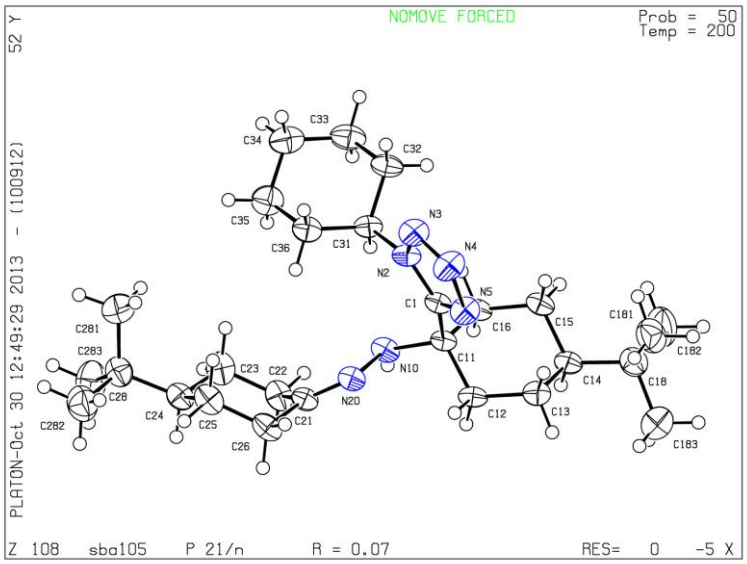


Table 1: Crystal data and structure refinement for sba105.

Identification code	sba105	
Empirical formula	$C_{27}H_{48}N_6$	
Formula weight	456.71	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Z	4	
Unit cell dimensions	a = 6.345(2) Å	α = 90 deg.
	b = 31.355(11) Å	β = 99.872(11) deg.
	c = 14.286(5) Å	γ = 90 deg.
Volume	2799.8(17) $Å^3$	
Density (calculated)	1.08 g/cm^3	
Absorption coefficient	0.06 mm^{-1}	
Crystal shape	needle	
Crystal size	0.420 x 0.050 x 0.030 mm^3	
Crystal colour	colourless	
Theta range for data collection	1.9 to 21.3 deg.	
Index ranges	$-6\le h\le 5$, $-31\le k\le 31$, $-14\le l\le 14$	
Reflections collected	11459	
Independent reflections	3120 (R(int) = 0.1049)	
Observed reflections	1898 ($I > 2\sigma(I)$)	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.96 and 0.82	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	3120 / 270 / 302	
Goodness-of-fit on F^2	1.01	
Final R indices (I>2sigma(I))	R1 = 0.074, wR2 = 0.159	
Largest diff. peak and hole	0.32 and -0.33 $eÅ^{-3}$	

Tabelle 2: Atomkoordinaten und äquivalente isotrope Auslenkungsparameter ($Å^2$) für sba105. U_{eq} wird berechnet als ein Drittel der Spur des orthogonalen U_{ij} Tensors. (Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for sba105. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.)

Atom	x	y	z	U_{eq}
C1	0.6738(7)	0.2531(1)	0.5585(3)	0.0265(11)
N2	0.6256(5)	0.2216(1)	0.6149(2)	0.0302(10)
N3	0.7585(6)	0.2247(1)	0.7003(3)	0.0376(11)
N4	0.8814(6)	0.2574(1)	0.6937(3)	0.0429(11)
N5	0.8334(6)	0.2759(1)	0.6057(3)	0.0367(10)
N10	0.5385(6)	0.2208(1)	0.4084(3)	0.0310(10)
H10	0.457(6)	0.2240(11)	0.357(3)	0.015(12)
C11	0.5596(7)	0.2621(1)	0.4580(3)	0.0254(11)
C12	0.6842(7)	0.2943(1)	0.4083(3)	0.0300(12)
H12A	0.8359	0.2853	0.4178	0.036
H12B	0.6285	0.2933	0.3392	0.036
C13	0.6753(7)	0.3404(1)	0.4412(3)	0.0347(12)
H13A	0.7440	0.3589	0.3991	0.042
H13B	0.7579	0.3430	0.5064	0.042
C14	0.4456(7)	0.3560(1)	0.4407(3)	0.0331(12)
H14	0.3696	0.3532	0.3737	0.040
C15	0.3355(7)	0.3244(1)	0.4995(3)	0.0331(12)
H15A	0.4099	0.3249	0.5663	0.040
H15B	0.1857	0.3336	0.4984	0.040
C16	0.3372(6)	0.2794(1)	0.4614(3)	0.0298(12)
H16A	0.2523	0.2788	0.3964	0.036
H16B	0.2660	0.2604	0.5017	0.036
C18	0.4303(8)	0.4035(2)	0.4673(4)	0.0474(14)
N20	0.7403(6)	0.2032(1)	0.4028(3)	0.0318(10)
C21	0.7457(7)	0.1723(2)	0.3461(3)	0.0321(12)
C22	0.5600(7)	0.1506(1)	0.2836(3)	0.0410(13)
H22A	0.5718	0.1547	0.2160	0.049
H22B	0.4238	0.1636	0.2944	0.049
C23	0.5606(7)	0.1034(2)	0.3064(4)	0.0454(14)
H23A	0.5280	0.0998	0.3713	0.054
H23B	0.4447	0.0894	0.2617	0.054
C24	0.7692(7)	0.0808(1)	0.3010(4)	0.0386(13)
H24	0.7949	0.0849	0.2344	0.046
C25	0.9511(7)	0.1044(2)	0.3647(4)	0.0430(13)
H25A	0.9348	0.1005	0.4319	0.052
H25B	1.0892	0.0914	0.3567	0.052
C26	0.9571(7)	0.1516(2)	0.3433(4)	0.0418(13)
H26A	1.0686	0.1654	0.3905	0.050
H26B	0.9955	0.1557	0.2796	0.050
C28	0.7668(8)	0.0319(2)	0.3169(4)	0.0476(14)
C31	0.4686(7)	0.1869(1)	0.6014(3)	0.0309(11)
H31	0.3768	0.1908	0.5378	0.037
C32	0.3247(7)	0.1890(2)	0.6757(3)	0.0403(13)
H32A	0.2532	0.2172	0.6728	0.048
H32B	0.4114	0.1856	0.7398	0.048
C33	0.1570(8)	0.1538(2)	0.6579(4)	0.0497(14)
H33A	0.0683	0.1545	0.7086	0.060
H33B	0.0619	0.1590	0.5964	0.060
C34	0.2598(8)	0.1105(2)	0.6562(4)	0.0542(15)
H34A	0.1467	0.0887	0.6400	0.065
H34B	0.3397	0.1039	0.7203	0.065

C35	0.4113(8)	0.1082(2)	0.5850(4)	0.0513(15)
H35A	0.3287	0.1106	0.5198	0.062
H35B	0.4850	0.0803	0.5906	0.062
C36	0.5770(7)	0.1438(1)	0.6018(3)	0.0396(13)
H36A	0.6717	0.1394	0.6637	0.048
H36B	0.6664	0.1431	0.5514	0.048
C181	0.5675(9)	0.4136(2)	0.5646(4)	0.0614(16)
H18A	0.5553	0.4440	0.5787	0.092
H18B	0.7174	0.4066	0.5629	0.092
H18C	0.5172	0.3966	0.6139	0.092
C182	0.1983(8)	0.4157(2)	0.4721(5)	0.0743(19)
H18D	0.1921	0.4458	0.4899	0.111
H18E	0.1455	0.3979	0.5196	0.111
H18F	0.1089	0.4111	0.4098	0.111
C183	0.5104(10)	0.4317(2)	0.3922(5)	0.078(2)
H18G	0.5037	0.4618	0.4104	0.117
H18H	0.4198	0.4272	0.3302	0.117
H18I	0.6585	0.4242	0.3883	0.117
C281	0.7092(10)	0.0202(2)	0.4128(4)	0.0759(19)
H28A	0.7093	-0.0109	0.4197	0.114
H28B	0.5667	0.0314	0.4168	0.114
H28C	0.8145	0.0327	0.4637	0.114
C282	0.9865(8)	0.0126(2)	0.3101(4)	0.0673(18)
H28D	0.9837	-0.0182	0.3218	0.101
H28E	1.0963	0.0259	0.3578	0.101
H28F	1.0195	0.0178	0.2466	0.101
C283	0.6047(9)	0.0111(2)	0.2366(4)	0.0719(19)
H28G	0.6001	-0.0197	0.2473	0.108
H28H	0.6489	0.0166	0.1752	0.108
H28I	0.4623	0.0233	0.2363	0.108

Tabelle 3: H-Atomkoordinaten und isotrope Auslenkungsparameter ($Å^2$) für sba105.
(Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for sba105.)

Atom	x	y	z	U_{eq}
H10	0.457(6)	0.2240(11)	0.357(3)	0.015(12)
H12A	0.8359	0.2853	0.4178	0.036
H12B	0.6285	0.2933	0.3392	0.036
H13A	0.7440	0.3589	0.3991	0.042
H13B	0.7579	0.3430	0.5064	0.042
H14	0.3696	0.3532	0.3737	0.040
H15A	0.4099	0.3249	0.5663	0.040
H15B	0.1857	0.3336	0.4984	0.040
H16A	0.2523	0.2788	0.3964	0.036
H16B	0.2660	0.2604	0.5017	0.036
H22A	0.5718	0.1547	0.2160	0.049
H22B	0.4238	0.1636	0.2944	0.049
H23A	0.5280	0.0998	0.3713	0.054
H23B	0.4447	0.0894	0.2617	0.054
H24	0.7949	0.0849	0.2344	0.046
H25A	0.9348	0.1005	0.4319	0.052
H25B	1.0892	0.0914	0.3567	0.052
H26A	1.0686	0.1654	0.3905	0.050
H26B	0.9955	0.1557	0.2796	0.050
H31	0.3768	0.1908	0.5378	0.037
H32A	0.2532	0.2172	0.6728	0.048
H32B	0.4114	0.1856	0.7398	0.048

H33A	0.0683	0.1545	0.7086	0.060
H33B	0.0619	0.1590	0.5964	0.060
H34A	0.1467	0.0887	0.6400	0.065
H34B	0.3397	0.1039	0.7203	0.065
H35A	0.3287	0.1106	0.5198	0.062
H35B	0.4850	0.0803	0.5906	0.062
H36A	0.6717	0.1394	0.6637	0.048
H36B	0.6664	0.1431	0.5514	0.048
H18A	0.5553	0.4440	0.5787	0.092
H18B	0.7174	0.4066	0.5629	0.092
H18C	0.5172	0.3966	0.6139	0.092
H18D	0.1921	0.4458	0.4899	0.111
H18E	0.1455	0.3979	0.5196	0.111
H18F	0.1089	0.4111	0.4098	0.111
H18G	0.5037	0.4618	0.4104	0.117
H18H	0.4198	0.4272	0.3302	0.117
H18I	0.6585	0.4242	0.3883	0.117
H28A	0.7093	-0.0109	0.4197	0.114
H28B	0.5667	0.0314	0.4168	0.114
H28C	0.8145	0.0327	0.4637	0.114
H28D	0.9837	-0.0182	0.3218	0.101
H28E	1.0963	0.0259	0.3578	0.101
H28F	1.0195	0.0178	0.2466	0.101
H28G	0.6001	-0.0197	0.2473	0.108
H28H	0.6489	0.0166	0.1752	0.108
H28I	0.4623	0.0233	0.2363	0.108

Tabelle 4: Anisotrope Auslenkungsparameter ($Å^2$) für sba105. Der Exponent für den anisotropen Auslenkungsparameter hat die Form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$
(Anisotropic displacement parameters ($Å^2$) for sba105. The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.020(3)	0.043(3)	0.013(2)	-0.003(2)	-0.0052(19)	-0.001(2)
N2	0.029(2)	0.048(2)	0.010(2)	0.0003(18)	-0.0068(17)	-0.0038(18)
N3	0.041(3)	0.051(3)	0.014(2)	0.0009(19)	-0.0152(19)	-0.004(2)
N4	0.049(3)	0.050(3)	0.021(2)	0.005(2)	-0.019(2)	-0.009(2)
N5	0.035(2)	0.048(2)	0.019(2)	0.0013(19)	-0.0165(19)	-0.0037(19)
N10	0.022(2)	0.049(2)	0.018(2)	-0.003(2)	-0.009(2)	0.0021(18)
C11	0.024(2)	0.040(2)	0.009(2)	-0.0004(19)	-0.0065(19)	-0.0010(19)
C12	0.023(3)	0.050(3)	0.014(3)	0.003(2)	-0.004(2)	-0.001(2)
C13	0.031(3)	0.045(3)	0.026(3)	-0.001(2)	-0.002(2)	-0.004(2)
C14	0.034(3)	0.047(3)	0.016(3)	0.000(2)	-0.002(2)	0.001(2)
C15	0.026(3)	0.049(3)	0.024(3)	-0.002(2)	0.002(2)	0.003(2)
C16	0.021(2)	0.050(3)	0.017(3)	0.000(2)	-0.003(2)	0.002(2)
C18	0.046(3)	0.048(3)	0.049(3)	-0.002(2)	0.010(3)	0.005(2)
N20	0.026(2)	0.044(2)	0.024(2)	-0.0013(19)	-0.0026(19)	0.0004(18)
C21	0.030(3)	0.043(3)	0.023(3)	-0.004(2)	0.001(2)	-0.001(2)
C22	0.038(3)	0.048(3)	0.031(3)	-0.010(2)	-0.009(2)	0.002(2)
C23	0.033(3)	0.051(3)	0.046(4)	-0.009(3)	-0.011(3)	-0.001(2)
C24	0.037(3)	0.047(3)	0.029(3)	-0.005(2)	-0.001(2)	0.003(2)
C25	0.027(3)	0.055(3)	0.045(3)	-0.009(3)	-0.001(2)	0.003(2)
C26	0.030(3)	0.056(3)	0.040(3)	-0.012(3)	0.005(2)	0.000(2)
C28	0.050(3)	0.047(3)	0.045(3)	-0.005(2)	0.006(3)	0.004(2)
C31	0.028(3)	0.046(3)	0.016(3)	0.001(2)	-0.005(2)	-0.006(2)
C32	0.035(3)	0.062(3)	0.025(3)	0.001(2)	0.005(2)	-0.001(2)
C33	0.038(3)	0.074(4)	0.039(3)	-0.002(3)	0.009(3)	-0.010(3)

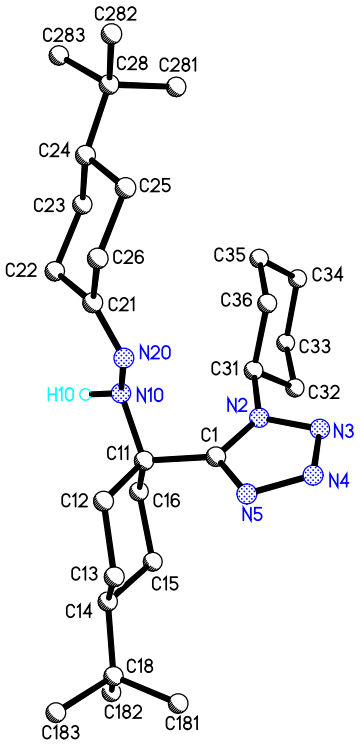
C34	0.052(3)	0.067(3)	0.044(4)	0.003(3)	0.008(3)	-0.014(3)
C35	0.042(3)	0.059(3)	0.052(4)	-0.003(3)	0.006(3)	-0.008(3)
C36	0.036(3)	0.051(3)	0.031(3)	-0.002(2)	0.001(3)	-0.003(2)
C181	0.068(4)	0.056(4)	0.060(4)	-0.017(3)	0.010(3)	-0.006(3)
C182	0.061(4)	0.059(4)	0.105(5)	-0.005(4)	0.019(3)	0.017(3)
C183	0.107(5)	0.056(4)	0.077(5)	0.009(3)	0.033(4)	0.002(3)
C281	0.113(5)	0.060(4)	0.061(4)	0.009(3)	0.034(4)	0.001(3)
C282	0.072(4)	0.059(4)	0.072(5)	-0.003(3)	0.014(3)	0.013(3)
C283	0.075(4)	0.054(4)	0.079(4)	-0.011(3)	-0.009(3)	-0.003(3)

Tabelle 5: Bindungslängen (Å) und -winkel (°) für sba105.
(Bond lengths (Å) and angles (deg) for sba105.)

C1-N5	1.325(5)	C28-C283	1.548(7)
C1-N2	1.342(5)	C31-C36	1.515(6)
C1-C11	1.519(6)	C31-C32	1.515(6)
N2-N3	1.362(5)	C31-H31	1.0000
N2-C31	1.466(5)	C32-C33	1.524(6)
N3-N4	1.301(5)	C32-H32A	0.9900
N4-N5	1.372(5)	C32-H32B	0.9900
N10-N20	1.409(5)	C33-C34	1.508(6)
N10-C11	1.473(6)	C33-H33A	0.9900
N10-H10	0.83(4)	C33-H33B	0.9900
C11-C16	1.521(5)	C34-C35	1.516(6)
C11-C12	1.531(6)	C34-H34A	0.9900
C12-C13	1.522(6)	C34-H34B	0.9900
C12-H12A	0.9900	C35-C36	1.525(6)
C12-H12B	0.9900	C35-H35A	0.9900
C13-C14	1.536(6)	C35-H35B	0.9900
C13-H13A	0.9900	C36-H36A	0.9900
C13-H13B	0.9900	C36-H36B	0.9900
C14-C15	1.539(6)	C181-H18A	0.9800
C14-C18	1.544(6)	C181-H18B	0.9800
C14-H14	1.0000	C181-H18C	0.9800
C15-C16	1.513(6)	C182-H18D	0.9800
C15-H15A	0.9900	C182-H18E	0.9800
C15-H15B	0.9900	C182-H18F	0.9800
C16-H16A	0.9900	C183-H18G	0.9800
C16-H16B	0.9900	C183-H18H	0.9800
C18-C182	1.534(7)	C183-H18I	0.9800
C18-C181	1.541(7)	C281-H28A	0.9800
C18-C183	1.543(7)	C281-H28B	0.9800
N20-C21	1.266(5)	C281-H28C	0.9800
C21-C26	1.496(6)	C282-H28D	0.9800
C21-C22	1.511(6)	C282-H28E	0.9800
C22-C23	1.517(6)	C282-H28F	0.9800
C22-H22A	0.9900	C283-H28G	0.9800
C22-H22B	0.9900	C283-H28H	0.9800
C23-C24	1.515(6)	C283-H28I	0.9800
C23-H23A	0.9900	N5-C1-N2	108.9(4)
C23-H23B	0.9900	N5-C1-C11	125.4(4)
C24-C25	1.532(6)	N2-C1-C11	125.7(4)
C24-C28	1.550(6)	C1-N2-N3	108.4(3)
C24-H24	1.0000	C1-N2-C31	133.4(4)
C25-C26	1.514(6)	N3-N2-C31	118.2(4)
C25-H25A	0.9900	N4-N3-N2	106.2(3)
C25-H25B	0.9900	N3-N4-N5	111.0(3)
C26-H26A	0.9900	C1-N5-N4	105.6(4)
C26-H26B	0.9900	N20-N10-C11	111.3(3)
C28-C281	1.522(7)	N20-N10-H10	115(3)
C28-C282	1.537(6)	C11-N10-H10	108(3)

N10-C11-C1	106.3(4)	C26-C25-C24	113.2(4)
N10-C11-C16	108.7(3)	C26-C25-H25A	108.9
C1-C11-C16	109.3(4)	C24-C25-H25A	108.9
N10-C11-C12	111.7(4)	C26-C25-H25B	108.9
C1-C11-C12	111.1(3)	C24-C25-H25B	108.9
C16-C11-C12	109.5(4)	H25A-C25-H25B	107.7
C13-C12-C11	115.7(4)	C21-C26-C25	111.4(4)
C13-C12-H12A	108.4	C21-C26-H26A	109.3
C11-C12-H12A	108.4	C25-C26-H26A	109.3
C13-C12-H12B	108.4	C21-C26-H26B	109.3
C11-C12-H12B	108.4	C25-C26-H26B	109.3
H12A-C12-H12B	107.4	H26A-C26-H26B	108.0
C12-C13-C14	112.7(4)	C281-C28-C282	108.8(5)
C12-C13-H13A	109.1	C281-C28-C283	109.6(5)
C14-C13-H13A	109.1	C282-C28-C283	106.4(4)
C12-C13-H13B	109.1	C281-C28-C24	112.3(4)
C14-C13-H13B	109.1	C282-C28-C24	110.3(4)
H13A-C13-H13B	107.8	C283-C28-C24	109.3(4)
C13-C14-C15	108.2(4)	N2-C31-C36	111.3(3)
C13-C14-C18	113.9(4)	N2-C31-C32	110.7(4)
C15-C14-C18	115.6(4)	C36-C31-C32	111.4(4)
C13-C14-H14	106.1	N2-C31-H31	107.8
C15-C14-H14	106.1	C36-C31-H31	107.8
C18-C14-H14	106.1	C32-C31-H31	107.8
C16-C15-C14	111.7(4)	C31-C32-C33	109.8(4)
C16-C15-H15A	109.3	C31-C32-H32A	109.7
C14-C15-H15A	109.3	C33-C32-H32A	109.7
C16-C15-H15B	109.3	C31-C32-H32B	109.7
C14-C15-H15B	109.3	C33-C32-H32B	109.7
H15A-C15-H15B	107.9	H32A-C32-H32B	108.2
C15-C16-C11	114.1(4)	C34-C33-C32	111.3(4)
C15-C16-H16A	108.7	C34-C33-H33A	109.4
C11-C16-H16A	108.7	C32-C33-H33A	109.4
C15-C16-H16B	108.7	C34-C33-H33B	109.4
C11-C16-H16B	108.7	C32-C33-H33B	109.4
H16A-C16-H16B	107.6	H33A-C33-H33B	108.0
C182-C18-C181	107.8(5)	C33-C34-C35	112.3(4)
C182-C18-C183	108.7(5)	C33-C34-H34A	109.1
C181-C18-C183	107.8(5)	C35-C34-H34A	109.1
C182-C18-C14	110.7(4)	C33-C34-H34B	109.1
C181-C18-C14	111.8(4)	C35-C34-H34B	109.1
C183-C18-C14	110.0(4)	H34A-C34-H34B	107.9
C21-N20-N10	117.4(4)	C34-C35-C36	111.0(4)
N20-C21-C26	118.1(4)	C34-C35-H35A	109.4
N20-C21-C22	128.1(4)	C36-C35-H35A	109.4
C26-C21-C22	113.6(4)	C34-C35-H35B	109.4
C21-C22-C23	109.8(4)	C36-C35-H35B	109.4
C21-C22-H22A	109.7	H35A-C35-H35B	108.0
C23-C22-H22A	109.7	C31-C36-C35	110.6(4)
C21-C22-H22B	109.7	C31-C36-H36A	109.5
C23-C22-H22B	109.7	C35-C36-H36A	109.5
H22A-C22-H22B	108.2	C31-C36-H36B	109.5
C24-C23-C22	114.6(4)	C35-C36-H36B	109.5
C24-C23-H23A	108.6	H36A-C36-H36B	108.1
C22-C23-H23A	108.6	C18-C181-H18A	109.5
C24-C23-H23B	108.6	C18-C181-H18B	109.5
C22-C23-H23B	108.6	H18A-C181-H18B	109.5
H23A-C23-H23B	107.6	C18-C181-H18C	109.5
C23-C24-C25	108.6(4)	H18A-C181-H18C	109.5
C23-C24-C28	115.1(4)	H18B-C181-H18C	109.5
C25-C24-C28	114.6(4)	C18-C182-H18D	109.5
C23-C24-H24	105.9	C18-C182-H18E	109.5
C25-C24-H24	105.9	H18D-C182-H18E	109.5
C28-C24-H24	105.9	C18-C182-H18F	109.5

H18D-C182-H18F	109.5
H18E-C182-H18F	109.5
C18-C183-H18G	109.5
C18-C183-H18H	109.5
H18G-C183-H18H	109.5
C18-C183-H18I	109.5
H18G-C183-H18I	109.5
H18H-C183-H18I	109.5
C28-C281-H28A	109.5
C28-C281-H28B	109.5
H28A-C281-H28B	109.5
C28-C281-H28C	109.5
H28A-C281-H28C	109.5
H28B-C281-H28C	109.5
C28-C282-H28D	109.5
C28-C282-H28E	109.5
H28D-C282-H28E	109.5
C28-C282-H28F	109.5
H28D-C282-H28F	109.5
H28E-C282-H28F	109.5
C28-C283-H28G	109.5
C28-C283-H28H	109.5
H28G-C283-H28H	109.5
C28-C283-H28I	109.5
H28G-C283-H28I	109.5
H28H-C283-H28I	109.5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N20
H10
N10
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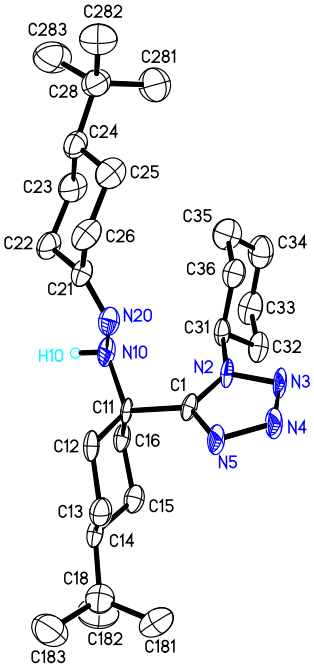
C15
C14
C18
C182
C183
C181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N20
H10
N10
N2
N3
C1
C11
N4
C12
C16
N5
C13
C15
C14
C18
C182
C183
C181

5d: colourless crystal (needle), dimensions 0.420 x 0.050 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/n$, Z=4, a=6.345(2) Å, b=31.355(11) Å, c=14.286(5) Å, alpha=90 deg, beta=99.872(11) deg, gamma=90 deg, V=2799.8(17) $Å^3$, rho=1.083 g/cm^3, T=200(2) K, $Theta_{max}$= 21.260 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.63and a completeness of 61.5% to a resolution of 0.98Å, 11459 reflections measured, 3120 unique (R(int)=0.1049), 1898 observed ($I > 2\sigma(I)$), intensities were corrected for Lorentz and polarization effects, an empirical absorption correction was applied using SADABS[1] based on the Laue symmetry of the reciprocal space, mu=0.06mm^{-1}, T_{min}=0.82, T_{max}=0.96, structure solved by direct methods and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXTL (Version 2013/3) software package[2], 302 parameters refined, hydrogen atoms were treated using appropriate riding models, except H10 at N10, which was refined isotropically, goodness of fit 1.01 for observed reflections, final residual values R1(F)=0.074, $wR(F^2)$=0.159 for observed reflections, residual electron density -0.33 to 0.32 $eÅ^{-3}$. CCDC 1030931 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Lit. 1: (program SADABS 2012/1 for absorption correction) G. M. Sheldrick, Bruker Analytical X-ray-Division, Madison, Wisconsin 2012

Lit. 2: (software package SHELXTL 2013/4 for structure solution and refinement) Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Lit. APEX, APEX2, SMART, SAINT, SAINT-Plus: Bruker (2007). "Program name(s)". Bruker AXS Inc., Madison, Wisconsin, USA.