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RESEARCH ARTICLE

Research in processes of the nitrogen-containing heterocycles obtaining from intermediate of the off-spec fuel disposal — 1,1-dimethyl-2-methylenehydrazone

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Abstract

Objectives. To introduce the intermediate obtained from the disposal of unusable rocket propellant—1,1-dimethyl-2-methylenehydrazone (DMH)—into the synthesis of pyrroloquinolines (and pyridines) with potential applications in medicine; to carry out reactions of DMH with tetracyanoethylated ketones (TCEKs) derived from acetone, methyl ethyl ketone, cyclohexanone, 4-propylcyclohexanone, and 2-methylcyclohexanone; to investigate the prospects for increasing the yields of target products by performing the same syntheses under microwave irradiation (MWI) and using an ultrasonic reactor.

Methods. TCEKs were prepared from tetracyanoethylene (TCNE) and the corresponding ketone in dioxane, acetone, or ethanol, with the presence of concentrated hydrochloric or sulfuric acid as a catalyst. Pyrroloquinolines (and pyridines) were synthesized from DMH and the corresponding TCEK in ethyl acetate with base as a catalyst. Syntheses were carried out under standard conditions using a magnetic stirrer, an ultrasonic reactor (*Vologda*, Russia), and a UWave-2000 microwave reactor (*Sineo Microwave Chemistry Technology Co*., China). Reaction progress and product purity were monitored by thin-layer chromatography on Sorbfil plates (*Sorbfil*, Russia). The TCNE presence in the reaction mixture was determined by the hydroquinone test. Melting and decomposition points were measured using an OptiMelt MPA100 apparatus (*OptiMelt*, USA). Structural identification was performed by infrared spectroscopy (FSM-1202, *SpektroLab*, Russia), ^{1}H and ^{13}C nuclear magnetic resonance spectroscopy in dimethyl sulfoxide d_6 on a Bruker AVANCE 400 WB spectrometer (*Bruker Corporation*, USA), and mass spectrometry using a quadrupole time-of-flight AB SCIEX TripleTOF 5600 spectrometer (*AB SCIEX PTE. Ltd*., Singapore) and a quadrupole gas chromatography–mass spectrometer GCMS-QP2020 NX (*Shimadzu*, Germany).

Results. Reliable procedures developed for the synthesis of pyrrolopyridines from acetone and methyl ethyl ketone without tar formation under ultrasonic stirring and MWI demonstrated significantly increased yields. The highest conversion of DMH to pyrroloquinoline was achieved from cyclohexanone, providing the target product in 92% yield within the shortest reaction time of 2 min under MWI conditions. However, for derivatives of 4-propylcyclohexanone and 2-methylcyclohexanone, the described synthesis modifications did not give the desired results: in the former case, a decrease in yield was observed as compared to standard methods, while in the latter, only a slight increase was obtained.

Conclusions. Ultrasonic stirring and MWI are effective for the conversion of DMH into pyrrolopyridines based on aliphatic TCEKs, but unsuitable for the synthesis of pyrroloquinolines derived from 4-propylcyclohexanone and 2-methylcyclohexanone. The high yield of pyrroloquinoline from cyclohexanone (92%) suggests potential for implementing the rapid MWI-promoted reaction between DMH and cyclohexanone-based TCEK in industrial production.

Keywords

1,1-dimethyl-2-methylenehydrazine, tetracyanoethylated ketones, tetracyanoethylene, tetracyanoethylation, pyrroloquinolines, pyrrolopyridines, microwave radiation, ultrasonic stirring

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НАУЧНАЯ СТАТЬЯ

Исследование процессов получения азотосодержащих гетероциклов из полупродукта утилизации некондиционного топлива — 1,1-диметил-2-метиленгидразона

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Аннотация

Цели. Ввести полупродукт утилизации непригодного к использованию ракетного топлива — 1,1-диметил-2-метиленгидразона (ДМГ) в синтез пирролохинолинов(пиридинов), потенциально применимых в медицине. Провести реакции МДГ с тетрацианоэтилированными кетонами (ТЦК) на основе ацетона, метилэтилкетона, циклогексанона, 4-пропилциклогексанона, 2-метилциклогексанона. Исследовать перспективы увеличения выходов целевых продуктов путем осуществления тех же синтезов при микроволновом излучении и с использованием ультразвукового реактора.

Методы. ТЦК были получены из тетрацианоэтилена (ТЦЭ) и соответствующего кетона в диоксане, ацетоне или этаноле, под катализом концентрированных кислот — соляной или серной. Пирролохинолины(пиридины) синтезированы из ДМГ и соответствующего ТЦК в этилацетате с каталитическими количествами щелочи. Синтезы проводились на магнитной мешалке при стадартных условиях, в ультразвуковом реакторе производства «*Вологда*» (Россия) и в микроволновой печи UWave-2000 марки Sineo (*Sineo Microwave Chemistry Technology Co*., Китай). Протекание реакции и чистоту продуктов контролировали методом тонкослойной хроматографии на пластинах Sorbfil (*Sorbfil*, Россия). Присутствие ТЦЭ в реакционной смеси определяли пробой на гидрохинон. Температуры плавления и разложения определены на приборе OptiMelt MPA100 (*OptiMelt*, США). Структуры идентифицированы методами инфракрасной спектроскопии на приборе ФСМ-1202 (*СпектроЛаб*, Россия), ядерно-магнитного резонанса ^{1}H и ^{13}C в диметилсульфоксиде d_6 на спектрометре Bruker AVANCE 400 WB (*Bruker Corporation*, США), масс-спектроскопии с использованием квадрупольного времяпролетного масс-спектрометра AB SCIEX TripleTOF 5600 (*AB SCIEX PTE. Ltd*., Сингапур) и квадрупольного газового хромато-масс-спектрометра GCMS-QP2020 NX (*Shimadzu*, Германия).

Результаты. Разработаны надежные методики получения пирролопиридинов на основе ацетона и метилэтилкетона без осмола в условиях ультразвукового перемешивания и микроволнового излучения (МВИ) со значительным увеличением выходов. Достигнута наивысшая степень конверсии ДМГ в пирролохинолин из циклогексанона с выходом целевого продукта 92% за наименьший промежуток времени 2 мин при МВИ. Однако для производных 4-пропилциклогексанона и 2-метилциклогексанона подобные модификации синтезов не привели к желаемым результатам, в первом случае наблюдалось уменьшение выходов в сравнении со стандартными условиями, во втором — лишь незначительное увеличение.

Выводы. Ультразвуковое перемешивание и МВИ эффективны в конверсии ДМГ в пирролопиридины на основе алифатических ТЦК и нерезультативны для пирролохинолинов на основе 4-пропилциклогексанона и 2-метилциклогексанона. Высокий выход пирролохинолина на основе циклогексанона (92%) позволяет рассматривать перспективы внедрения быстропротекающей при действии МВИ реакции ДМГ и ТЦК на основе циклогексанона в производство.

Ключевые слова

1,1-диметил-2-метиленгидразон, тетрацианоэтилированные кетоны, тетрацианоэтилен, тетрацианоэтилирование, пирролохинолины, пирролопиридины, микроволновое излучение, ультразвуковое перемешивание

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ВВЕДЕНИЕ

In our previous publications [1–4], we highlighted the problem of disposing of water-contaminated rocket propellants, which are unsuitable for missile launch purposes. At present, this issue has become more urgent due to the transition of many rocket systems to gas-based fuels[1,2] [5]. An established industrial method for the destruction of such waste is known [1–4]. This approach involves several stages: (1) preparation of low-toxic monomer 1,1-dimethyl-2-methylenehydrazone (DMH)—the methylene derivative—by treatment of off-specification propellant with a 40% formalin solution; (2) acetic acid-catalyzed polymerization of DMH to resin formation; (3) combustion of the resulting resin. We proposed to replace stages (2) and (3) of this process with the reaction of monomer DMH with tetracyanoethylated ketones (TCEKs) [1–3]. Remarkably, simple mixing of the initial reactants leads to the precipitation of complex heterocyclic compounds of the pyrroloquinoline [1–3] and pyrrolopyridine [2] classes. The frameworks of the obtained structures are closely related to biologically active compounds and known pharmaceutical agents [1–3]. On this basis, we anticipate their various potential applications in medicine or in field of drug-oriented organic synthesis. Our interest was also motivated by the possibility of rapid, facile, and comparatively safe disposal of DMH. Nevertheless, we encountered certain difficulties during the syntheses. For example, while cyclohexanone-derived TCEKs readily afford the desired products upon reaction with DMH, aliphatic ketone derivatives frequently undergo resinification prior to the target transformation. Under identical reaction conditions, aliphatic TCEKs may either furnish the expected heterocycles or result in polymerization of the starting compounds. In order to stabilize the methodology for aliphatic TCEKs and to increase the conversion of DMH, we experimentally carried out the reactions under conditions of ultrasonic (US) stirring [6, 7] and microwave irradiation (MWI)[3] [8, 9].

MATERIALS AND METHODS

The reagents, all of which were procured from commercial vendors and employed without undergoing additional purification, include: 1,1-dimethyl-2-methylenehydrazone 98% (CAS 2035-89-4), 4-propylcyclohexanone 98% (CAS 40649-36-3), 2-methylcyclohexanone 98% (CAS 583-60-8), ethyl acetate 98% (CAS 141-78-6), tetracyanoethylene (TCNE) 98% (CAS 670-54-2), hydrochloric acid 35–38% (CAS 7647-01-0), sulfuric acid 93.6–95.6% (CAS 7664-93-9).

US stirring was conducted with the use of US reactor (*Vologda*, Russia) [6, 7]. MWI syntheses were carried out in microwave multifunctional system of microwave syntheses and extraction, UWave-2000 (*Sineo Microwave Chemistry Technology Co*., Shanghai, China) [9, 10], supplied with 50-mL quartz reaction vessels, under atmospheric pressure and working environment temperature 40°C. The progress of reactions and the purity of products were monitored via thin-layer chromatography (TLC) on Sorbfil plates (*Sorbfil,* Krasnodar, Russia).

Visualization of spots was achieved under ultraviolet (UV) light, upon treatment with iodine vapor, or through heating. Melting and decomposition points were determined using the OptiMelt MPA100 apparatus (*OptiMelt*, Danbury, Connecticut, USA). Infrared (IR) spectra were obtained using the FSM-1202 spectrometer (*SpektroLab*, Russia) equipped with IR Fourier transform technology, with samples dispersed in Nujol (*Schering-Plough*, USA). Proton nuclear magnetic resonance (^{1}H NMR) and carbon-13 nuclear magnetic resonance (^{13}C NMR) spectra were acquired with the provision of the Carr–Purcell–Meiboom–Gill (CPMG) pulse sequence, employing dimethyl sulfoxide d_6 (DMSO-d_6) solvent and utilizing the tetramethylsilane internal

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[2] Weichert B.J. Russia Is Building a Reusable Methane-Powered Rocket for Space Missions. *The National Interest*. URL: https://nationalinterest.org/blog/buzz/russia-building-reusable-methane-powered-rocket-space-bw. Accessed July 14, 2025.

[3] UWave-2000. Multifunctional Microwave Chemistry Reaction Workstation. *Sineo Microwave Chemistry Technology Co.*, *Ltd*, 2022. https://www.sineomicrowave.com/product/uwave-2000-multifunctional-microwave-chemistry-reaction-workstation. Accessed January 20, 2024.

standard. The measurements were conducted on a Bruker AVANCE 400 WB spectrometer (*Bruker Corporation*, Hanau, Germany) operating at the frequencies of 400.13 MHz for ^{1}H and 100.61 MHz for ^{13}C.

High resolution mass spectrometry (HRMS) of pyrroloquinolines derived from cyclic TCEKs was carried out using the quadrupole time-of-flight AB SCIEX TripleTOF 5600 mass spectrometer (*AB SCIEX PTE. Ltd*., Singapore) equipped with a turbo-ion spray source. The nebulizer gas used was nitrogen; the ionization polarity was positive (+). The needle voltage was set at 5500 V. The spectra were recorded in the time-of-flight mass spectrometry mode with a collision energy of 10 eV, declustering potential of 100 eV, and a resolution exceeding 30000 full width at half maximum. Sample solutions with an analyte concentration of 5 μmol/L were prepared by dissolving the test compounds in methanol hypergrade for liquid chromatography–mass spectrometry (*Merck*, Darmstadt, Germany).

An analytical reversed-phase high-performance liquid chromatography (HPLC) was used for determining the uncalibrated purity of pyrroloquinolines derived from cyclic TCEKs and conducted using an Atlantis T3 C18 column (*Waters Corporation*, USA) (5 μm, 150 × 4.6 mm); eluent A, 1.2% solution of triethylamine in water; eluent B CH_3CN; and the gradient elution (0 min A : B = 85 : 15; 15 min A : B = 65 : 35; 21 min A : B = 65 : 35) flow rate was 1.0 mL/min. HPLC analysis was performed at 40°C during 21 min at 311 nm.

Mass spectra of pyrrolopyridines derived from aliphatic TCEKs were acquired using the quadrupole gas chromatography–mass spectrometer GCMS-QP2020 NX (*Shimadzu*, Duisburg, Germany). Gas chromatograph was equipped with the column SH-I-5MS. Dimethyl polysiloxane was utilized as the stationary phase. A sample solution with an analyte concentration of 5 μmol/L was prepared by dissolving the test compound in isopropanol.

Syntheses of TCEKs based on cyclohexanone, 4-propylcyclohexanone, and 2-methylcyclohexanone

The solution of 1.25 mmol TCNE in 5 mL of dioxane was added to the solution of 1.22 mmol of appropriative ketone in 5 mL of dioxane along with a catalytic amount of hydrochloric acid (1 drop). The progress of the process was determined via a hydroquinone (HQ) test that forms a blue *p*-complex when reacting with TCNE. After the blue color ceased to appear, the dioxane solution was maintained at the temperature range of 0–5°C in the freezer for 10 min. After this period of time, cold distilled water was added to the frozen reaction mixture in a volume equal to the dioxane solution. The reaction mixture was stirred until the precipitation. The desired product was separated by filtration through a Schott filter, followed by washing with cold distilled water.

1-(2-Oxocyclohexyl)ethane-1,1,2,2-tetracarbonitrile **2a** [cyclohexanone + TCNE]. Yield: 74% (0.2 g).

1-(2-Oxo-5-propylcyclohexyl)ethane-1,1,2,2-tetracarbonitrile **2b** [4-propylcyclohexanone + TCNE]. Yield: 75% (0.27 g).

1-(1-Methyl-2-oxocyclohexyl-)ethane-1,1,2,2-tetracarbonitrile **2c** [2-methylcyclohexanone + TCNE]. Yield: 66% (0.2 g).

Synthesis of 4-oxopentane-1,1,2,2-tetracarbonitrile [acetone + TCNE]

TCNE (3.9 mmol) was dissolved in acetone (5 mL) containing a catalytic amount of hydrochloric acid. The reaction mixture was stirred at ambient temperature for 5 h, after which the HQ test indicated complete consumption of the starting material. The solvent was then combined with deionized water (15 mL), resulting in precipitation. The solid was collected by vacuum filtration through a Schott glass filter, washed successively with cold water and diethyl ether, and dried under reduced pressure. Yield of **2d** is 70% (0.51 g).

Synthesis of 4-oxohexane-1,1,2,2-tetracarbonitrile [methylethylketone + TCNE]

TCNE (50 mmol) was dissolved in methyl ethyl ketone (20 mL) and absolute ethanol (20 mL). The reaction mixture was stirred on a rotary evaporator at 45°C for 2–3 h, until the HQ test confirmed complete consumption of the starting material. The solvents were then removed by distillation at 36°C, affording a viscous resinous residue. The residue was triturated with deionized water (200 mL), and the resulting solid was isolated by filtration and purified by recrystallization from ethyl acetate. Yield of **2e** is 80% (8.00 g).

Synthesis of methyl-4-oxopentane-1,1,2,2-tetracarbonitrile [methylethylketone + TCNE]

TCNE (50 mmol) was dissolved in methyl ethyl ketone (30 mL) and absolute ethanol (20 mL) in the presence of a catalytic amount of sulfuric acid. The reaction mixture was stirred on a rotary evaporator at 30°C for 5 min. Ethanol was then removed by evaporation, and the resulting solid residue was triturated with deionized

water to afford a white flocculent precipitate. Yield of **2f** is 72% (7.20 g).

Syntheses of pyrroloquinolines(pyridines) under magnetic stirring and standard conditions

DMH (8 mmol) was carefully added to a solution of TCNE (8 mmol) along with a catalytic amount of sodium hydroxide in 5 mL of ethyl acetate. The reaction was stirring till sand-colored crystals precipitated. The desired product was separated by filtration through a Schott filter, followed by washing with cold ethyl acetate. The obtained compounds were purified by the following methods: pyrroloquinolines (derivatives of propylcyclohexanone, 4-propylceclohexanone, 2-methylcyclohexanone) by recrystallization from hot isopropanol; pyrrolopyridines (derivatives of acetone and methyletylketone) by recrystallization from hot mixture EtOAc/isopropanol with 1 mg of activated coal with subsequent flash chromatography (column length is 1 cm).

3-Amino-5-(dimethylamino)-1-oxo-4,5,6,7,8,9-hexahydro-1*H*-pyrrolo[3,4-c]quinoline-3a,9b-dicarbonitrile **3a** [cyclohexanone TCEK + DMH]. Yield: 84% (2.00 g).

3-Amino-5-(dimethylamino)-1-oxo-8-(propyl)-4,5,6,7,8,9-hexahydro-1*H*-pyrrolo[3,4-c]quinoline-3a,9b-dicarbonitrile **3b** [4-propylcyclohexanone TCEK + DMH]. Yield: 75% (2.04 g).

2-(Dimethylamino)-4a-methyl-10-oxo-5,6,7,8-tetrahydro-1*H*-8a,4-(epimino)quinoline-3,4(4aH)-dicarbonitrile **3c** [2-methylcyclohexanone TCEK + DMH]. Yield: 48% (1.09 g).

3-Amino-5-dimethylamino-6-methyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine-3a,7a-dicarbonitrile **3d** [acetone TCEK + DMH]. Yield: 57% (1.17 g).

3-Amino-5-dimethylamino-6-ethyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine-3a,7a-dicarbonitrile **3e** [methylethylketone TCEK + DMH]. Yield: 54% (1.18 g).

3-Amino-5-dimethylamino-6,7-dimethyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine3a,7a-dicarbonitrile **3f** [methylethylketone TCEK + DMH]. Yield: 52% (1.13 g).

Syntheses of pyrroloquinolines(pyridines) under US stirring

DMH (8 mmol) was carefully added to a solution of TCNE (8 mmol) in ethyl acetate containing HQ (1 mg), catalytic amount of sodium hydroxide in a vessel equipped with a copper ring and immersed in an ice bath. The mixture was stirred at 10°C in an US reactor for 10–30 min, until the reaction was complete as indicated by TLC. The resulting precipitate was collected by vacuum filtration through a Schott glass filter and washed with chilled ethyl acetate.

3-Amino-5-(dimethylamino)-1-oxo-4,5,6,7,8,9-hexahydro-1*H*-pyrrolo[3,4-c]quinoline-3a,9b-dicarbonitrile **3a** [cyclohexanone TCEK + DMH]. Yield: 79% (1.88 g).

3-Amino-5-(dimethylamino)-1-oxo-8-(propyl)-4,5,6,7,8,9-hexahydro-1*H*-pyrrolo[3,4-c]quinoline-3a,9b-dicarbonitrile **3b** [4-propylcyclohexanone TCEK + DMH]. Yield: 60% (1.63 g).

2-(Dimethylamino)-4a-methyl-10-oxo-5,6,7,8-tetrahydro-1*H*-8a,4-(epimino)quinoline-3,4(4a*H*)-dicarbonitrile **3c** [2-methylcyclohexanone TCEK + DMH]. Yield: 50% (1.14 g).

3-Amino-5-dimethylamino-6-methyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine-3a,7a-dicarbonitrile **3d** [acetone TCEK + DMH]. Yield: 73% (1.50 g).

3-Amino-5-dimethylamino-6-ethyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine-3a,7a-dicarbonitrile **3e** [methylethylketone TCEK + DMH]. Yield: 62% (1.35 g).

3-Amino-5-dimethylamino-6,7-dimethyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine3a,7a-dicarbonitrile **3f** [methylethylketone TCEK + DMH]. Yield: 67% (1.46 g).

Syntheses of pyrroloquinolines(pyridines) under MWI

DMH (8 mmol) was carefully added to a solution of TCNE (8 mmol) in ethyl acetate along with a catalytic amount of sodium hydroxide. The reaction vessel was placed in a refrigerator for 10 min to lower the temperature to 5–10°C, after which the mixture was subjected to MWI for 2–5 min with stirring. The resulting precipitate was collected by vacuum filtration through a Schott glass filter and washed with chilled ethyl acetate.

3-Amino-5-(dimethylamino)-1-oxo-4,5,6,7,8,9-hexahydro-1*H*-pyrrolo[3,4-c]quinoline-3a,9b-dicarbonitrile **3a** [cyclohexanone TCEK + DMH]. Yield: 92% (2.19 g).

3-Amino-5-(dimethylamino)-1-oxo-8-(propyl)-4,5,6,7,8,9-hexahydro-1*H*-pyrrolo[3,4-c]quinoline-3a,9b-dicarbonitrile **3b** [4-propylcyclohexanone TCEK + DMH]. Yield: 62% (1.69 g).

2-(Dimethylamino)-4a-methyl-10-oxo-5,6,7,8-tetrahydro-1*H*-8a,4-(epimino)quinoline-3,4(4a*H*)-dicarbonitrile **3c** [2-methylcyclohexanone TCEK + DMH]. Yield: 52% (1.19 g).

3-Amino-5-dimethylamino-6-methyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine-3a,7adicarbonitrile **3d** [acetone TCEK + DMH]. Yield: 82% (1.69 g).

3-Amino-5-dimethylamino-6-ethyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine-3a,7adicarbonitrile **3e** [methylethylketone TCEK + DMH]. Yield: 70% (1.53 g).

3-Amino-5-dimethylamino-6,7-dimethyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine3a,7a-dicarbonitrile **3f** [methylethylketone TCEK + DMH]. Yield: 85% (1.85 g).

Characteristics of the obtained heterocycles

Characteristics of compounds **3a–c** are presented in [1] and patent RU 2802332 C1 [3]. The same for products **3d–f** is in [2].

3-Amino-5-(dimethylamino)-1-oxo-4,5,6,7,8,9-hexahydro-1*H*-pyrrolo[3,4-c]quinoline-3a,9b-dicarbonitrile **3a** [cyclohexanone TCEK + DMH]. T_{melt} = 217–219°C. IR (Nujol), ν_{max}/cm^{-1}: 1659 (s) (C=C), 1736 (vs) (C=O), 2241 (w) (C≡N), 3291 (vs, br) (NH_2). ^{1}H NMR spectrum, δ, ppm: 1.53–1.29 (m, 2H, CH_2^8), 1.65–1.54 (m, 2H, CH_2^9), 2.24–2.10 (m, 2H, CH_2^7), 2.28 (m, 2H, CH_2^{10}), 2.33 (s, 6H, $N(CH_3)_2$), 3.34 (d, J = 11.75 Hz, 1H, CH_2^4), 3.73 (d, J = 11.67 Hz, 1H, CH_2^4), 9.63 (d, J = 30.32 Hz, 2H, NH_2) (see Fig. 1, p. 313 at the Supplementary Section). ^{13}C NMR, δ_C, ppm: 21.88 (CH_2^{10}), 22.01 (CH_2^9), 24.55(CH_2^8), 24.92 (CH_2^7), 39.52 ($N(CH_3)_2$), 51.08 ($CH_2{}^4$), 52.94 (C^{12}), 59.76 (C^3), 95.07 (C^{11}), 115.24 (C≡N^{16}), 116.16 (C≡N^{21}), 144.94 (C^6), 177.07 (C^2), 178.15 (C=O) (see Fig. 2, p. 313 at the Supplementary Section). Found: C 60.28; H 6.31; N 28.05. $C_{15}H_{18}N_6O$. Calculated, %: C 60.39; H 6.08; N 28.17. HRMS (electrospray ionization, ESI) and mass-spectrum, *m/z*: $[M + H]^+$ 299.1620 (calculated for $[C_{15}H_{19}N_6O]^+$ 299.1620) (see Fig. 3, p. 314 at the Supplementary Section).

Amino-5-(dimethylamino)-1-oxo-8-(propyl)-4,5,6,7,8,9-hexahydro-1*H*-pyrrolo[3,4-c]quinoline-3a,9b-dicarbonitrile **3b** [4-propylcyclohexanone TCEK + DMH]. T_{melt} = 214–216°C. IR (Nujol), ν_{max}/cm^{-1}: 1640 (s) (C=C), 1745 (s) (C=O), 2251 (s) (C≡N), 3494 (vs, br) (NH_2) ^{1}H NMR spectrum, δ, ppm: 0.88 (t, J = 7.01 Hz, 3H, CH_3), 1.03 (s, 3H, NCH_3^{18}), 1.04 (s, 3H, NCH_3^{17}), 1.28–1.17 (m, 2H, CH_2^{15}), 1.38–1.28 (m, 2H, CH_2^{14}), 1.49–1.42 (m, 2H, CH^{11}), 1.72 (m, 2H, CH_2^{12}), 1.91 (t, J = 12.45 Hz, 2H, CH_2^{13}), 2.47 (m, 2H, CH_2^{10}), 3.20 (d, J = 11.63 Hz, 1H, CH_2^6), 3.96 (d, J = 11.66 Hz, 1H, CH_2^6), 9.61 (d, J = 15.42 Hz, 2H, NH_2) (see Fig. 4, p. 314 at the Supplementary Section). ^{13}C NMR, δ_C, ppm: 14.18 (CH_3), 19.63 (CH_2^{15}), 25.33 ($(CH_2^{10, 12})_2$), 28.41 (CH_2^{13}), 31.91 (CH_2^{14}, CH^{11}), 37.85 (CH^6), 39.55 $(N(CH_3)_2^{17, 18})$, 51.57 (CH_2^6), 53.33 (C^3), 62.07 (C^4), 94.70 (C^8), 115.30 (C≡N^{19}), 116.19 (C≡N^{20}), 145.03 (C^9), 177.15 (C^5), 178.07 (C=O) (see Fig. 5, p. 315 at the Supplementary Section). Found: C 63.48; H 7.13; N 24.65. $C_{18}H_{24}N_6O$. Calculated, %: C 63.51; H 7.11; N 24.69. HRMS(ESI), *m/z*: $[M+H]^+$ 341.2090 (calculated for $[C_{18}H_{25}N_6O]^+$ 341.2090) (see Fig. 6, p. 315 at the Supplementary Section).

2-(Dimethylamino)-4a-methyl-10-oxo-5,6,7,8-tetrahydro-1*H*-8a,4-(epimino)quinoline-3,4(4a*H*)-dicarbonitrile **3c** [2-methylcyclohexanone TCEK + DMH] T_{melt} > 250°C. IR (Nujol), ν_{max}/cm^{-1}: 1579 (s) (C=C), 1717 (vs) (C=O), 2168 (vs) (=C–C≡N), 2242 (w) (C≡N), 3167 (br), 3282 (w) (NH). ^{1}H NMR spectrum, δ, ppm: 0.89 (s, 3H, CH_3), 2.03–1.07 (m, 8H, $(CH_2^{7-12})_4$), 2.91 (s, 6H, $N(CH_3)_2$), 7.06 (s, 1H, NH^6), 8.42 (s, 1H, NH^1) (see Fig. 7, p. 316 at the Supplementary Section). ^{13}C NMR, δ_C, ppm: 13.72 (CH_3), 20.06 (CH_2^{10}), 20.86 (CH_2^9), 26.66 (CH_2^{11}), 31.86 (CH_2^8), 40.09 $(N(CH_3)_2^{19, 20})$, 40.67 (C^3), 51.62 (C^{12}), 54.29 (C^7), 71.98 (C^5), 115.88 (C≡N^{11}), 120.99 (C≡N^{14}), 157.75 (C^5), 168.68 (C=O) (see Fig. 8, p. 316 at the Supplementary Section). Found: C 63.08; H 6.69; N 24.55. $C_{15}H_{19}N_5O$. Calculated, %: C 63.14; H 6.71; N 24.54. HRMS(ESI), *m/z*: $[M + H]^+$ 286.1668 (calculated for $[C_{15}H_{20}N_5O]^+$ 286.1668) (see Fig. 9, p. 317 at the Supplementary Section).

3-Amino-5-dimethylamino-6-methyl-1-oxo-4,5-dihydro-1H-pyrrolo[3,4-c]pyridine-3a,7a-dicarbonitrile **3d** [acetone TCEK + DMH]. $T_{melt.}$ = 160–161°C. IR (Nujol), ν_{max}/cm^{-1}: 1651 (s) (C=C), 1750 (s) (C=O), 2257 (vs) (C≡N), 3480 (vs, br) (NH_2). ^{1}H NMR spectrum, δ, ppm: 1.80–2.10 (m, 3H, CH_3^7), 2.33 (s, 6H, $N(CH_3)_2$), 3.35 (d, J = 9.2 Hz, 2H, CH_2^6), 3.73 (d, J = 11.7 Hz, 1H, CH^3), 9.63 (d, J = 78.4 Hz, 2H, NH_2^{17}) (see Fig. 10, p. 317 at the Supplementary Section). ^{13}C NMR, δ_C, ppm: 21.88 (CH_3^{16}), 40.15 ($N(CH_3)_2$), 51.08 (CH_2^6), 52.94 (C^4), 59.76 (C^5), 95.07 (CH^3), 115.24 (CN^{13}), 116.16 (CN^{12}), 144.94 (C^2), 177.07 (C^9), 178.27 (C=O) (see Fig. 11, p. 318 at the Supplementary Section). Found: C 56.01; H 5.33; N 32.75. $C_{12}H_{14}N_6O$. Calculated, %: C 55.80; H 5.46; N 32.54. Mass spectrum, *m/z* (I_{rel}, %): $C_{12}H_{14}N_6O$ 258.12 (100%).

3-Amino-5-dimethylamino-6-ethyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine-3a,7a-dicarbonitrile **3e** [methylethylketone TCEK + MH]. T_{melt} = 165–166°C. IR (Nujol), ν_{max}/cm^{-1}: 1639 (s) (C=C), 1745 (s) (C=O), 2260 (s) (C≡N), 3395 (vs, br) (NH_2). ^{1}H NMR spectrum, δ, ppm: 0.88 (t, J = 7.1 Hz, 3H, CH_3^{20}), 1.03 (d, J = 6.2 Hz, 6H, $N(CH_3)_2$), 1.17–1.37 (m, 2H, CH_2^7), 3.00–4.08 (m, 2H, CH_2^6), 4.35 (d, J = 4.2 Hz, 1H, CH^3), 9.61 (d, J = 115.6 Hz, 2H, NH_2) (see Fig. 12, p. 318 at the Supplementary Section). ^{13}C NMR, δ_C, ppm: 14.18 (CH_3^{20}), 19.63 (CH_2^{16}), 40.17 ($N(CH_3)_2$), 51.57 (CH_2^6), 53.33 (C^4), 62.07 (C^5), 94.70 (CH^3), 115.30 (CN^{13}), 116.19 (CN^{12}), 145.03 (C^2), 177.15 (C^9), 178.07 (C=O) (see Fig. 13, p. 319 at the Supplementary Section). C 57.21; H 6.08; N 30.56. $C_{13}H_{16}N_6O$. Calculated, %: C 57.34; H 5.92; N 30.86. Mass spectrum, *m/z* (I_{rel}, %): $C_{13}H_{16}N_6O$ 272.14 (100%).

3-Amino-5-dimethylamino-6,7-dimethyl-1-oxo-4,5-dihydro-1*H*-pyrrolo[3,4-c]pyridine-3a,7a-dicarbonitrile **3f** [methylethylketone TCEK + DMH]. T_{melt} = 167–169°C. IR (Nujol), ν_{max}/cm^{-1}: 1624 (s) (C=C), 1736 (s) (C=O), 2243 (s) (C≡N), 3457 (vs, br) (NH_2). ^1H NMR spectrum, δ, ppm: 1.49 (s, 3H, CH_3^{20}), 2.19 (s, 3H, CH_3^7), 2.61 (s, 6H, $N(CH_3)_2$), 4.02 (q, J = 7.0 Hz, 2H, CH_2^6), 5.27 (s, 3H, NH_2) (see Fig. 14, p. 319 at the Supplementary Section). ^{13}C NMR, δ_C, ppm: 14.18 (CH_3^{20}), 19.63 (CH_3^7), 39.96 ($N(CH_3)_2$), 41.14 (CH_2^6), 51.57 (C^5), 62.08 (C^4), 94.69 (C^3), 115.30 (CN^{15}), 116.19 (CN^{14}), 145.03 (C^2), 177.03 (C^{13}), 178.07 (C=O) (see Fig. 15, p. 320 at the Supplementary Section). C 57.30; H 6.01; N 30.63 $C_{13}H_{16}N_6O$. Calculated, %: C 57.34; H 5.92; N 30.86. Mass spectrum, m/z (I_{rel}, %): $C_{13}H_{16}N_6O$ 272.14 (100%).

RESULTS AND DISCUSSION

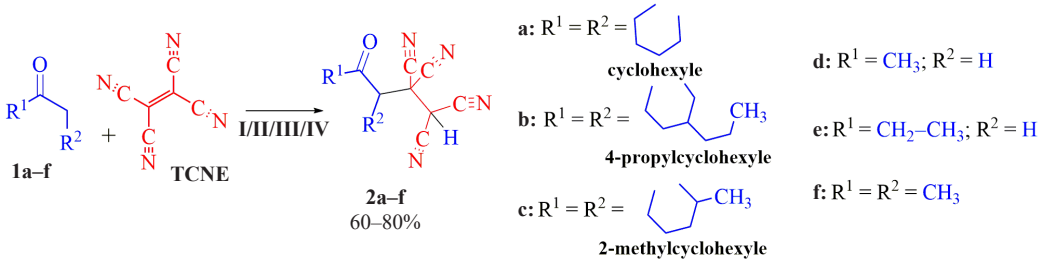
In order to utilize the off-specification intermediate DMH propellant, we employed TCEKs. As demonstrated in practice, these strong C–H acids readily and unambiguously undergo reactions with basic hydrazones. The possibility of preparing TCEKs from readily available reagents by relatively simple and low-labor synthetic methods is additionally advantageous for their practical application. Their synthesis is depicted in Scheme 1.

Cyclohexanone-derived TCEK **2a**, 4-propylcyclohexanone-derived TCEK **2b**, and 2-methylcyclohexanone-derived TCEK **2c** were obtained according to a general procedure: TCNE corresponding ketone **1a**–**c**, and a drop of concentrated hydrochloric acid were allowed for reacting at room temperature (20°C) (r.t.) in dioxane until completion. The products were isolated by crystallization from dioxane solutions, followed by precipitation with water. Tetracyanoethylation of acetone **1d** was carried out by dissolving TCNE in acetone under hydrochloric acid catalysis; target product **2d** was precipitated with water. TCEKs based on methyl ethyl ketone **1e**,**f**, bearing a polycyano substituent in the first (**2e**) and third (**2f**) positions, were obtained in ethanol either without additives (**2e**) or in presence of the sulfuric acid drop (**2f**), respectively. Target products **2e**–**f** were isolated by complete evaporation of ethanol, followed by trituration of the residue with water. Completion of the tetracyanoethylation reaction was confirmed by the HQ test, which develops an instantaneous blue coloration indicating presence of TCNE.

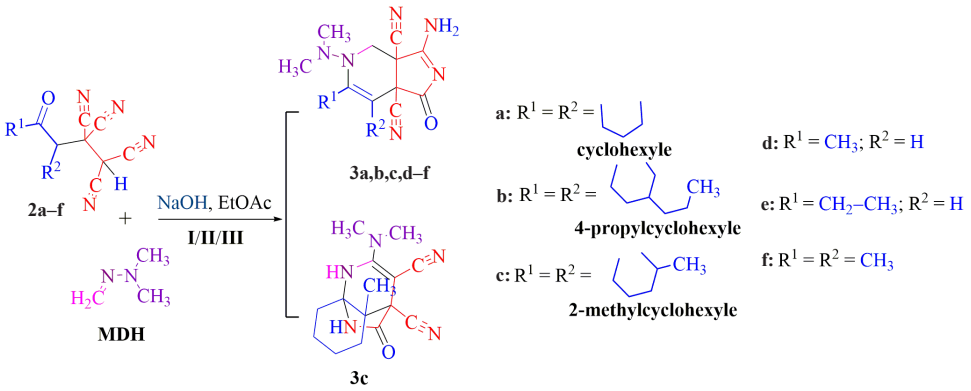
The obtained TCEKs **2a**–**f** were subjected to reaction with DMH under sodium hydroxide catalysis to afford pyrroloquinolines (pyridines) **3a**–**f**. The basic medium was required not only to optimize the cascade transformations described in [1, 2], but also to stabilize DMH. This reagent is prone to dimerization and polymerization [9]; the freshly distilled, dry compound rapidly turns yellow and gradually resinifies. When neutral DMH is mixed with TCEKs in ethyl acetate, polymerization of the reagent occurs prior to the desired transformation, as confirmed by TLC analysis. Thus, the basic catalyst plays a decisive role in the formation of the target products.

We conversed DMH into pyrroloquinolines (pyridines) **3a**–**f** under three different conditions: magnetic stirring, in an US reactor, and in a microwave oven. The synthesis under standard conditions has already been reported in our previous publications [1–3]. For US stirring, the reaction vessel was equipped with a copper ring, an HQ, and an ice bath. We hypothesized that the resin formation observed during the reactions, particularly with aliphatic TCEKs, has a radical character. Accordingly, common radical traps—copper [11] and HQ [12]—were employed. An ice bath was used to maintain a temperature of 10°C, which is the highest temperature at which resinification does not occur during US stirring.

For MWI, the ethyl acetate solutions were pre-cooled to 3–5°C. The low temperature also helped to suppress undesired polymerization and polycondensation of the starting reagents and intermediates during MWI at a minimum power of 250 W. The conversion of DMH into pyrroloquinolines (pyridines) **3a**–**f** is depicted in Scheme 2.



Scheme 1. Syntheses of TCEKs **2a**–**f** by the following procedures: (**I**) concentrated HCl, dioxane, r.t., for **2a**–**c**; (**II**) concentrated HCl, acetone, r.t., for **2d**; (**III**) concentrated H_2SO_4, ethanol, 45°C, for **2e**; (**IV**) ethanol, 40°C, for **2f**



Scheme 2. Syntheses of pyrroloquinolines(pyridines) **2a–f** by the following procedures: (**I**) magnetic stirring, r.t.; (**II**) US stirring, copper ring, HQ; (**III**) MWI, 250 Wt

The results of the experiment confirmed that the most efficient conversion of DMH is achieved by mixing it with cyclohexanone-derived TCEK **2a** under conditions where the reaction mixture was pre-cooled to 3–5°C, followed by MWI at 250 W for 2 min. This procedure afforded target pyrroloquinoline **3a** in 92% yield.

Nevertheless, for other cyclohexanone-derived TCEKs **2b–c**, modifications of the synthetic conditions did not lead to significant improvements. The standard conditions proved to be optimal for TCEK **2b**, yielding 75% of target product **3b**, whereas US and MWI irradiation reduced the yield by more than 10%. Mixing TCEK **2c** with basic DMH immediately caused darkening of the ethyl acetate solution and precipitation of heterocycle **3c** with a yield of 48%. Under US and MWI conditions, this yield increased only slightly, to 50–52%.

However, for aliphatic TCEKs **2d–f**, US and MWI irradiation ensured consistent formation of target pyrrolopyridines **3d–f** in amounts significantly higher than those obtained under standard synthesis conditions. In practice, magnetic stirring at room temperature with TCEKs **2d–f** does not always guarantee formation of the target products and often leads to polymerization of the starting reagents. The maximum yields of products **3d**, **3e**, and **3f** under standard conditions were 57%, 54%, and 52%, respectively. US stirring increased these yields to 73%, 62%, and 67%, while MWI further improved them to 82%, 70%, and 85%.

Table. Results of the DMH conversion optimization into pyrroloquinolines **3a–c** and pyrrolopyridines **3d–f** via US and MWI irradiation in comparison with standard magnetic stirring

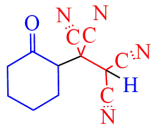
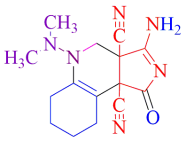
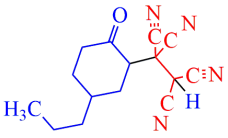
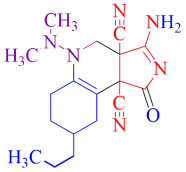
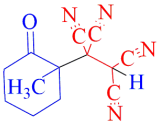
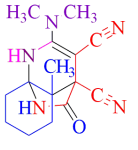
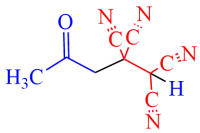
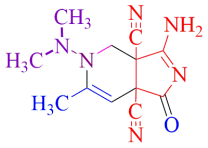
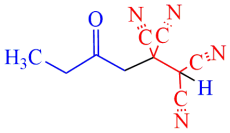
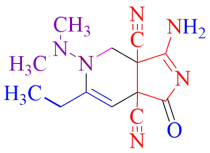
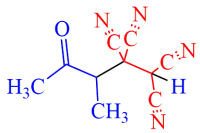
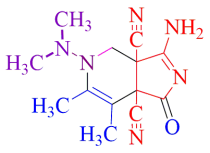
Initial compounds	Reaction condition	Reaction product	Yield, %	Time, min
 **2a**	Magnetic stirring	 **3a**	84	30
	US stirring		79	30
	MWI		92	2
 **2b**	Magnetic stirring	 **3b**	75	30
	US stirring		60	30
	MWI		62	2.5
 **2c**	Magnetic stirring	 **3c**	48	2
	US stirring		50	30
	MWI		52	5

Table. Continued

Initial compounds	Reaction condition	Reaction product	Yield, %	Time, min
 **2d**	Magnetic stirring	 **3d**	0–57	8
	US stirring		73	30
	MWI		82	4
 **2e**	Magnetic stirring	 **3e**	0–54	30
	US stirring		62	30
	MWI		70	5
 **2f**	Magnetic stirring	 **3f**	15–52	10
	US stirring		67	25
	MWI		85	3.5

CONCLUSIONS

The described work led to optimal conversion of the semi-product of low-grade rocket fuel—DMH—into pyrroloquinolines and pyrrolopyridines, which are potentially applicable in medicine. The best result was obtained in the reaction of DMH with cyclohexanone-derived TCEK under MWI, affording 92% of the pyrroloquinoline product in just two minutes. In addition, existing methods for synthesizing pyrrolopyridines from aliphatic TCEKs using US stirring and MWI were refined and stabilized. A direct correlation was observed between the use of US or MWI treatment and the increased yields of pyrrolopyridines. However, the proposed modifications did not yield significant improvements for 2-methylcyclohexanone derivative, and only slightly reduced yield of 4-propylcyclohexanone product.

Nevertheless, the near-quantitative conversion (92%) of the low-grade rocket fuel semi-product into pyrroloquinoline within two minutes highlights the potential of this approach for the efficient transformation of DMH into practically valuable products.

Acknowledgments

The work was carried out within Ulyanov Chuvash State University.

Authors' contributions

O.E. Nasakin—consultation on planning, methodology, and research implementation.

E.S. Ivanova—conducting research, reviewing publications on the topic of articles.

O.S. Korolevskaya—collecting and processing material, writing the text of the article.

S.Yu. Vasilieva—collecting and processing material, statistical processing.

Y. Kadyrov—development of the concept of scientific work, critical revision with the introduction of valuable intellectual content.

The authors declare no conflicts of interest.

SUPPLEMENTARY

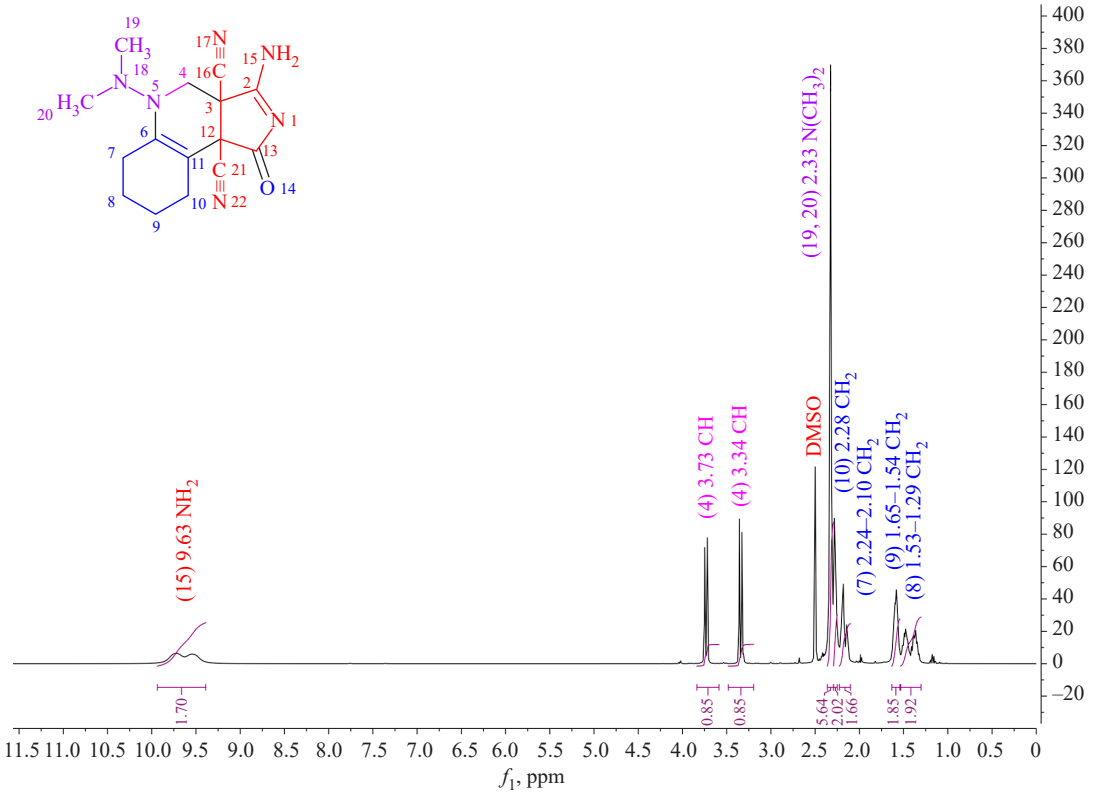


Fig. 1. ^{1}H NMR spectrum of **3a** (400 MHz, DMSO-d_6, 298 K)

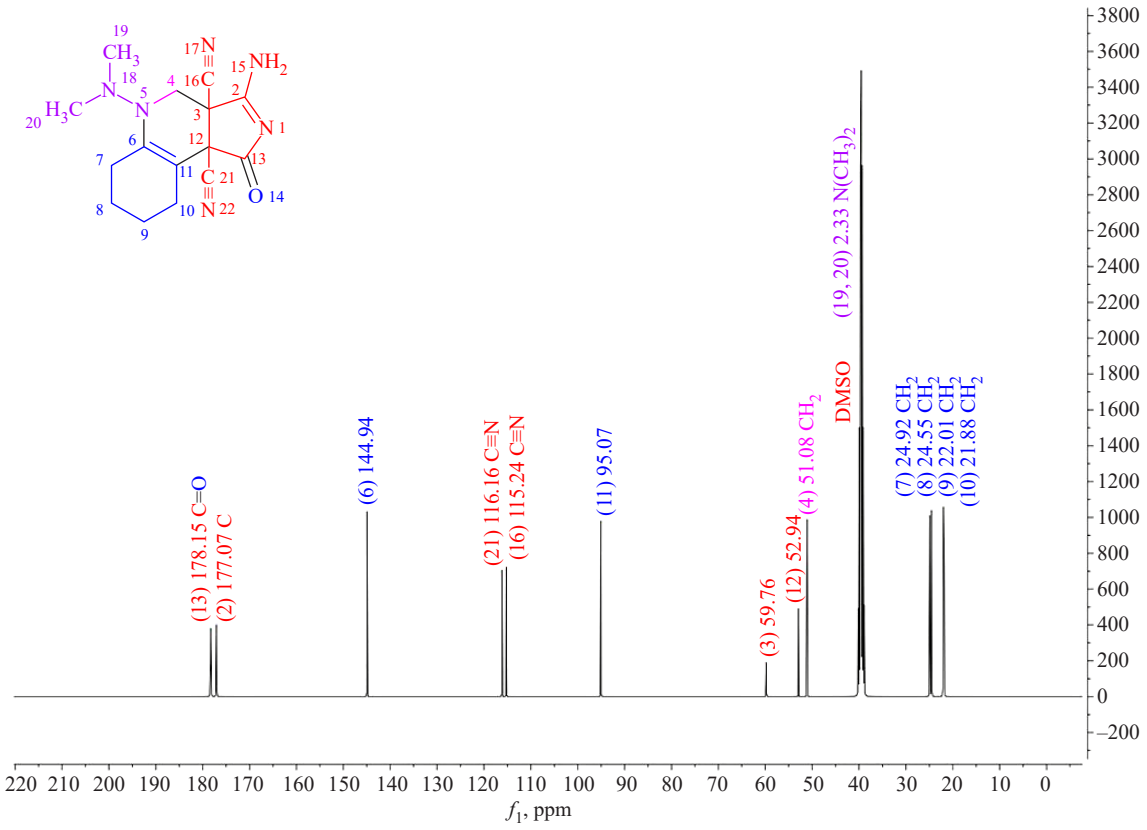


Fig. 2. ^{13}C NMR spectrum of **3a** (100 MHz, DMSO-d_6, 299 K)

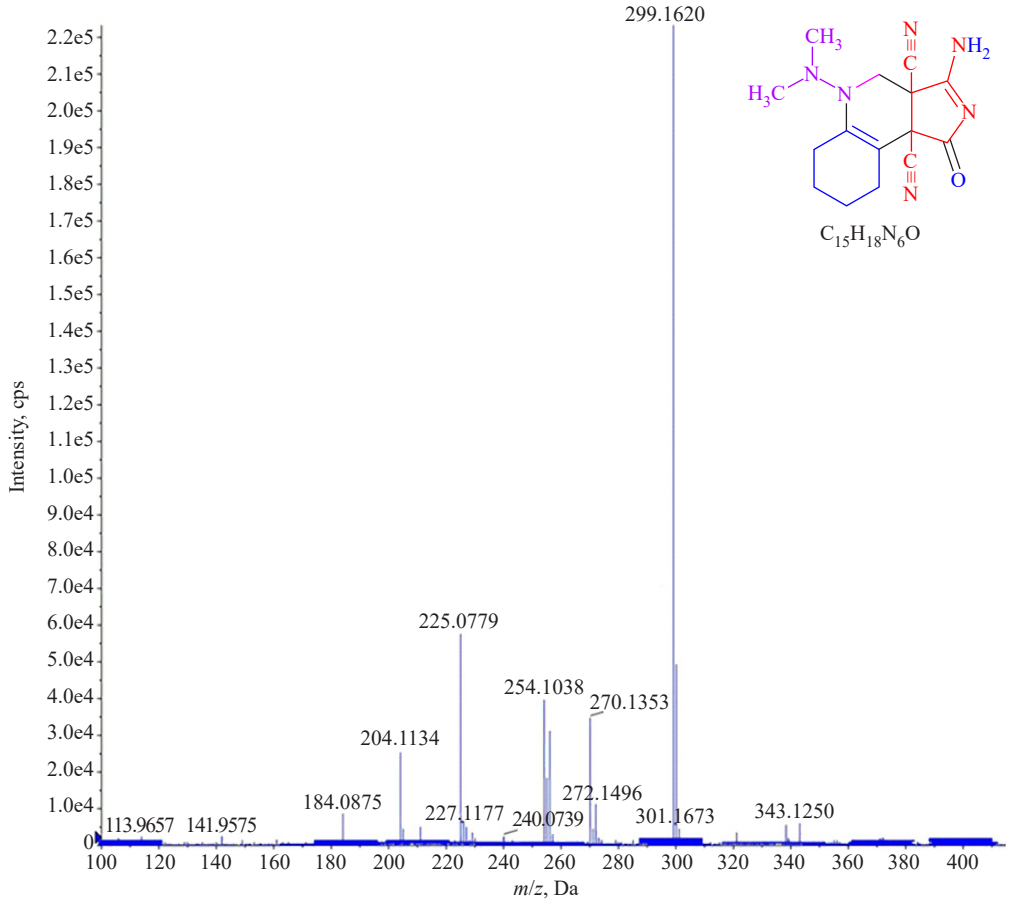


Fig. 3. HRMS spectrum of **3a**

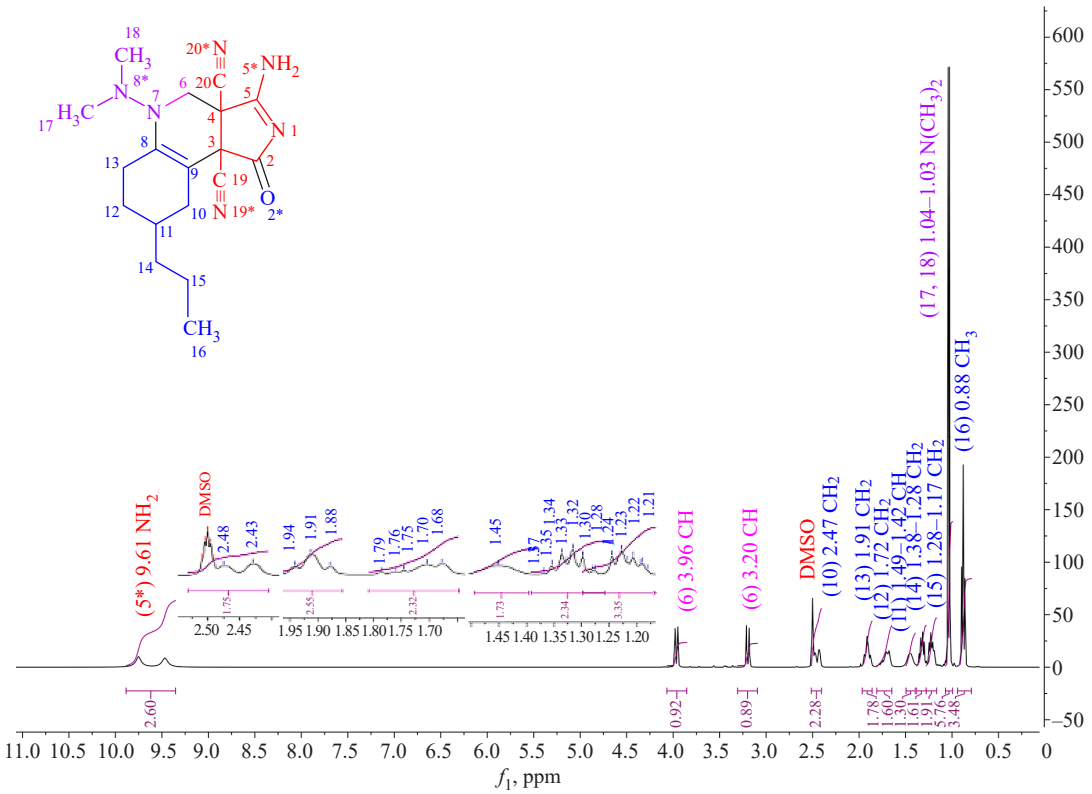


Fig. 4. ^{1}H NMR spectrum of **3b** (400 MHz, DMSO-d_6, 299 K)

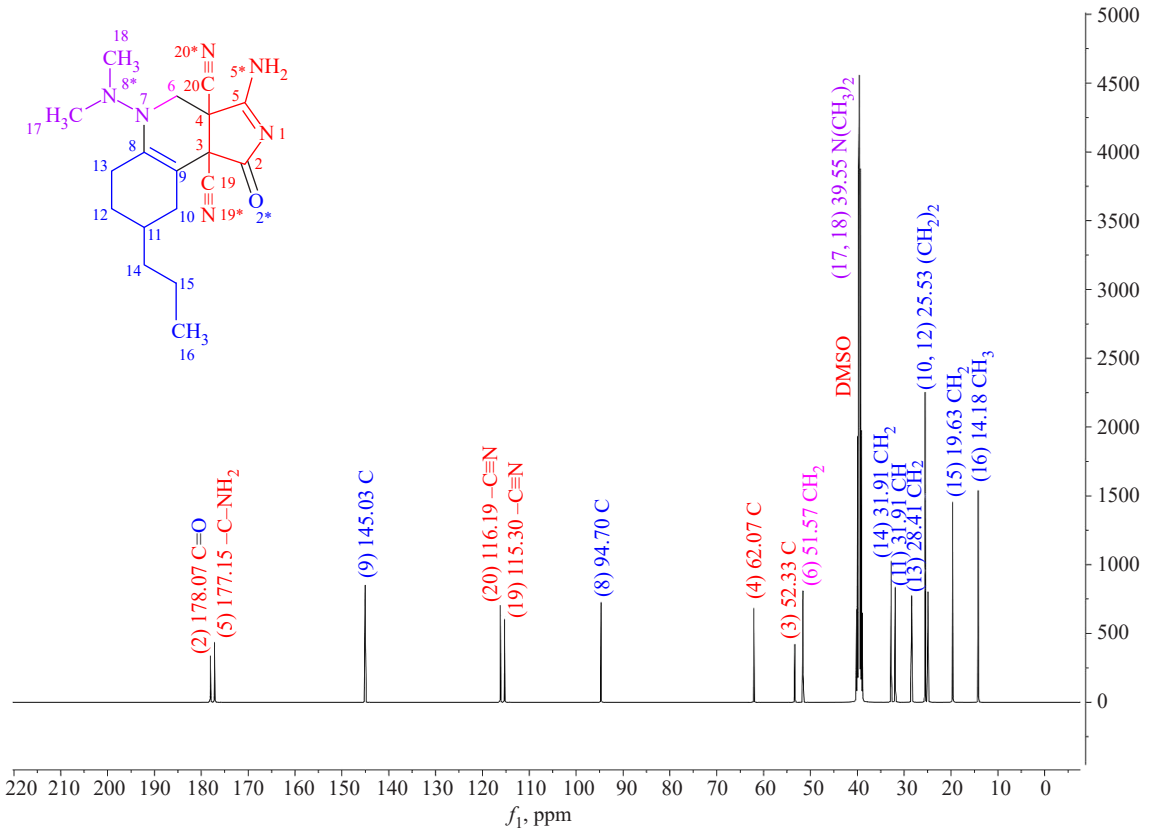


Fig. 5. ^{13}C NMR spectrum of **3b** (100 MHz, DMSO-d_6, 299 K)

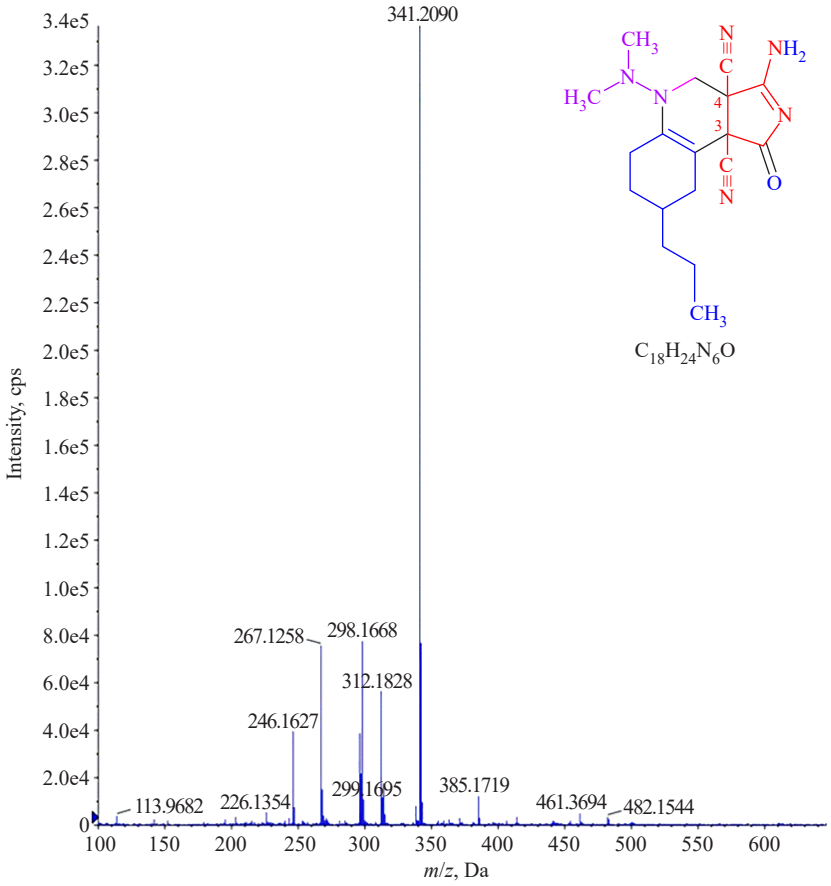


Fig. 6. HRMS spectrum of **3b**

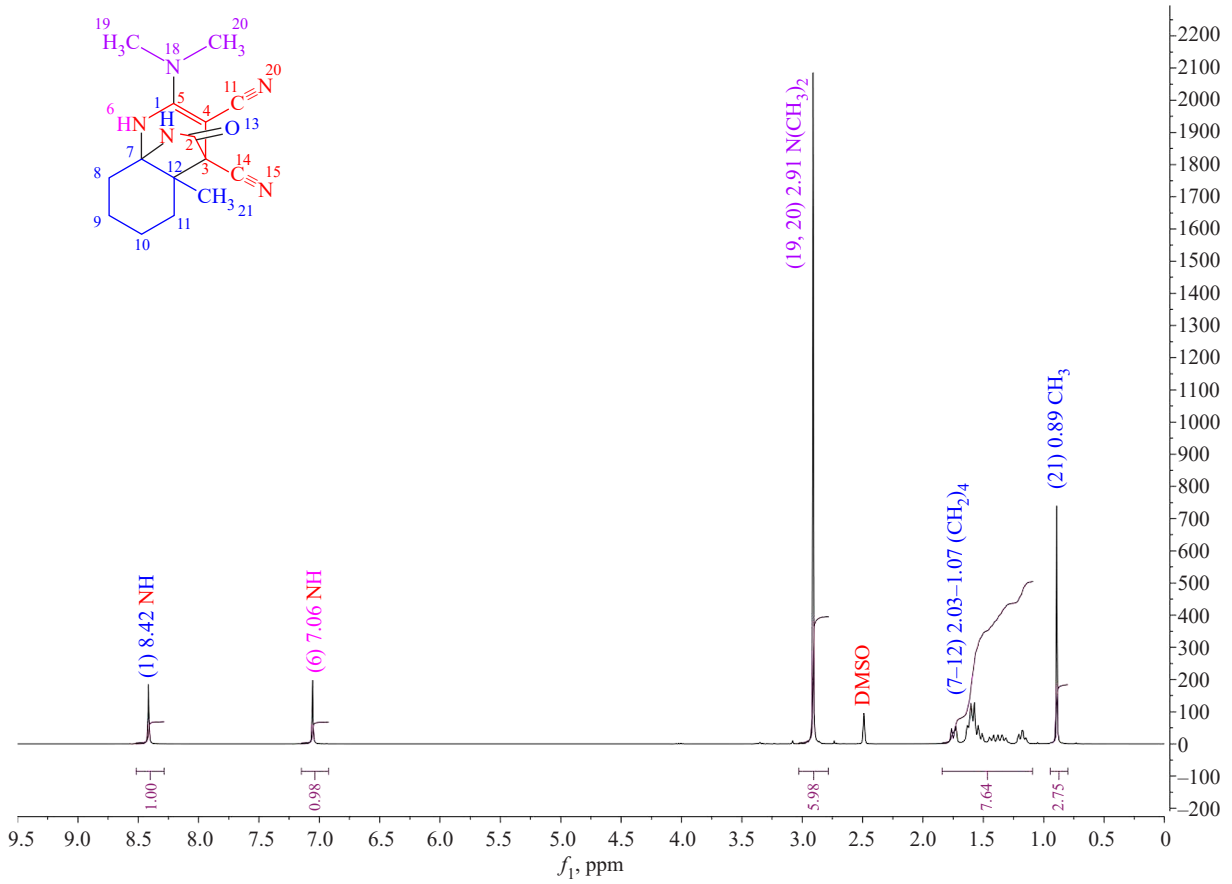


Fig. 7. ^{1}H NMR spectrum of **3c** (400 MHz, DMSO-d_6, 296 K)

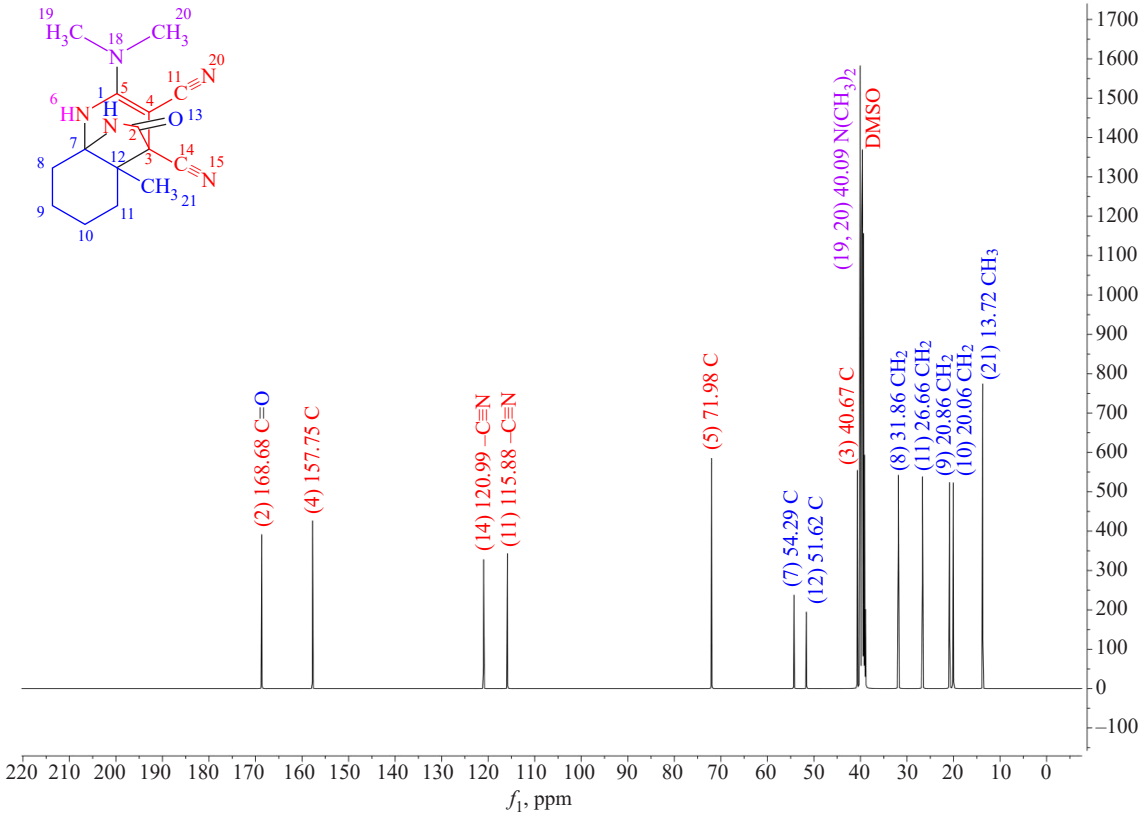


Fig. 8. ^{13}C NMR spectrum of **3c** (100 MHz, DMSO-d_6, 297 K)

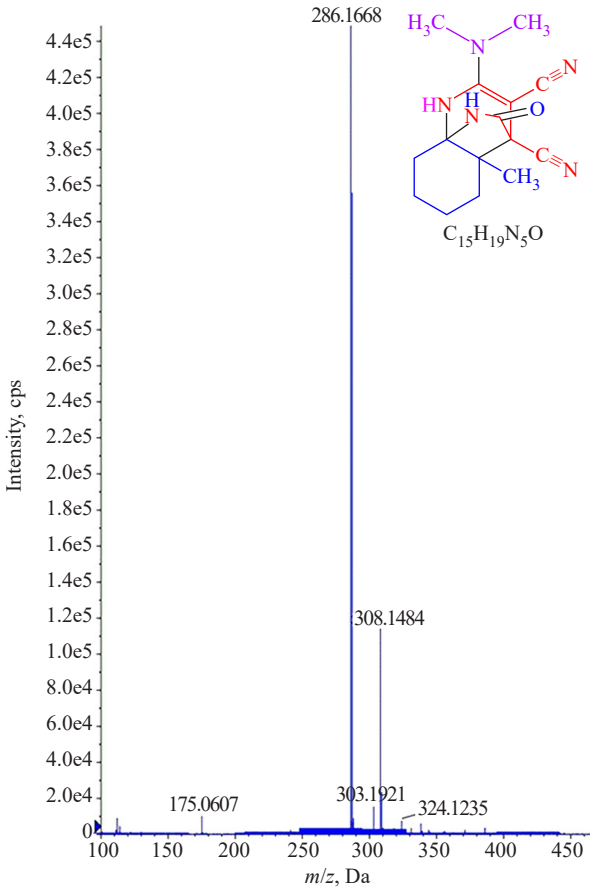


Fig. 9. HRMS spectrum of **3c**

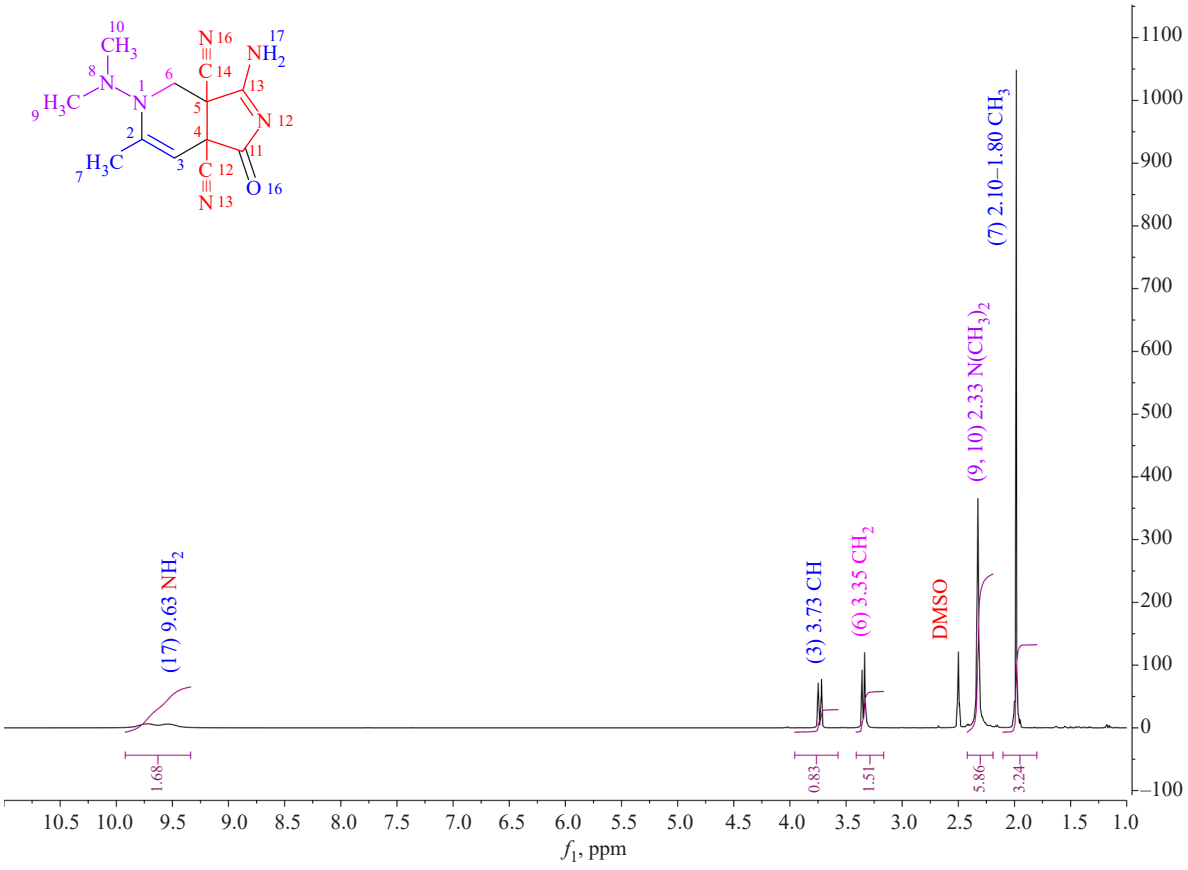


Fig. 10. ^{1}H NMR spectrum of **3d** (500.13 MHz, DMSO-d_6, 299 K)

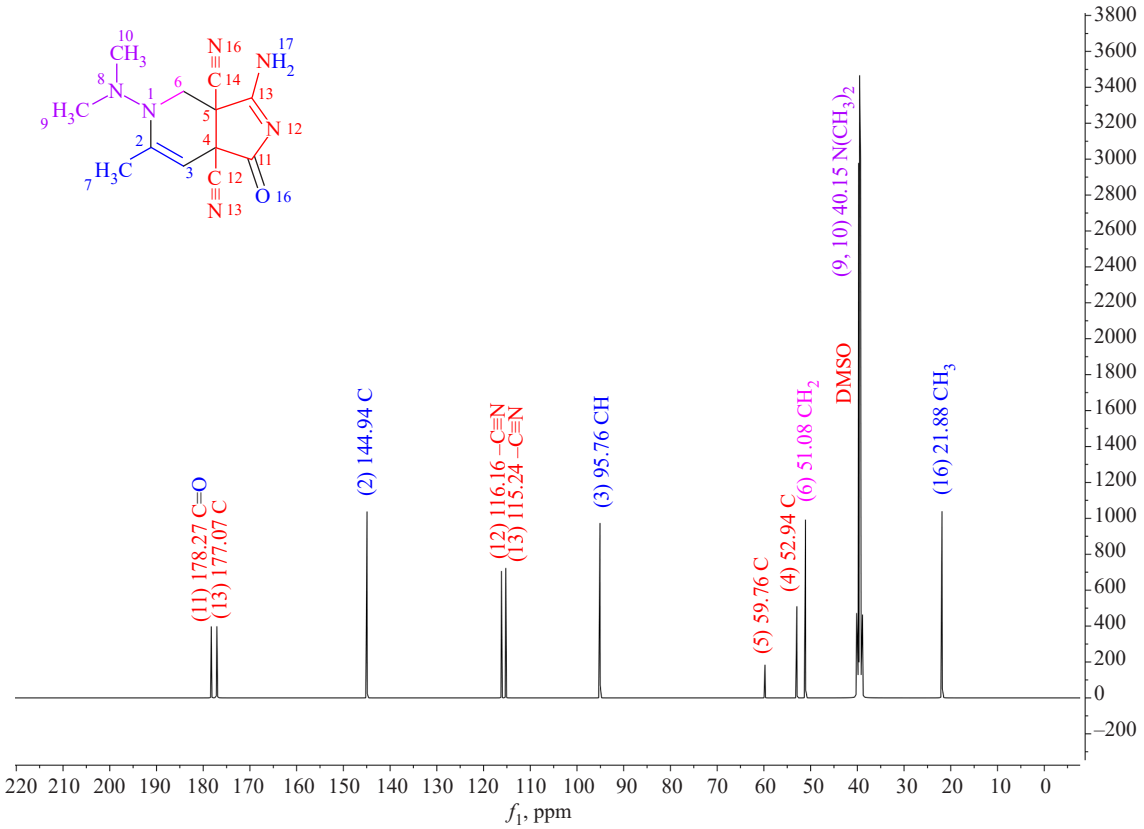


Fig. 11. ^{13}C NMR spectrum of **3d** (500.13 MHz, DMSO-d_6, 299 K)

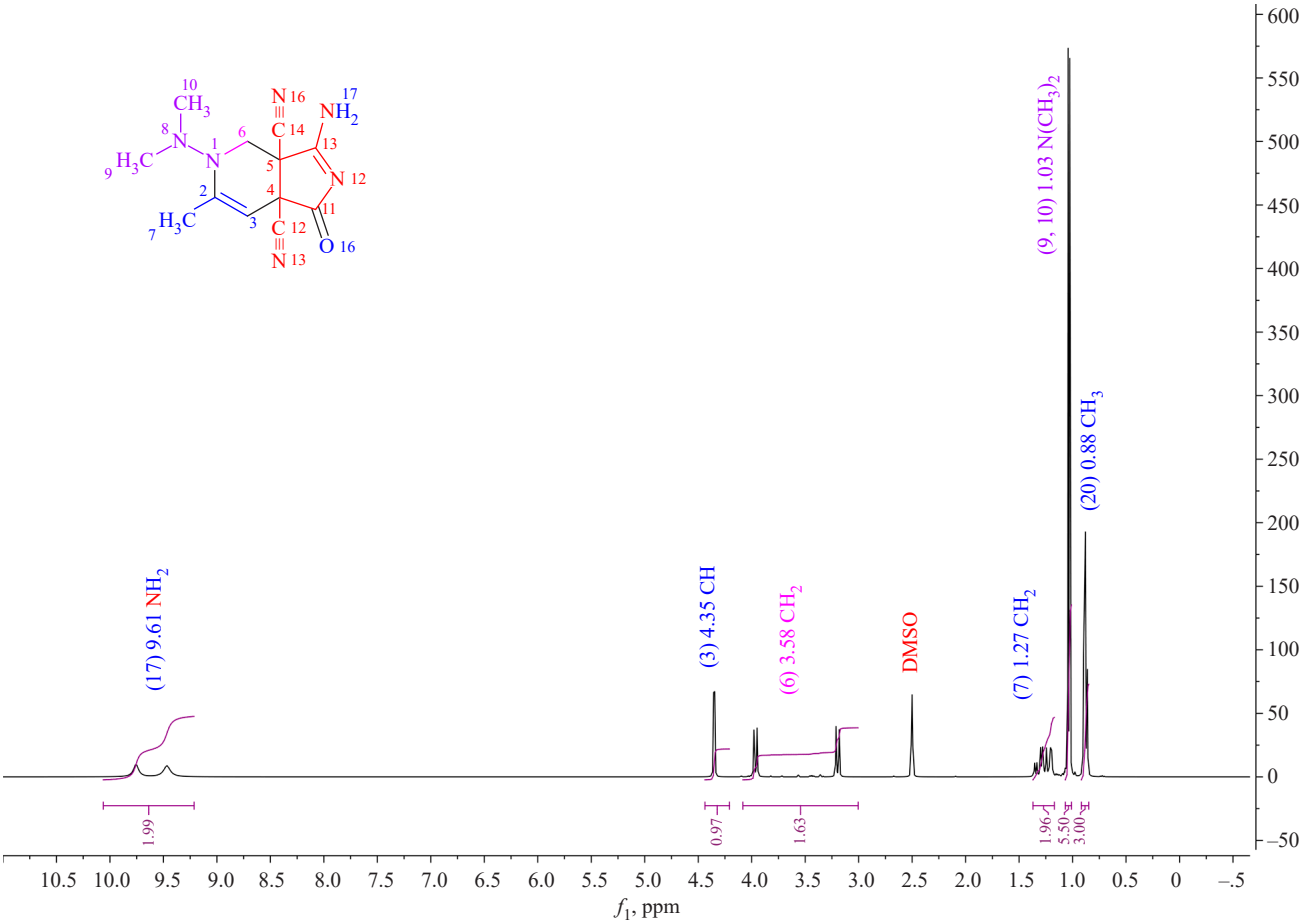


Fig. 12. ^{1}H NMR spectrum of **3e** (500.13 MHz, DMSO-d_6, 299 K)

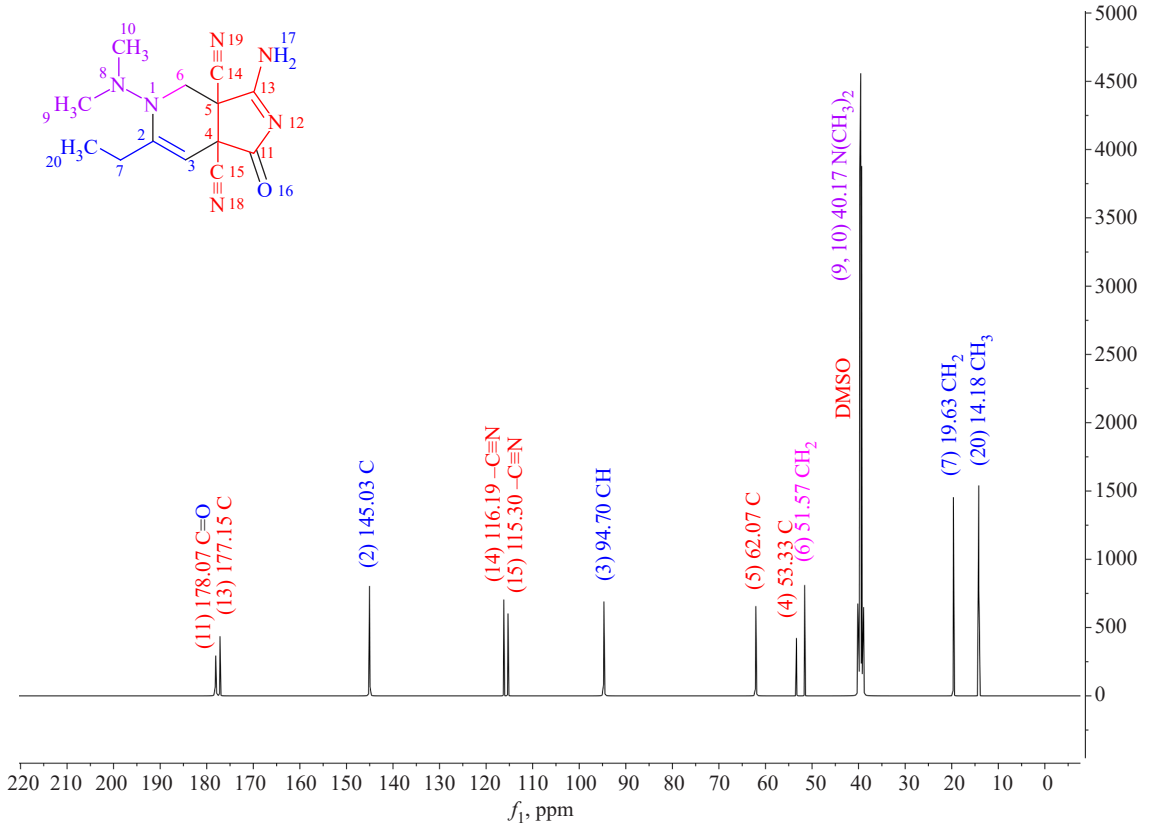


Fig. 13. ^{13}C NMR spectrum of **3e** (125.76 MHz, DMSO-d_6, 299 K)

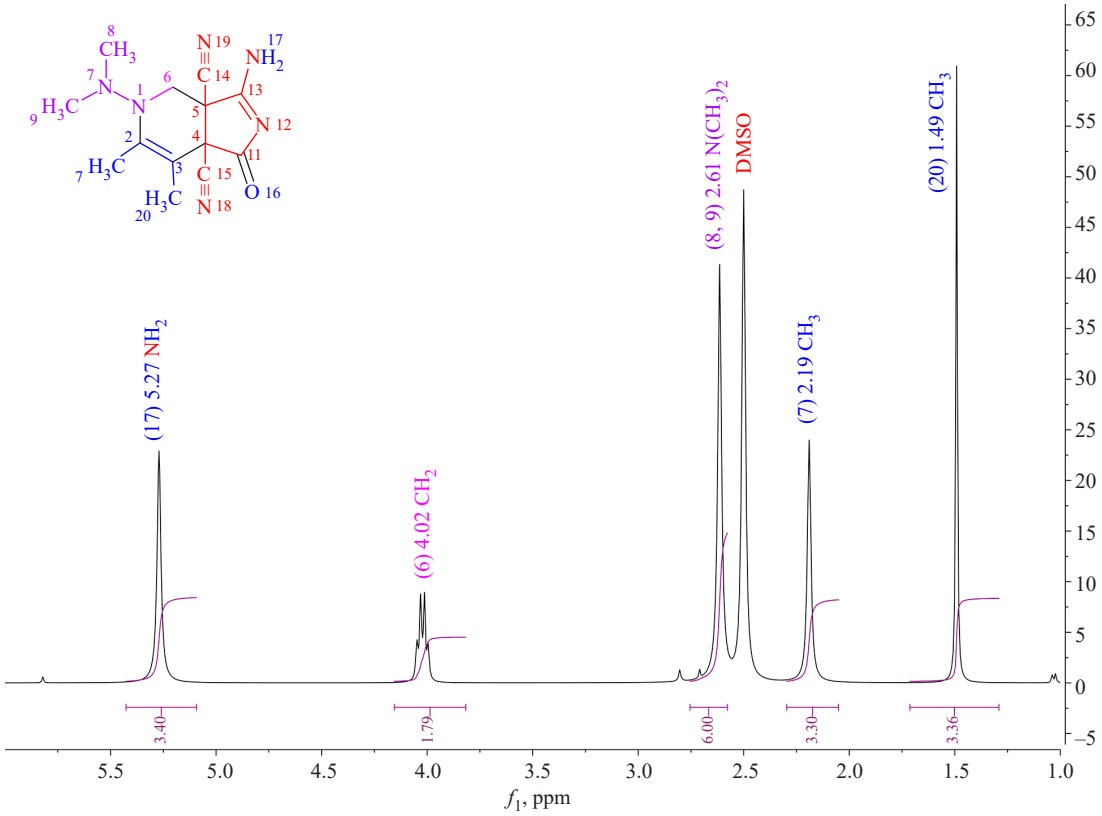


Fig. 14. ^{1}H NMR spectrum of **3f** (500.13 MHz, DMSO-d_6, 299 K)

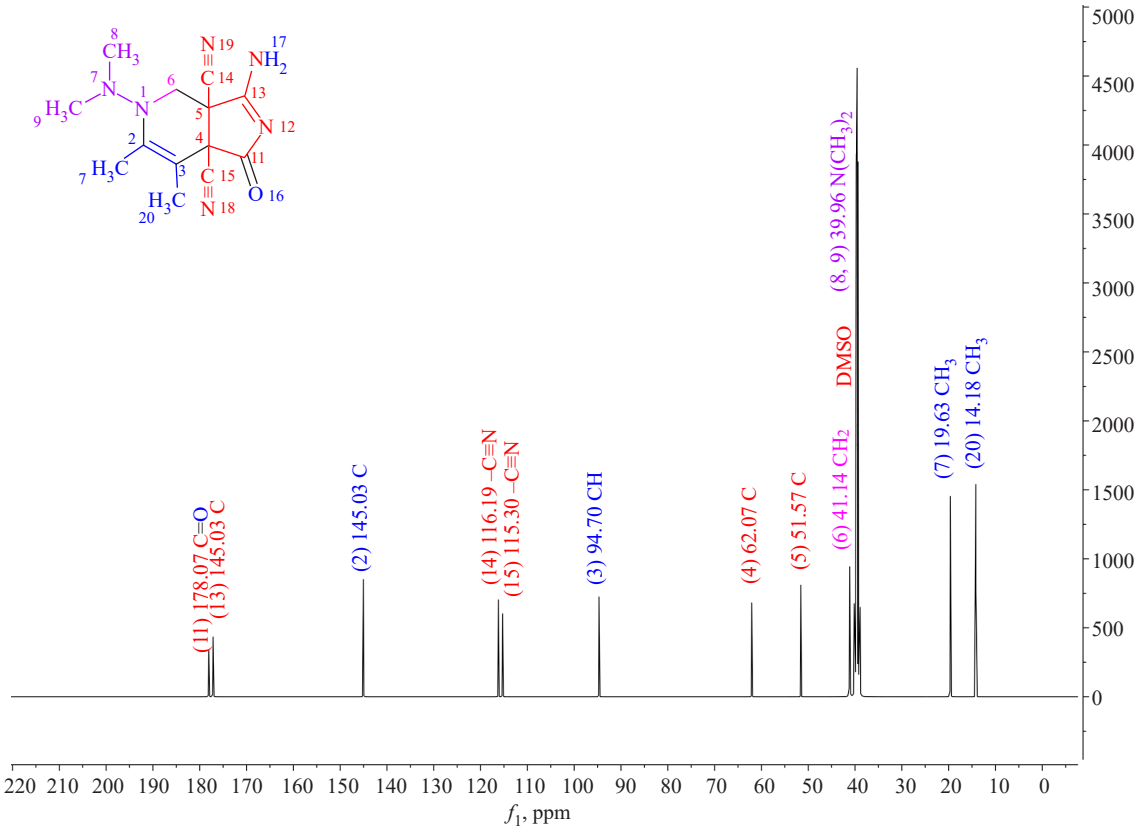


Fig. 15. ^{13}C NMR spectrum of **3f** (125.76 MHz, DMSO-d_6, 299 K)

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