Stereoselective electrocatalytic transformations of malononitrile and aromatic aldehydes into (1*R*,5*S*,6*R*)*-4,4-dialkoxy-2-amino-6-aryl-1,5-dicyano-3-azabicyclo[3.1.0]hex-2-enes

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Electrolysis of aromatic aldehydes and malononitrile in alcohols in an undivided cell in the presence of a NaBr—NaOAc mediatory system stereoselectively gave bicyclic compounds containing the cyclopropane and pyrroline fragments in 60 to 70% yields. In the bicyclic products, the benzene and pyrroline rings are on different sides of the cyclopropane ring.

Key words: electrolysis, electrocatalytic transformation, stereoselectivity, malononitrile, aromatic aldehydes, mediators, pyrrolines, cyclopropanes, heterocyclization.

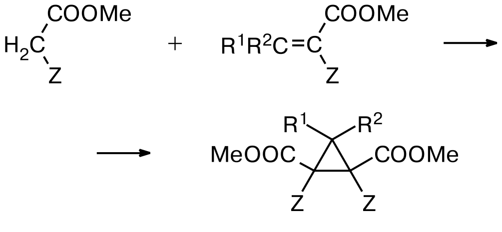
Functionalized cyclopropanes exhibit a broad spectrum of physiological activity.[1] In addition, they represent an important class of compounds used for the syntheses of biologically active natural substances.[1,2] Cyclopropanecarboxylic acid derivatives have been successfully used in medicine and agriculture. The most known area of their application is natural and synthetic pyrethroids as insecticidal preparations.[1,3] For the above reasons, development of simple and efficient methods for the synthesis of functionalized cyclopropanes still remains of topical interest.

In the last few decades, mediators and mediatory systems have been effectively used for electroreduction and electrooxidation of organic compounds. Among a great number of mediators, the halide anion—halogen redox system holds out much promise for use in organic synthesis.[4]

The present work is intended to continue a series of our investigations of electrochemical transformations of CH acids such as malononitrile, ethyl cyanoacetate, and ethyl malonate into functionalized cyclopropanes with alkali metal halides as mediators. Earlier, while studying electrocatalytic oxidation of organic compounds in the presence of mediators, we carried out electrochemical cyclotrimerization of ethyl malonate[5] and ethyl cyanoacetate,[6] as well as transformations of aldehydes and ethyl cyanoacetate[7] into functionalized cyclopropanes and of ketones and malononitrile into 3,3-disubstituted tetracyanocyclopropanes.[8]

Recently,[9—11] we proposed a new approach to the synthesis of functionalized cyclopropanes by co-electrolysis of CH acids with activated olefins in the presence of halides as mediators (Scheme 1).

Scheme 1



Z = COOMe, CN

Reagents and conditions: electrolysis, NaHal (Hal = Br or I), MeOH.

This method was used to obtain, starting from cycloalkylidenemalononitriles and malononitrile, tetracyanospirobicyclic compounds containing a cyclopropane fragment and spirotricyclic compounds containing cyclopropane and pyrroline fragments[12,13] (Scheme 2). Such compounds are promising for use in the synthesis of natural biologically active compounds[14,15] and modern drugs[16] and are themselves potential antitumor agents.[17]

In the preceding paper,[18] we reported that co-electrolysis of malononitrile with arylidenemalononitriles in

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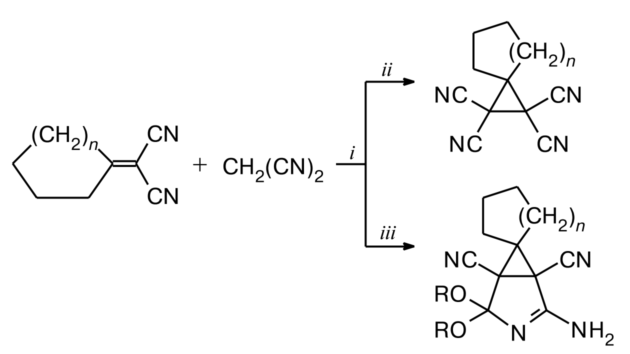
Scheme 2

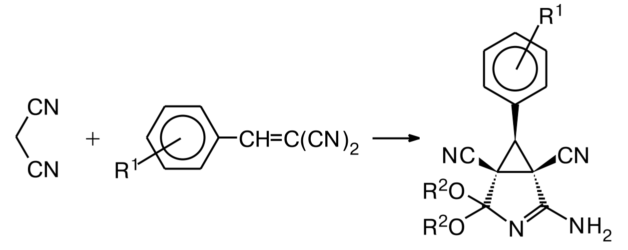


n = 1, 2, 3, 8

Reagents and conditions: *i*. Electrolysis; *ii*. NaBr, EtOH; *iii*. NaBr—NaOR, ROH.

alcohols in the presence of sodium halides stereoselectively yields (1*R*,5*S*,6*R*)*-4,4-dialkoxy-2-amino-6-aryl-1,5-dicyano-3-azabicyclo[3.1.0]hex-2-enes (Scheme 3).

Scheme 3

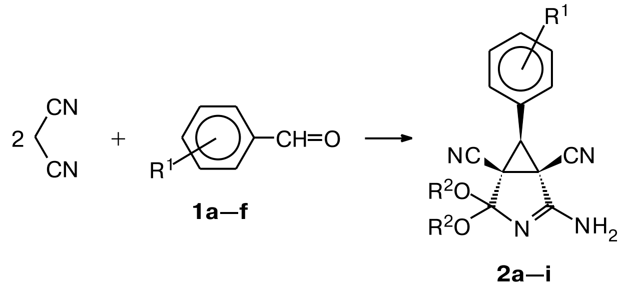


Reagents and conditions: electrolysis, NaHal (Hal = I or Br); R^2OH.

The latter result made it practically feasible to carry out a more complex stereoselective electrochemical process "domino", namely, direct transformation of malononitrile and an aromatic aldehyde into the corresponding bicyclic pyrroline. To do this, it was necessary to select electrolysis conditions for the initial Knoevenagel condensation of malononitrile with the aromatic aldehyde into arylidenemalononitrile and its subsequent reaction with malononitrile to give bicyclic pyrroline. Previously,[19,20] we found that the Knoevenagel reaction is catalyzed by alkoxide ions generated at the cathode by the passage of an electric current through alcoholic solutions.

In the present paper, we present our data on one-pot stereoselective electrocatalytic domino transformation of malononitrile and aromatic aldehydes **1a—f** into (1*R*,5*S*,6*R*)*-4,4-dialkoxy-2-amino-6-aryl-1,5-dicyano-3-azabicyclo[3.1.0]hex-2-enes **2a—i**. The process was carried out in an undivided cell in methanol or ethanol; NaBr or the new system NaBr—NaOAc were used as mediators (Scheme 4, Table 1).

Scheme 4



Reagents and conditions: electrolysis, NaBr—NaOAc, R^2OH.

1	**a**	**b**	**c**	**d**	**e**	**f**
R^1	H	4-Me	4-OMe	2-Cl	3-Br	4-I

2	**a**	**b**	**c**	**d**	**e**	**f**	**g**	**h**	**i**
R^1	H	H	4-Me	4-OMe	2-Cl	2-Cl	3-Br	4-I	4-I
R^2	Me	Et	Me	Me	Me	Et	Me	Me	Et

It follows from the data obtained (see Table 1) that the suggested electrocatalytic process can really be accomplished in one step, directly starting from malononitrile and an aromatic aldehyde in the presence of NaBr as a mediator. The optimal temperature is 10 °C; the yields of the corresponding bicyclic pyrrolines at this temperature were 45 to 50%. At 20 or 0 °C, their yields were reduced to 30%.

Table 1. Stereoselective electrocatalytic domino transformation of aldehydes **1a—f** and malononitrile into bicyclic pyrrolines **2a—i**

Aldehyde, R^1	R^2	Conditions[a]		Pyrroline	Yield of **2**[b] (%)
		Mediator	*T*/°C		
1a, H	Me	NaBr	20	**2a**	33
	Me	NaBr	10	**2a**	51
	Me	NaBr	0	**2a**	38
	Me	NaBr[c]	10	**2a**	35
	Me	NaBr—NaOAc	10	**2a**	67
	Et	NaBr—NaOAc	10	**2b**	64
1b, 4-Me	Me	NaBr—NaOAc	10	**2c**	61
	Me	NaBr	10	**2c**	45
1c, 4-OMe	Me	NaBr—NaOAc	10	**2d**	58
1d, 2-Cl	Me	NaBr—NaOAc	10	**2e**	63
	Me	NaBr	10	**2e**	46
	Et	NaBr—NaOAc	10	**2f**	59
1e, 3-Br	Me	NaBr—NaOAc	10	**2g**	57
1f, 4-I	Me	NaBr—NaOAc	10	**2h**	62
	Et	NaBr—NaOAc	10	**2i**	71

[a] Aldehyde (10 mmol), malononitrile (20 mmol), NaBr (5 mmol), NaOAc (5 mmol), ethanol (20 mL), Fe cathode, C anode, current density 100 mA cm^{-2}, electricity passed 2.5 F mol^{-1}.

[b] With respect to the isolated pyrroline.

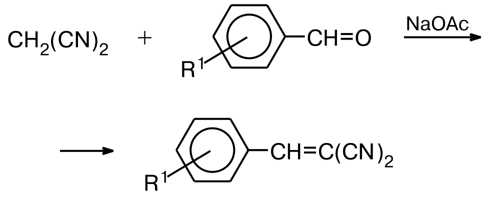
[c] Malononitrile (25 mmol).

With an excess of malononitrile (malononitrile : aldehyde = 2.5 : 1, while the theoretical ratio for the preparation of bicyclic pyrroline **2** is 2 : 1), intermediate 3-aryl-1,1,2,2-tetracyanocyclopropane was incompletely converted into the corresponding bicyclic pyrroline. In this case, the yield of compound **2a** decreased to 35% (versus a 51% yield attained under the optimal conditions at 10 °C); the reaction mixture contained tetracyano-3-phenylcyclopropane (14%) (^{1}H NMR data). As noted earlier, an excess of malononitrile inhibits the transformation of tetracyanocyclopropanes into the corresponding pyrrolines[12] by reducing the current concentration of alkoxide ions.

The yield of bicyclic pyrrolines **2** in the electrolysis was increased from 45—50% to 60—70% with the use of the NaBr—NaOAc mediatory system. Here, NaOAc serves as a catalyst for the Knoevenagel reaction. The reaction of malononitrile with an aromatic aldehyde in the presence of NaOAc and in the absence of electric current quantitatively gave the corresponding arylidenemalononitrile, the reaction time being shorter than half the time of the electrochemical process (40 min). Under analogous conditions without NaOAc, this reaction did not occur. Earlier, NaOAc was used as a catalyst in the Perkin condensation.[21]

Based on the data obtained, data on electrochemical transformations of malononitrile and aldehydes into 3-substituted tetracyanocyclopropanes,[19] and data on stereoselective electrocatalytic transformations of malononitrile and arylidenemalononitriles into (1*R*,5*S*,6*R*)*-4,4-dialkoxy-2-amino-6-aryl-1,5-dicyano-3-azabicyclo[3.1.0]hex-2-enes,[18] we proposed a mechanism of the stereoselective electrocatalytic transformation of malononitrile and aromatic aldehydes into bicyclic pyrrolines **2**.

The NaOAc-catalyzed reaction of malononitrile with an aromatic aldehyde yields arylidenemalononitrile (reaction (1)).



(1)

The electrode reactions, which are typical of the Br^-—Br_2 mediatory system in alcohols, involve anodic formation of bromine (reaction (2)) and cathodic evolution of hydrogen with generation of alkoxide ions (reaction (3)).

$$\text{Anode:}\quad 2\,Br^- - 2\,e \longrightarrow Br_2 \qquad (2)$$

$$\text{Cathode:}\quad 2\,R^2OH + 2\,e \longrightarrow 2\,R^2O^- + H_2 \qquad (3)$$

Then, the alkoxide ion reacts with malononitrile in solution to give the malononitrile anion (reaction (4)).

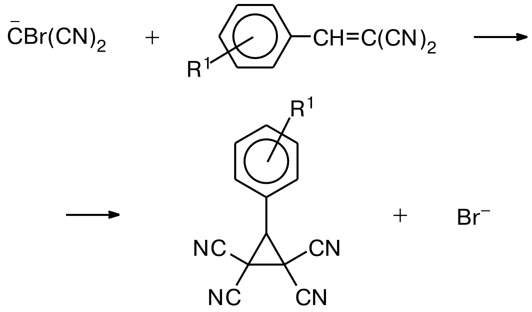
$$CH_2(CN)_2 + R^2O^- \longrightarrow \bar{C}H(CN)_2 + R^2OH \qquad (4)$$

The reaction of the latter with bromine gives bromomalononitrile (reaction (5)).

$$\bar{C}H(CN)_2 + Br_2 \longrightarrow CHBr(CN)_2 + Br^- \qquad (5)$$

Being generated in the presence of the alkoxide ion (reaction (6)), the bromomalononitrile anion adds to the double bond of arylidenemalononitrile to give tetracyanocyclopropane (reaction (7)).

$$CHBr(CN)_2 + R^2O^- \longrightarrow \bar{C}Br(CN)_2 + R^2OH \qquad (6)$$



(7)

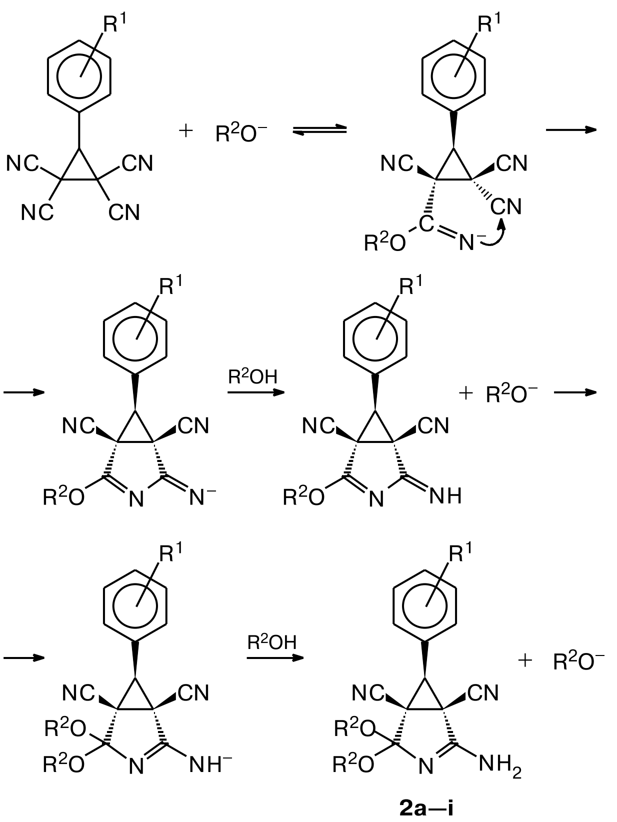
Its subsequent reaction with alkoxide ions stereoselectively yields bicyclic pyrroline **2** as the final product of sequential cascade transformations (Scheme 5).

The stereoselectivity of the process is ensured by the formation of the pyrroline ring from the CN groups that are *trans* to the aryl substituent. The formation of only one isomer out of two possible was confirmed by ^{1}H and ^{13}C NMR data. Structure **2a** was determined earlier[18] by X-ray diffraction analysis. Because the formation of the pyrroline ring should be sterically least hindered, one can believe that in all the bicyclic pyrrolines **2** obtained, the pyrroline ring and the aryl substituent are *trans* relative to the cyclopropane ring.

Thus, the domino cascade process conducted in an undivided cell in the presence of mediator allows, *via* co-electrolysis of malononitrile with aromatic aldehydes, stereoselective one-step synthesis of bicyclic pyrrolines containing the cyclopropane and pyrroline fragments. In terms of classic organic chemistry, this process can be accomplished at least in four steps: (1) halogenation of malononitrile, (2) preparation of arylidenemalononitrile from malononitrile and an aromatic aldehyde by the Knoevenagel reaction, (3) addition of halomalononitrile to the double bond of the cyclic alkylidenemalononitrile followed by cyclization,[22] and (4) reaction of the resulting tetracyanocyclopropane with alkoxide ions in alcohols.[19]

The developed electrochemical process is a convenient and efficient method for direct stereoselective trans-

Scheme 5



formations of malononitrile and aromatic aldehydes into bicyclic compounds containing the cyclopropane and pyrroline fragments since common and accessible reagents, inexpensive equipment, and an undivided cell are used. Electrolysis and isolation of products are simple and can be carried out both under laboratory conditions and in larger reactors.

Experimental

^{1}H NMR spectra were recorded on Bruker WM-250 and Bruker AM-300 instruments (250 and 300 MHz, respectively) in DMSO-d_6 or $CDCl_3$. Chemical shifts are given on the δ scale with reference to Me_4Si.

Malononitrile was purchased from Aldrich; aromatic aldehydes were purchased from Aldrich and Merck. Tetracyano-3-phenylcyclopropane was prepared from malononitrile and benzaldehyde as described earlier.[19]

Co-electrolysis of malononitrile with aromatic aldehydes (general procedure). A solution of malononitrile (20 mmol), aromatic aldehyde **1** (10 mmol), NaBr (5 mmol), and NaOAc (5 mmol) in ethanol (20 mL) was electrolyzed in an undivided cell with the carbon anode and the iron cathode (electrode area 5 cm^2) at a constant current density of 100 mA cm^{-2} by passing the quantity of electricity specified in Table 1. After the electrolysis was completed, the precipitate of bicyclic pyrroline **2** was filtered off and washed with cold ethanol. An additional crop of bicyclic pyrroline **2** was isolated as follows. The mother liquor was concentrated and the product was extracted with ethyl acetate; the extract was washed with water and dried over Na_2SO_4. The solvent was removed and the residue was recrystallized from acetone—hexane.

2-Amino-1,5-dicyano-4,4-dimethoxy-6-phenyl-3-azabicyclo[3.1.0]hex-2-ene (2a),[18] m.p. >350 °C. ^{1}H NMR (DMSO-d_6), δ: 3.35, 3.38 (both s, 3 H each, OMe); 3.45 (s, 1 H, CH); 7.40—7.65 (m, 7 H, C_6H_5 and NH_2). ^{13}C NMR (DMSO-d_6), δ: 35.50, 37.61 (2 C); 41.62 (CH); 49.63, 51.26 (2 OMe); 112.30, 113.61 (2 CN); 118.75 ($C(OMe)_2$); 128.71, 128.93, 129.17, 129.73 (C_6H_5); 159.36 (C=N). IR (KBr), ν/cm^{-1}: 3440, 3064, 2248, 1680, 1428, 1172, 1136, 1076, 1104, 700.

2-Amino-1,5-dicyano-4,4-diethoxy-6-phenyl-3-azabicyclo[3.1.0]hex-2-ene (2b),[18] m.p. 246—248 °C. ^{1}H NMR ($CDCl_3$), δ: 1.15, 1.17 (both t, 3 H each, Me, J = 7 Hz); 3.47 (s, 1 H, CH); 3.71, 3.83 (both q, 2 H each, CH_2O, J = 7 Hz); 7.35—7.65 (m, 7 H, C_6H_5 and NH_2). ^{13}C NMR (DMSO-d_6), δ: 15.01, 15.23 (2 Me); 35.41, 38.22 (2 C); 41.43 (CH); 57.71, 59.32 (2 CH_2O); 112.33, 113.51 (2 CN); 118.12 ($C(OEt)_2$); 128.80, 128.83, 129.06, 129.73 (C_6H_5); 159.01 (C=N). IR (KBr), ν/cm^{-1}: 3396, 2980, 2248, 1664, 1444, 1140, 1076, 1048, 700.

2-Amino-1,5-dicyano-4,4-dimethoxy-6-(4-methylphenyl)-3-azabicyclo[3.1.0]hex-2-ene (2c),[18] m.p. >350 °C. ^{1}H NMR (DMSO-d_6), δ: 2.32 (s, 3 H, Me); 3.33 (s, 1 H, CH); 3.35, 3.37 (both s, 3 H each, OMe); 7.30, 7.45 (both d, 2 H each, H arom., J = 8 Hz); 7.51 (s, 2 H, NH_2). ^{13}C NMR (DMSO-d_6), δ: 20.71 (Me); 35.44, 37.60 (2 C); 40.05 (CH); 49.54, 51.17 (2 OMe); 112.27, 113.57 (2 CN); 118.62 ($C(OMe)_2$); 126.61, 128.69, 129.36, 138.67 (C arom.); 159.25 (C=N). IR (KBr), ν/cm^{-1}: 3404, 3045, 2252, 1688, 1436, 1136, 1116, 1076, 808.

2-Amino-1,5-dicyano-4,4-dimethoxy-6-(4-methoxyphenyl)-3-azabicyclo[3.1.0]hex-2-ene (2d),[18] m.p. >350 °C. ^{1}H NMR (DMSO-d_6), δ: 3.31 (s, 1 H, CH); 3.35, 3.38, 3.81 (all s, 3 H each, OMe); 7.05, 7.47 (both d, 2 H each, H arom., J = 8 Hz); 7.49 (s, 2 H, NH_2). ^{13}C NMR (DMSO-d_6), δ: 35.61, 37.71 (2 C); 41.27 (CH); 49.57, 51.23, 55.29 (3 OMe); 112.37, 113.71 (2 CN); 118.66 ($C(OMe)_2$); 114.32, 121.38, 130.19, 159.30 (C arom.); 159.72 (C=N). IR (KBr), ν/cm^{-1}: 3016, 2248, 1684, 1520, 1256, 1180, 1136, 1008, 828.

2-Amino-6-(2-chlorophenyl)-1,5-dicyano-4,4-dimethoxy-3-azabicyclo[3.1.0]hex-2-ene (2e),[18] m.p. >350 °C. ^{1}H NMR (DMSO-d_6), δ: 3.33 (s, 1 H, CH); 3.37, 3.45 (both s, 3 H each, OMe); 7.45—7.75 (m, 6 H, H arom. and NH_2). ^{13}C NMR (DMSO-d_6), δ: 35.74, 37.55 (2 C); 40.04 (CH); 49.72, 51.63 (2 OMe); 112.14, 113.63 (2 CN); 118.74 ($C(OMe)_2$); 127.51, 127.74, 130.06, 130.13, 131.06, 134.77 (C arom.); 159.09 (C=N). IR (KBr), ν/cm^{-1}: 3388, 3082, 2256, 1688, 1432, 1176, 1114, 1056, 736.

2-Amino-6-(2-chlorophenyl)-1,5-dicyano-4,4-diethoxy-3-azabicyclo[3.1.0]hex-2-ene (2f),[18] m.p. >350 °C. ^{1}H NMR (DMSO-d_6), δ: 1.26, 1.31 (both t, 3 H each, Me, J = 7 Hz); 3.22 (s, 1 H, CH); 3.72, 3.88 (both q, 2 H each, OCH_2, J = 7 Hz); 7.39—7.74 (m, 6 H, H arom. and NH_2). ^{13}C NMR (DMSO-d_6), δ: 15.39, 15.63 (2 Me); 36.07, 38.55 (2 C); 40.14 (CH); 58.30, 60.09 (2 OCH_2); 112.59, 114.04 (2 CN); 118.47 ($C(OMe)_2$); 127.99, 130.37, 130.52, 131.58, 135.13 (C arom.); 159.14 (C=N). IR (KBr), ν/cm^{-1}: 3400, 3076, 2984, 2248, 1660, 1440, 1144, 1080, 760.

2-Amino-6-(3-bromophenyl)-1,5-dicyano-4,4-dimethoxy-3-azabicyclo[3.1.0]hex-2-ene (2g),[18] m.p. 261—263 °C. ^{1}H NMR (DMSO-d_6), δ: 3.33, 3.38 (both s, 3 H each, OMe); 3.51 (s, 1 H, CH); 7.45—7.70 (m, 5 H, H arom. and NH_2); 7.78 (m, 1 H, H arom.). ^{13}C NMR (DMSO-d_6), δ: 35.86, 37.81 (2 C); 40.82 (CH); 50.07, 51.75 (2 OMe); 112.57, 113.88 (2 CN); 119.12 ($C(OMe)_2$); 122.36, 128.35, 131.51, 132.31, 132.55, 132.71 (C arom.); 159.45 (C=N). IR (KBr), ν/cm^{-1}: 3444, 3072, 2244, 1676, 1428, 1416, 1172, 1140, 1076, 776.

2-Amino-1,5-dicyano-6-(4-iodophenyl)-4,4-dimethoxy-3-azabicyclo[3.1.0]hex-2-ene (2h),[18] m.p. >350 °C. ^{1}H NMR (DMSO-d_6), δ: 3.40, 3.42 (both s, 3 H each, OMe); 3.54 (s, 1 H, CH); 7.41 (d, 2 H, H arom., J = 8 Hz); 7.65 (s, 2 H, NH_2); 7.96 (d, 2 H, H arom., J = 8 Hz). ^{13}C NMR (DMSO-d_6), δ: 35.29, 37.45 (2 C); 40.91 (CH); 49.60, 51.22 (2 OMe); 95.86 (C arom.); 112.10, 113.41 (2 CN); 118.63 ($C(OMe)_2$); 129.49, 130.96, 137.68 (C arom.); 159.04 (C=N). IR (KBr), ν/cm^{-1}: 3436, 3064, 2248, 1684, 1428, 1172, 1140, 1104, 1008.

2-Amino-1,5-dicyano-4,4-diethoxy-6-(4-iodophenyl)-3-azabicyclo[3.1.0]hex-2-ene (2i), m.p. >350 °C. Found (%): C, 46.67; H, 3.85; I, 28.87; N, 12.71. $C_{17}H_{17}IN_4O_2$. Calculated (%): C, 46.80; H, 3.93; I, 29.09; N, 12.84. ^{1}H NMR (DMSO-d_6), δ: 1.26, 1.30 (both t, 3 H each, Me, J = 7 Hz); 3.52 (s, 1 H, CH); 3.73, 3.86 (both q, 2 H each, OCH_2, J = 7 Hz); 7.42 (d, 2 H, H arom., J = 8 Hz); 7.59 (s, 2 H, NH_2); 7.94 (d, 2 H, H arom., J = 8 Hz). ^{13}C NMR (DMSO-d_6), δ: 15.01, 15.22 (2 Me); 35.21, 38.02 (2 C); 40.82 (CH); 57.72, 59.31 (2 OCH_2); 96.21 (C arom.); 112.11, 113.42 (2 CN); 118.12 ($C(OMe)_2$); 129.43, 130.62, 137.63 (C arom.); 158.71 (C=N). IR (KBr), ν/cm^{-1}: 3421, 3076, 2973, 2248, 1676, 1440, 1144, 1080, 1044.

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