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Cyanoethylation of the salts of cyanoguanidine in aprotic solvents

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The reactions of lithium or sodium salts of cyanoguanidine (**1**) with acrylonitrile in aprotic solvents result in the rapid formation of *N,N*-bis(2-cyanoethyl)-*N'*-cyanoguanidine (**3**) followed by a slower intramolecular cyclization of **3** leading to 4-amino-1-(2-cyanoethyl)-5,6-dihydro-2-pyrimidinylidenecyanamide (**4**). A tentative assignment of the tautomeric forms to the cyclic derivatives of (β-cyanoethyl) cyanoguanidines and the products of their hydrolysis is described.

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L'addition de sels de lithium et de sodium de la cyanoguanidine (**1**) sur l'acrylonitrile en milieu non protique conduit à la formation rapide de la *N,N*-bis(cyano-2 ethyl)cyanoguanidine (**3**) qui subit une cyclisation intramoléculaire plus lente pour donner l'amino-4 cyanimino-2-(cyano-2 ethyl)-1 dihydro-5,6 pyrimidine (**4**). Les formes tautomèrs de dérivés cycliques de (cyano-2 ethyl)cyanoguanidines et de produits de leur hydrolyse sont discutées.

The present investigations have been made in consideration of cyanoguanidine derivatives as monomers to make them available for polyguanamine synthesis (1).

The reaction of cyanoguanidine with acrylonitrile in the presence of strong alkaline bases such as sodium hydroxide was employed to prepare syrups suitable for the impregnation of textiles (2). All our attempts to isolate or identify distinct products of this reaction were unsuccessful; complex reaction mixtures resulted apparently from several competitive and consecutive reactions such as conjugate additions, inter- and intramolecular cyclizations, etc. On the basis of preliminary experiments it appeared to us that the cyanoethylation process should be favored with respect to others by a high concentration of cyanoguanidine anion providing the reaction time was sufficiently short. To confirm this prediction we have carried out the cyanoethylation of lithium and sodium salts of **1** in dipolar aprotic solvents. Cyanoguanidine, a weak acid of $pK_a^{25} = 12$ (3), was converted into its lithium or sodium salt by reaction with lithium or sodium hydride, respectively, in dimethylformamide or dimethyl sulfoxide. Upon rapid addition of an equimolar amount of acrylonitrile an exothermic reaction occurred which subsided after 1–2 min; stirring was continued for another 8–13 min, followed by quenching the reaction with an equimolar amount of an acid. A crude product was proved to consist only, apart from starting material, of the 1:2 adduct **4** of cyanoguanidine (**1**) and acrylonitrile. The same reaction brought about under milder conditions resulted in a main product **3** of the same molecular weight but for which both the melting point and spectra were quite different from those for **4**. On the basis of spectra and chemical behaviour **3** was identified as *N,N*-bis(2-cyanoethyl)-*N'*-cyanoguanidine. The ir spectrum of **3** shows four intense bands in the N—H stretching vibration region. The strong sharp band at 3438 cm^{-1} is likely related to the NH_2 group. If this is so, the strong band at 1660 cm^{-1} which disappears on deuteration will be the NH_2 scissoring band. Two weak absorptions at 2258 and 2249 are due to the C≡N stretching vibration in the cyanoethyl group, although the nature of this splitting is not clear. The strong band at 2174 with a shoulder at 2185 cm^{-1} is undoubtedly due to the stretching vibration of the =N''—CN grouping and may be compared to the similar bands in **1**; similarly, the NCN' and NCN'' asymmetric vibrations may be tentatively assigned to a broad strong band with maxima at 1554 and 1523 cm^{-1} (4). A proper choice of structure **3** among three possible bis(2-cyanoethyl)-cyanoguanidines was confirmed by treatment of **3** with barium hydroxide. The resulting monobarium salt of iminodipropionic acid was converted into monoammonium salt which was identical with the product of the independent synthesis (5).

Before discussing the nmr spectrum of **3**, which is not contradictory with the proposed structure, it seems that the nmr spectra of cyanoguanidine and 5,6-dihydropyrimidine derivatives described in this paper merit some general comment. Unlike the nmr spectra of some 5,6-dihydropyrimidine derivatives (6), so far as we know, those of cyanoguanidines have

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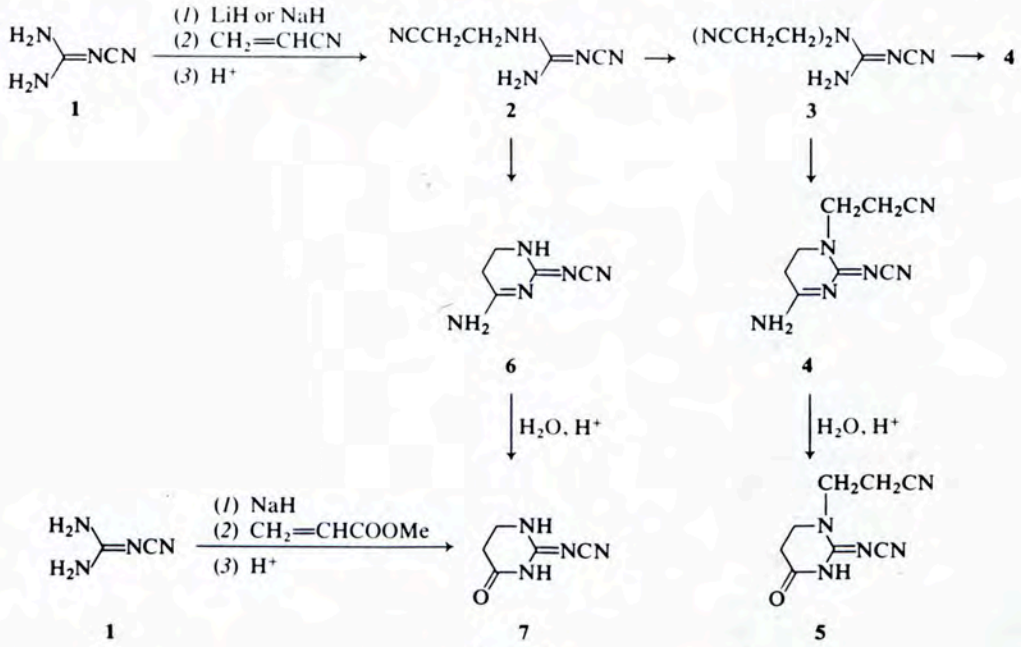
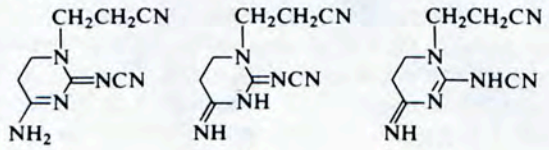


SCHEME 1

not been studied yet. It seems, generally, that for both cyanoguanidines and dihydropyrimidines a N—H proton exchange in aprotic solvents is slow on the nmr time scale, and this is probably due to both low acidity and basicity of these compounds. On the other hand, quadrupolar relaxation appears to be rather efficient, leading to relatively narrow signals of the N—H resonances in these types of compound unless the NH group is adjacent to a carbonyl function, and therefore, in some cases to a splitting of these resonances resulting from coupling to α-carbon protons (7). In the course of another investigation it was found, e.g., that the N—H protons in *N*-(methyloxymethyl)-*N'*-cyanoguanidine in $(CD_3)_2SO$ show a triplet at 7.64 ppm and a singlet at 6.98 ppm which correspond to the —NH— and $—NH_2$ groups, respectively. The N—methylene resonance appears as a doublet resulting from coupling to the NH proton which itself, therefore, constitutes a triplet.[2]

The nmr spectrum of **3** in $(CD_3)_2SO$ reveals a single peak at 7.36 ppm due to two protons of the NH_2 group and pair of triplets at 3.63 and 2.74 ppm due to the CH_2N and CH_2CN groups, respectively, coupled to each other (J = 6.9 Hz).

A close relationship between **3** and **4** was established by the observation that **3** was easily converted to **4** on being heated above its melting point. In the infrared spectrum of **4**, in addition to strong bands at 3403 and 1665 cm^{-1} due to the NH_2 group, the high intensity doublet at 2172 and 2189 cm^{-1} related to the N''—CN grouping, and the medium-weak intensity band at 2255 cm^{-1} due to the aliphatic nitrile, there are several additional strong bands in the region 1300–1600 cm^{-1}. Some of these bands may be assigned to a dihydropyrimidine ring although this assignment must still be regarded as tentative. The above data suggest that **4** is the product of intramolecular cyclization in which the amino and cyano groups are involved. The cyclic product **4** could, in principle, exist in three tautomeric forms, **4***a*–**4***c*, but it appears that its infrared spectrum rules out, at least in the solid state, **4***b* and **4***c* (Scheme 2).



4*a* **4***b* **4***c*

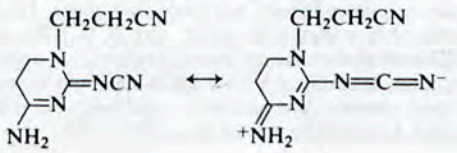
SCHEME 2

In the nmr spectrum of **4** there are two pairs of triplets in the methylene region. Assuming that the cyclization does not affect essentially the chemical shifts of methylenes in the cyanoethyl group in **4** with respect to those in **3** we have assigned the triplets at 3.62 and 2.76 ppm to the CH_2N and CH_2CN groups, respectively, in the cyanoethyl group. Some support for this assignment has been obtained from the observation that the addition of a drop of trifluoroacetic acid does not affect these

[2]M. Bukowska and M. Maciejewski. Unpublished results.

resonances. The other two triplets at 3.42 and ca. 2.5 ppm we have assigned to the CH_2N and $CH_2C=$ groups of the 5,6-dihydropyrimidine ring. The fact that these resonances appear as triplets instead of as more complex multiplets due, e.g., to an AA'BB' system might be perhaps explained on a stereochemical basis as has been done for dihydrouracil (6). In the NH region there are two singlets at 8.05 and 8.34 ppm ($w_{1/2}$ ca. 6 Hz) each integrating for one proton. If **4** had structure **4***b* or **4***c* it would be difficult to explain this relatively high deshielding of an imine proton, for it is known that in somewhat similar structures the imine proton resonances occur at ca. 6 ppm (8, 9). On the other hand, structure **4***a* can be assigned from its nmr spectrum, assuming that the nonequivalence of the two amino protons results from hindered rotation about the C—NH_2 bond owing to its partial double bond character. Both the restricted rotation and the relatively high deshielding of the amino protons can be attributed to a marked contribution of structure **4***a'* to a resonance hybrid. Nuclear magnetic resonance temperature variation experiments and uv spectra support also the structure of 4-amino-1-(2-cyanoethyl)-2(1*H*)-pyrimidinylidenecyanamide (**4***a*) (*vide infra*).

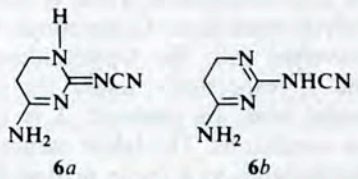
Several runs performed with variation of molar ratio of reactants, time, and temperature led us to the supposition that (cyanoethyl)cyanoguanidine anion resulting from addition of the guanidine anion to acrylonitrile undergoes addition to another molecule of acrylonitrile, with the formation of the asymmetric derivative **3**, at a rate much higher than that of the first step. The subsequent intramolecular cyclization reaction evidently occurs more slowly than the cyanoethylation reaction under these conditions. Analysis by nmr spectroscopy of samples withdrawn at various reaction times offered some support for this assumption. The nmr spectrum of a sample taken after 3 min (starting with the addition of acrylonitrile at room temperature, molar ratio 1:1) revealed in the NH region, apart from the cyanoguanidine peak, three signals at 6.9, 7.0, and 7.4 ppm which might be attributed to the NH resonances of bis(2-cyanoethyl)- and probably (2-cyanoethyl)cyanoguanidine (**2**) (Scheme 1). The lack of cyclic products in this earlier stage of the reaction was also shown in the methylene region where there were only two multiplets centered at 3.67 and 2.79



SCHEME 3

ppm. On continuing the reaction (7–17 min) the above mentioned NH resonances disappeared and the new ones appeared in the 8 ppm region; also in the methylene region there appeared two pairs of triplets as those in **4**. Longer reaction times (45 min and more) led to substantial changes in both the NH and methylene regions of the nmr spectrum, and gave unidentifiable syrups.

Isolation of cyanoethylation products was generally difficult due to their low solubility in common solvents and to the unstable nature of the cyclic products to hydrolysis (*vide infra*). All our attempts to isolate *N*-(2-cyanoethyl)-*N'*-cyanoguanidine (**2**) failed. In the course of the laborious and tedious process of multiple crystallizations the NH signals of the supposed monocyanoethyl derivative **2** disappeared and new signals in the 8 ppm region appeared. In fact, we have only been able to obtain a very small amount of crystalline product, presumably a side effect of the isolation procedure, assigning to it structure **6**. Microanalysis and mass spectrometric measurement show a molecular composition for **6** consistent with that of a 1:1 adduct of **1** and acrylonitrile. The ir spectrum of **6** displays no band in the aliphatic nitrile region and, therefore, indicates clearly the absence of the cyanoethyl group in the molecule. The strong bands at 3435 and 1666 cm^{-1}, disappearing on deuteration, indicate the presence of the amino group and this suggests that **6**, at least in the solid state, is similar to **4** and exists in the tautomeric form with the amino group at C-4. This excludes from further considerations two of four possible tautomeric forms, and the choice between the other two, the cyanoimino **6***a* and the cyanoamino form **6***b* may also be made tentatively by



SCHEME 4

means of its ir spectrum. The former appears to be the more likely, based on the observed low frequency of a high intensity band at 2165 cm^{-1} due to the —NC≡N grouping as compared with an expected higher frequency of the —NHC≡N group, reinforced by a cross-conjugation in **6***b* (10, 11). The uv spectra of **4** and **6** are consistent with structures **4***a* and **6***a* in that a strong absorption at ca. 250 nm may provide evidence for the presence of a conjugated cyanoguanidine chromophore in their molecules. Indicative of a more conjugated system in **4** than in its open-chain precursor **3** is a bathochromic shift

of the wavelength maximum, $\Delta\lambda = 32$ nm, in the ultraviolet spectrum after cyclization.

The nmr spectrum of **6** reveals a pair of triplets at 2.44 and 3.22 ppm which we have assigned to the $CH_2C{=}$ and CH_2N groupings, respectively. The NH protons give rise to three peaks at 8.32 ($w_{1/2}$ ca. 14 Hz), 8.07 ($w_{1/2}$ ca. 14 Hz), and 7.84 ($w_{1/2}$ ca. 7 Hz), each with an area due to one proton. The two lower field peaks are attributable, by analogy to **4**, to two protons of the amino group with hindered rotation due to partial double bond character in structure ***6a***. The different nature of these protons with respect to that at 7.84 ppm can be shown by temperature variation experiment. The two lower field resonances coalesce in $(CD_3)_2SO$ at around 80°C, while the higher field peak may be distinctly seen even at 110°C. Similarly, the NH proton resonances in **4** coalesce at about 80°C. Some ambiguity remains in assignment of the higher field NH peak and, therefore, in the choice between structures ***6a*** and ***6b*** for this compound in a DMSO solution. First of all, tautomeric structure ***6a*** might be expected to reveal coupling between H-1 and H-6, and the lack of splitting of the CH_2N resonance due to this coupling, if not fortuitous, might show evidence for ***6b***. On the other hand, the NHCN proton resonance in ***6b*** might be expected to occur at lower field than that experimentally determined. Since, intuitively, chemical resonance in ***6b*** does not seem to be sufficiently significant to hinder a rotation of the amino group, we have tentatively assigned structure ***6a*** to this compound in the DMSO solution too.

A striking difference between the cyanoethyl derivative of cyanoguanidine **3** and the cyclic products of cyanoethylation, **4** and **6**, can be seen in their hydrolytic behaviour. Compounds **4** and **6** are rapidly converted into the 4-pyrimidinone derivatives, **5** and **7**, respectively, under the influence of dilute mineral acid. In contrast, **3** is quite stable under these conditions. The labile nature of **4** and **6** is likely attributable to a facile protonation of N-3 resulting in some increase of positive charge at C-4. This, consequently, would facilitate the attack of a water molecule at C-4. This suggestion may be somewhat substantiated by the nmr spectrum of **4** in which, upon the addition of a drop of trifluoroacetic acid to its $(CD_3)_2SO$ solution, the NH_2 doublet collapses into a broad singlet and the $CH_2C{=}$ resonance is shifted a little downfield. Both **5** and **7** show a strong carbonyl band at ca. 1730 cm^{-1} which is only slightly shifted towards the lower frequency on deuteration. The relatively high values of the C=O and N—C≡N stretching frequencies in **5** and **7** could be likely attributed to a cross-conjugation between the carbonyl and cyanoguanidine moieties in these compounds. Their uv spectra are consistent with that conclusion. In contrast to **4** and **6** which exhibit only one band at λ_{max} ca. 250 nm due to a conjugation of cyanoguanidine chromophore, **5** and **7** display two bands at λ_{max} ca. 215 and 240 nm.

The cyclic structure of **5** and **7** was also confirmed chemically by the reaction of the sodium salt of **1** with methyl acrylate (12) resulting in a product which was identical with that obtained by acid hydrolysis of **6** (Scheme 1).

The nmr spectrum of **5** exhibits in the methylene region two pairs of triplets integrating for eight protons; these centered at 3.74 ($J = 7.0$ Hz) and 2.67 ppm we have assigned to NCH_2 and CH_2CO of the 5,6-dihydro-4-pyrimidinone ring, respectively, and the other two centered at 3.64 and 2.82 ppm ($J = 6.7$ Hz) to NCH_2 and CH_2CN, respectively, of the cyanoethyl group. A broad band at 10.9 ppm integrating for one proton can be attributed to the N-3 proton adjacent to the C=O and C=NCN groups. This accounts for a marked deshielding of this proton. The nmr spectrum of **7** shows a pair of triplets at 3.42 (3H, $J = 7$ Hz) and ca. 2.5 ppm partly obscured by residual DMSO due to the CH_2N and CH_2CN groups respectively. In the NH region there is a broad featureless resonance at 10.65 and the other at 8.72 ($w_{1/2} = 20$ Hz), each integrating for one proton, assigned to the N-3 and N-1 protons, respectively. Although a tentative choice between the cyanoimimo- and cyanoamino tautomer in favour of the former was based on the ir and uv spectra, some ambiguity persists in that the N-1 proton is more strongly deshielded than might be expected as compared with similar structures, and in the lack of splitting of the NCH_2 resonances due to coupling to this proton.

Experimental

All melting points are uncorrected. Dimethylformamide and dimethyl sulfoxide were dried by refluxing over calcium hydride and distilled *in vacuo* in an argon atmosphere. Acrylonitrile was distilled just before use. Reagent grade cyanoguanidine was used without further purification.

Ultraviolet spectra were measured in water with a Cary Model 118 C spectrophotometer. Infrared spectra were recorded on a Perkin-Elmer Model 577 spectrometer. Proton magnetic resonance spectra were recorded with a JEOL JMN-MH 100 spectrometer in δ values with $(CD_3)_2SO$ as solvent and tetramethylsilane as an internal reference standard. Mass spectra were determined with a LKB-9000 instrument (70 eV, direct inlet system). The elemental analyses were made on an Elemental Analyser Perkin-Elmer CHNO 240.

Preparation of the Lithium Salt of Cyanoguanidine

A solution of 0.1–0.2 mol of **1** in 50 cm^3 of DMSO or DMF was added to a suspension of an equimolar amount of lithium hydride in 50 cm^3 of the same solvent. The mixture was stirred and heated at 50–60°C under argon until no further evolution of hydrogen was observed.

Preparation of the Sodium Salt of Cyanoguanidine

A solution of 0.1–0.2 mol of **1** was added with stirring and cooling to a solution of an equimolar amount of the sodium salt of dimethyl sulfoxide prepared from DMSO and sodium hydride by heating at 70°C in an argon atmosphere. The sodium salt of **1** was prepared prior to use.

4-Amino-1-(2-cyanoethyl)-5,6-dihydro-2(1H)-pyrimidinylidene-cyanamide (4)

A. Acrylonitrile (5.3 g, 0.1 mol) was added rapidly to 50 cm^3 of a DMSO solution of 0.1 mol of the lithium salt of **1** with stirring and water cooling under an argon atmosphere. The temperature rose immediately to 67°C and began rapidly to fall. After 10–15 min the reaction mixture was poured into 150 cm^3 of ice-chilled tetrahydrofuran. The resulting slurry mixture was centrifuged to remove an amorphous precipitate. Tetrahydrofuran and most of the dimethyl sulfoxide were distilled off *in vacuo*. A semisolid residue was triturated with ethanol yielding 6.5 g of crystalline product **4**, mp 226–229°C (yield 68%).

B. Acrylonitrile (10.6 g, 0.2 mol) was added rapidly to 50 cm^3 of a DMSO solution containing 0.1 mol of the sodium salt of **1**. The temperature rose to 86°C. After 10 min the reaction mixture was subject to the same work-up as described in *A*, yielding **4** in 86% yield. Crystallization from water gave an analytical sample of mp 233–234°C; ir (KBr) ν_{max}: 3403 (NH_2), 3340, 3165 (NH), 2255 (C≡N), 2172 and 2189 (N—C≡N), 1665 (NH_2) cm^{-1}; uv λ_{max} (log ε): 255 (4.42); ms *m/e*: 190 (M^+); nmr δ: ca. 2.5 (CH_2C=), 3.42 (t, 2H, J = 7 Hz, ring CH_2N), 2.76 (t, 2H, J = 6.6 Hz, CH_2CN), 3.62 (t, 2H, J = 6.6 Hz, CH_2N), 8.05 (s, $w_{1/2}$ = 6.6 Hz, 1H, NH), 8.34 (s, $w_{1/2}$ = 6.6 Hz, 1H, NH). *Anal.* calcd. for $C_8H_{10}N_6$: C 50.52, H 5.30, N 44.18; found: C 50.85, H 5.45, N 43.59.

N,N-*Bis(2-cyanoethyl)*-N′-*cyanoguanidine (3)*

To a stirred solution of 0.118 mol of the lithium salt of **1** in 100 cm^3 of dimethylformamide at 3°C under argon was added dropwise 12.5 g (0.236 mol) of acrylonitrile in 30 cm^3 of DMF. The reaction mixture was stirred for 1 h at around 3°C, and then poured into 200 cm^3 of chilled tetrahydrofuran with 0.06 mol of sulfuric acid. After centrifuging an amorphous solid and evaporating the solvents *in vacuo*, a semisolid residue was triturated with 100 cm^3 of acetone yielding 18.6 g of **3** (yield 82%). Crystallization from water gave an analytical sample of mp 166–167°C; ir (KBr) ν_{max}: 3438, 3320 (NH_2), 3192, 3164 (NH), 2258, 2249 (C≡N), 2174, 2185 (sh N—C≡N), 1660 (NH_2), 1554, 1523 cm^{-1}; uv λ_{max} (log ε): 223 (4.29) nm; nmr δ: 2.74 (t, 2H, J = 6.9 Hz, CH_2CN), 3.63 (t, 2H, J = 6.9 Hz, CH_2N), 7.36 (s, 2H, NH_2); ms *m/e*: 190 (M^+). *Anal.* calcd. for $C_8H_{10}N_6$: C 50.52, H 5.30, N 44.18; found: C 50.49, H 5.26, N 44.30.

4-Amino-5,6-dihydro-2(1H)-pyrimidinylidenecyanamide (6)

To a stirred solution of 0.08 mol of the salt of **1** in 35 cm^3 of dimethyl sulfoxide was added dropwise 4.27 g (0.08 mol) of acrylonitrile. Throughout the addition the mixture was cooled in a water bath and the temperature did not rise above 30°C. When the exothermic reaction subsided the mixture was removed from the water bath, stirred for 30 min at room temperature, and then poured into a chilled solution of 0.08 mol of sulfuric acid in 80 cm^3 of acetone. A crystalline material was recrystallized from water yielding 3.4 g of **4**. The filtrate was concentrated *in vacuo* yielding a material which was found to be a mixture of **1**, **3**, **4**, and an alleged monocyanoethyl derivative **2** (see the text). The multiple crystallizations from acetone gave, apart from **3** and **4**, a small amount (60 mg) of **6**, mp 250–252°C; ir (KBr) ν_{max}: 3455 (NH_2), 3235, 3145 (NH), 2165, 2170 (sh, N—C≡N), 1666 (NH_2) cm^{-1}; uv λ_{max} (log ε): 253 (4.31) nm; nmr δ: ca. 2.44 (CH_2C=), 3.22 (t, 2H, J = 7 Hz, CH_2N), 7.84 (s, $w_{1/2}$ = 9 Hz, 1H, NH), 8 07 (s, $w_{1/2}$ = 14 Hz, 1H, NH), 8.32 (s, $w_{1/2}$ = 14 Hz, 1H, NH); ms *m/e*: 137 (M^+). *Anal.* calcd. for $C_5H_7N_5$: C 43.79, H 5.14, N 51.07; found: C 43.56, H 5.17, N 50.77.

Conversion of 3 into 4

One hundred milligrams of **3** was heated above its melting point. After several seconds the liquid substance began to resolidify yielding 100 mg of a crystalline product which was proved to be identical with **4**.

Hydrolysis of 3 with Barium Hydroxide

A mixture of 0.58 g (0.003 mol) of **3** and 3 g of barium hydroxide octahydrate in 20 cm^3 of water was heated under reflux until the evolution of ammonia ceased. The mixture was saturated with carbon dioxide and the precipitate was filtered off. The filtrate was evaporated *in vacuo* to dryness. The resulting monobarium salt was dissolved in a small amount of water and treated with an equivalent amount of ammonium sulfate. After filtering off barium sulfate the filtrate was concentrated and diluted with methanol. The precipitate was recrystallized twice from aqueous methanol yielding a crystalline product, mp 178–179°C, which was proved to be identical with the ammonium salt of β,β′-iminobispropionic acid obtained by alkaline hydrolysis of β,β′-iminobispropanenitrile.

Hydrolysis of 6

An equivalent amount of hydrochloric acid was added dropwise, as it was consumed, to a stirred suspension of 0.027 g (2×10^{-4} mol) of **6** in 3 cm^3 of water at 40°C. The addition was continued until a slight excess of hydrochloric acid was present. The colorless crystals were filtered off and washed with water yielding 0.024 g of tetrahydro-4-oxo-2(1*H*)-pyrimidinylidenecyanamide (**7**), mp 273–274°C; ir (KBr) ν_{max}: 3200, 3095, 3060 (NH), 2200, 2185 (sh, N—C≡N), 1725 (C=O) cm^{-1}; uv λ_{max} (log ε): 216 (4.22), 239 (4.17) nm; nmr δ: ca. 2.5 (CH_2CO), 3.42 (t, 2H, J = 7 Hz, CH_2N), 8.72 (s, $w_{1/2}$ ca. 20 Hz, 1H, NH), 10.65 (br s, 1H, NHCO); ms *m/e*: 138 (M^+). *Anal.* calcd. for $C_5H_6N_4O$: C 43.48, H 4.38, N 40.56; found: C 43.28, H 4.65, N 40.46.

Hydrolysis of 4

Compound **4** (0.19 g) was hydrolysed in a similar way to that described above for **6** yielding 0.17 g of 1-(2-cyanoethyl)-tetrahydro-4-oxo-2(1*H*)-pyrimidinylidenecyanamide (**5**), mp 148–149°C; ir (KBr) ν_{max}: 3175 (NH), 2256 (C≡N), 2196 (N—C≡N), 1731 (C=O) cm^{-1}; uv λ_{max} (log ε): 218 (4.16), 244(4.17) nm; nmr δ: 2.67 (t, 2H, J = 6.6 Hz, CH_2CO), 3.74 (t, 2H, J = 6.6 Hz, CH_2N ring), 2.82 (t, 2H, J = 6.7 Hz, CH_2CN), 3.64 (t, 2H, J = 6.7 Hz, CH_2N), 10.92 (br s, 1H, NH); ms *m/e* 191 (M^+). *Anal.* calcd. for $C_8H_9N_5O$: C 50.26, H 4.75, N 36.63; found: C 50.31, H 4.71, N 36.44.

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