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Studies on Pyrazolo[3,4-*d*]pyrimidine Derivatives. XIV.[1] Preparation and Reactions of 1-Phenyl-1*H*-pyrazolo-[3,4-*d*]pyrimidine Reissert Compound

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The Reissert reaction of 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (**4**) in anhydrous methylene chloride using benzoyl chloride, trimethylsilyl cyanide, and a catalytic amount of aluminium chloride gave the corresponding Reissert compound (**3**, 5-benzoyl-4,5-dihydro-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile) in 95% yield.

The alkaline hydrolysis of **3** in methanol resulted in the formation of **4**, benzoic acid (**7**), and the 4,4′-dimer (**9**) of **4**. The acid hydrolysis in dimethyl sulfoxide and in methanol proceeded with ring fission to give 5-amino-α-benzamido-1-phenyl-1*H*-pyrazole-4-acetonitrile (**13**), the acetamide (**14**), and the acetate (**15**). Compound **3** reacted with sodium hydride in dimethylformamide to give **4**, **9**, 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile (**20**), α,1-diphenyl-1*H*-pyrazolo[3,4-*d*]-pyrimidin-4-ylmethyl benzoate (**21**), and *O*-benzoylmandelonitrile (**22**).

In the present paper, we compare the chemical properties of **3** with those of the isoquinoline Reissert compound (**1**, 2-benzoyl-1,2-dihydro-1-isoquinolinecarbonitrile).

Keywords——pyrazolopyrimidine Reissert compound; preparation; chemical property; hydrolysis; pyrazoleacetonitrile; carbanion; rearrangement; pyrazolopyrimidinemethanol

It is well known that 2-benzoyl-1,2-dihydro-1-isoquinolinecarbonitrile,[2] the so-called isoquinoline Reissert compound (**1**), is easily prepared by the reaction of isoquinoline (**2**) with benzoyl chloride in aqueous potassium cyanide.[3] Since **1** can be used as the key compound in the introduction of a carbon function group into the 1-position of the isoquinoline ring system by reaction of the anion of **1** with electrophiles, much work has been done on **1**.[3]

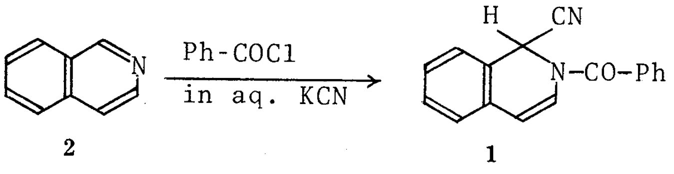

Chart 1

In the pyrazolo[3,4-*d*]pyrimidine area, it was reported that when attempts[4] were made to form 5-benzoyl-4,5-dihydro-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile (**3**, the 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine Reissert compound) by the standard method[3] using benzoyl chloride and aqueous potassium cyanide, 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (**4**) underwent ring fission, resulting in the formation of 5-amino-1-phenyl-1*H*-pyrazole-4-carbaldehyde (**5**)[5] and *N*-formylbenzamide (**6**).[6] Thus, the method in aqueous media is not effective for the preparation of **3**, which has not been described in the literature. In the present paper, we describe a convenient one-step preparation and some reactions of **3**, and compare

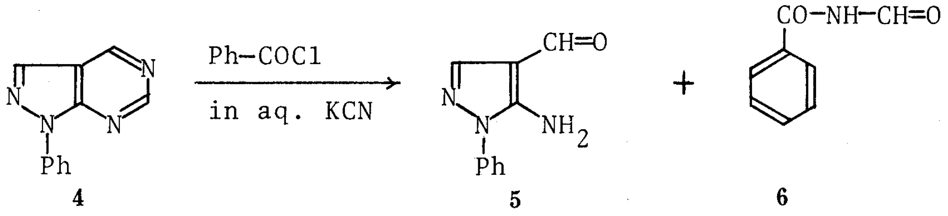

Chart 2

the chemical properties of **3** with those of **1**.

Recently, Ruchirawat *et al.* carried out Reissert compound formation on **2** in anhydrous media using benzoyl chloride, trimethylsilyl cyanide, and a catalytic amount of aluminium chloride, which was an effective catalyst, in methylene chloride, and succeeded in obtaining **1**.[7] The preparation of **3** was achieved by application of the above procedure to **4**. Thus, the desired Reissert compound **3**, mp 175—176 °C, was obtained in 95% yield when **4** in methylene chloride was stirred for 13 h at room temperature with equimolar amounts of trimethylsilyl cyanide and benzoyl chloride in the presence of a catalytic amount of aluminium chloride.

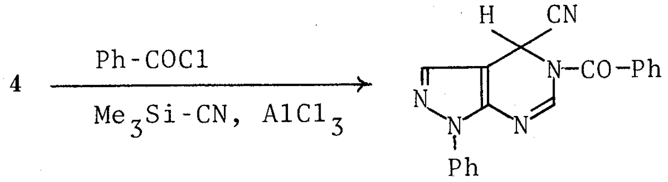

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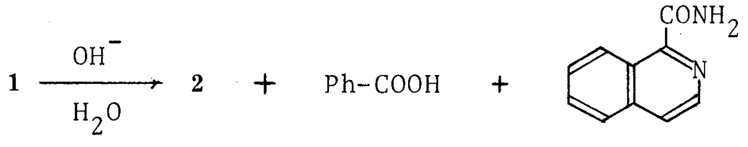
Chart 3



7 **8**

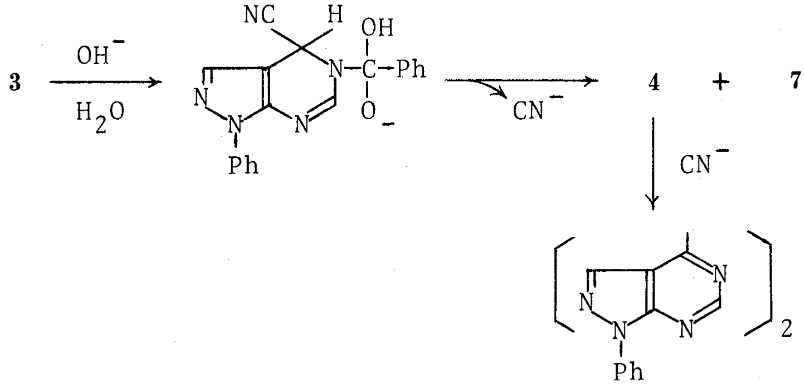


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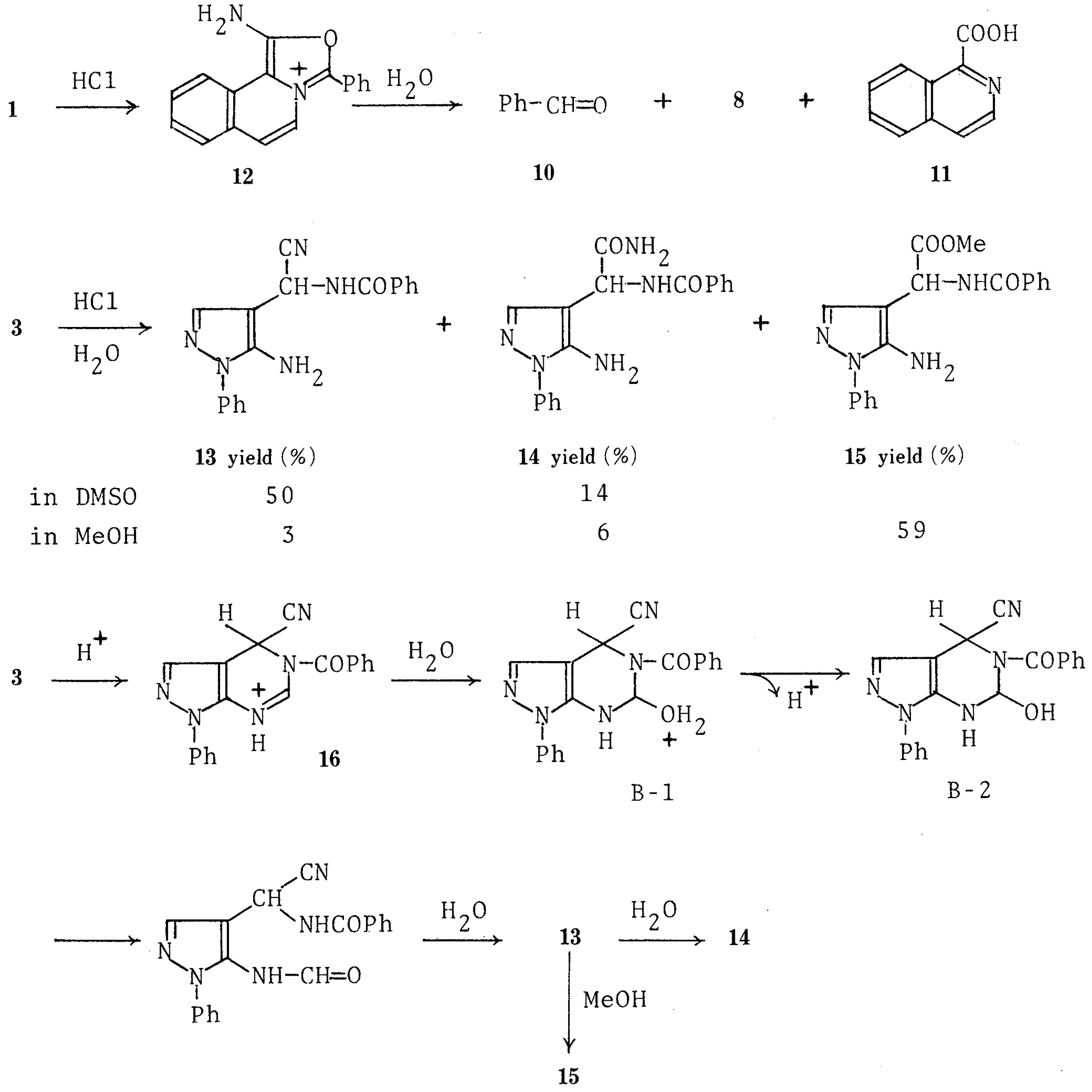
Chart 4

The infrared absorption (IR) spectrum of **3** showed a carbonyl absorption peak (1680 cm^{-1}), and did not show any absorption due to the cyano group. The latter result is compatible with the reported absence of the absorption peak of a cyano group located at an electron-deficient carbon, such as in *O*-benzoylmandelonitriles.[1] The ^{1}H-nuclear magnetic resonance (^{1}H-NMR) spectrum showed a characteristic singlet due to C^{4}-H (6.3 ppm). Moreover, the ^{13}C-NMR spectrum showed two doublets due to C^{4} (41.8 ppm) and C^{6} (143.5 ppm), and two singlets due to the cyano (115.6 ppm) and carbonyl (169.5 ppm) carbons. The elemental analyses were consistent with the structure of 5-benzoyl-4,5-dihydro-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile (**3**).

It was reported that the alkaline hydrolysis of **1** in methanol resulted in the formation of **2**, benzoic acid (**7**), and 1-isoquinolinecarboxamide (**8**).[8]

Under the same conditions **3** was similarly hydrolyzed to give **4** and **7** together with 4,4′-bis[1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine] (**9**)[9] as a by-product. The reaction may occur by the initial attack of hydroxide ion at the carbonyl carbon, followed by the ready loss of a cyanide ion, leading to **4** and **7**, as shown in Chart 4. Reaction of the resulting **4** with cyanide ion causes dimerization to give **9**. Compound **9** was identical with a sample prepared by another route.[9]

It was reported by Reissert that **1** was hydrolyzed in an acid medium to give benzaldehyde (**10**) plus **8** and 1-isoquinolinecarboxylic acid (**11**).[10] Moreover, Katritzky *et al.*



	13 yield (%)	14 yield (%)	15 yield (%)
in DMSO	50	14	
in MeOH	3	6	59

Chart 5

reported that the salt (**12**) isolated by the treatment of **1** with acid should be regarded as having the oxazolo[4,3-*a*]isoquinolinium structure, as shown in Chart 5.[11)]

On the other hand, reaction of **3** with acid in dimethyl sulfoxide (DMSO) proceeded in a different way from that of **1**, and resulted in the formation of 5-amino-α-benzamido-1-phenyl-1*H*-pyrazole-4-acetonitrile (**13**) as a main product together with 5-amino-α-benzamido-1-phenyl-1*H*-pyrazole-4-acetamide (**14**) as a by-product. When methanol instead of DMSO was used as a solvent in the reaction, methyl 5-amino-α-benzamido-1-phenyl-1*H*-pyrazole-4-acetate (**15**) was mainly obtained together with **13** and **14**. The results are summarized in Chart 5. Moreover, the nitrile **13** was easily convertible into the amide **14** by means of the Radziszewsky reaction[12)] using hydrogen peroxide and aqueous sodium carbonate in acetone.

It is assumed that the initial step in the acid hydrolysis is the formation of the quaternary salt (**16**), which should be regarded as having the cyclic amidinium structure shown in Chart 5. Thus, the nucleophilic attack of water at the carbon of the cyclic amidinium moiety, followed by ring opening, leads to the nitrile **13**. In the second step, hydrolysis and methanolysis of the nitrile **13** give the amide **14** and the ester **15**, respectively.

The structures of **13**, **14**, and **15** were suggested by their elemental analyses, and

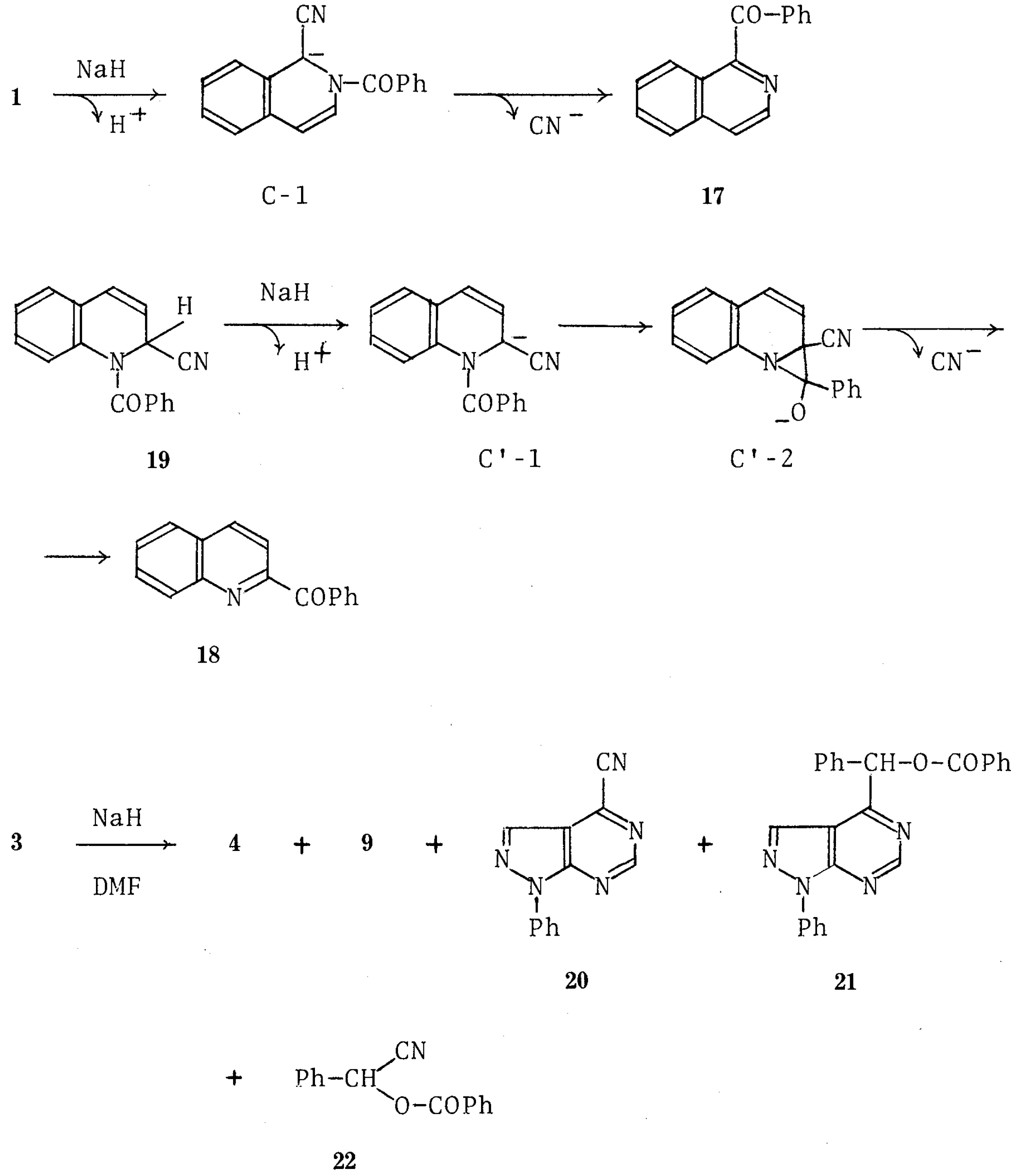


Chart 6

confirmed by analyses of their IR, ^{1}H-NMR, and ^{13}C-NMR spectra, as described in the experimental section.

It was reported by Boekelheide and Weinstock that the anion (C-1), which was generated by the removal of the acidic hydrogen at the 1-position of **1** with sodium hydride, rearranged to 1-benzoylisoquinoline (**17**) with expulsion of a cyanide ion.13) Moreover, McEwen and Cobb proposed a mechanism whereby 2-benzoylquinoline (**18**) arose from the anion (C′-1) of the quinoline Reissert compound (**19**) by way of the aziridine intermediate (C′-2) in an intramolecular process, as shown in Chart 6.3a)

In the case of **3**, the reaction with sodium hydride in dimethylformamide (DMF) took a different route from that of **1**, resulting in the formation of **4**, **9**,9) 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile (**20**),14) α,1-diphenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-ylmethyl benzoate (**21**), and *O*-benzoylmandelonitrile (**22**)15) in poor yields. Compounds **9**,9) **20**,14) and **22**15) showed underpressed melting points on admixture with the corresponding authentic samples. The structure of **21** was suggested by the elemental analysis, and confirmed by analyses of the IR and ^{1}H-NMR spectral data, as described in the experimental section.

The formation of the pyrazolopyrimidine **4**, the dimer **9**, the nitrile **20**, the ester **21**, and *O*-benzoylmandelonitrile **22** may be explained by the following three steps, as shown in Chart 7.

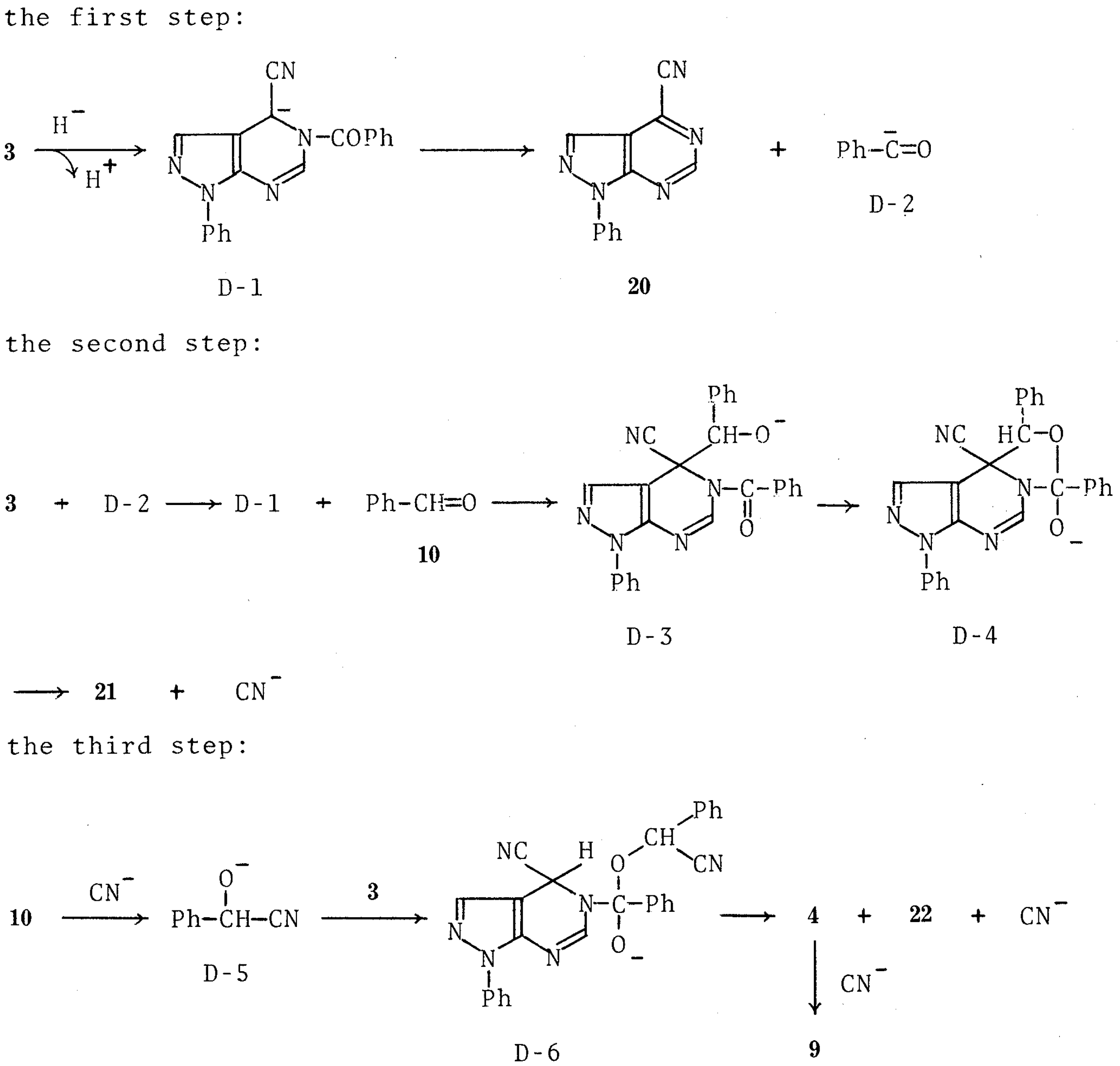


Chart 7

In the first step, the anion (D-1), which was initially generated by the removal of the acidic hydrogen at the 4-position of **3** with sodium hydride, leads to **20** with the expulsion of a benzaldehyde anion (D-2). The effect of the N^7-atom in the anion D-1 as well as the N^5-benzoyl group may favor the expulsion of the anion D-2 to yield the aromatic system rather than the formation of the corresponding aziridine intermediate.

In the second step, the nucleophilic addition of the anion D-1 to the carbonyl carbon of benzaldehyde (**10**), which is generated by the reaction of **3** with the resulting anion D-2, gives an intermediate (D-3). Then D-3 leads to the ester **21** with the expulsion of a cyanide ion by way of an intermediate (D-4), similar to the cyclic intermediate[16)] observed in the reaction of the anion C-1 with aromatic aldehydes.

The third step is the formation of the anion (D-5) of mandelonitrile by the addition of the resulting cyanide ion to benzaldehyde **10**, followed by nucleophilic addition to the carbonyl carbon of **3** to give **4** and **22** together with the expulsion of a cyanide ion. Compound **4** reacts with a cyanide ion to give the dimer **9**. The dimerization mechanism was previously proposed by Higashino *et al.*[9,17)]

Experimental

All melting points are uncorrected. IR spectra were recorded on a Jasco A-102 diffraction grating IR spectrometer. ^{1}H-NMR spectra were measured at 60 MHz on a Hitachi R-24B high-resolution NMR spectrometer, and ^{13}C-NMR spectra were taken at 90 MHz on a JEOL JNM-FX90Q FTNMR spectrometer. Chemical shifts are quoted in parts per million (ppm) with tetramethylsilane as an internal standard, and coupling constants (J) are given in Hz. The following abbreviations are used: s = singlet, d = doublet, q = quartet, m = multiplet, and br s = broad singlet. Mass spectra (MS) were recorded on a JEOL JMS D-100 mass spectrometer. Column chromatography was carried out on SiO_2, Wakogel C-200 (200 mesh).

Preparation of 5-Benzoyl-4,5-dihydro-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile (3)——A solution of benzoyl chloride (9.59 g, 68.25 mmol) in CH_2Cl_2 (30 ml) was added to a well stirred solution of **4** (12.74 g, 65 mmol) and trimethylsilyl cyanide (6.75 g, 68.25 mmol) in methylene chloride (190 ml). After 5 min, $AlCl_3$ (867 mg, 6.5 mmol) was added, and the whole was stirred for 13 h at room temperature. The solution was washed with H_2O, 5% HCl, H_2O, 5% NaOH, and H_2O. The CH_2Cl_2 solution was dried over Na_2SO_4 and concentrated. The residue was passed through a column of SiO_2 to remove impurities. Recrystallization from benzene gave **3** as colorless needles, mp 175—176 °C, in 95% yield (20.2 g). *Anal.* Calcd for $C_{19}H_{13}N_5O$: C, 69.71; H, 4.00; N, 21.40. Found: C, 69.94; H, 4.01; N, 21.32. MS *m*/*e*: 327 (M^+). IR ν_{max}^{KBr} cm^{-1}: 1680 (C=O). ^{1}H-NMR ($CDCl_3$) δ: 6.3 (1H, s, C^4-H), 7.0—8.1 (12H, m, aromatic H). ^{13}C-NMR ($CDCl_3$) δ: 41.8 (d), 97.7 (s), 115.6 (s), 122.5 (d), 127.4 (d), 128.3 (d), 129.0 (d), 129.2 (d), 131.3 (s), 133.0 (d), 135.2 (d), 138.4 (s), 140.9 (s), 143.5 (d), 169.5 (s).

Alkaline Hydrolysis of 3——An aqueous solution of 10% NaOH (2 ml) was added to a well stirred suspension of **3** (327 mg, 1 mmol) in MeOH (5 ml), and the mixture was stirred for 1 h at room temperature. The reaction mixture was neutralized with AcOH and concentrated under reduced pressure. The residue was extracted with $CHCl_3$. The $CHCl_3$ extract was extracted twice with 5% $NaHCO_3$.

The $CHCl_3$ solution was dried over Na_2SO_4 and concentrated to dryness. The residue was chromatographed on a column of SiO_2 with benzene. The first fraction gave **9** as yellow needles, mp above 300 °C, in 1.8% yield (3.5 mg). The second fraction gave **4** as colorless needles from petr. ether, mp 79—81 °C, in 68.9% yield (135 mg).

The $NaHCO_3$ solution was neutralized with 5% HCl and extracted three times with $CHCl_3$. The $CHCl_3$ extract was dried over Na_2SO_4 and recrystallized from water to give **7**, mp 121—122 °C, in 65.5% yield (80 mg).

Acid Hydrolysis of 3 in DMSO——An aqueous solution of 20% HCl (4 ml) was added to a well stirred solution of **3** (327 mg, 1 mmol) in DMSO (4 ml), and the mixture was stirred for 30 min at room temperature. The reaction mixture was poured into an excess of H_2O–ice mixture and extracted with $CHCl_3$. The $CHCl_3$ solution was dried over Na_2SO_4 and concentrated under reduced pressure to dryness. The residue was recrystallized from benzene–acetone to give **13** as colorless needles, mp 201—203 °C (dec.), in 49.5% yield (157 mg). *Anal.* Calcd for $C_{18}H_{15}N_5O$: C, 68.12; H, 4.76; N, 22.07. Found: C, 68.23; H, 4.72; N, 21.93. MS *m*/*e*: 317 (M^+). IR ν_{max}^{KBr} cm^{-1}: 3410, 3340, 3250 (NH or NH_2), 2250 (C≡N), 1630 (C=O). ^{1}H-NMR (DMSO-d_6) δ: 5.6 (2H, br s, exchangeable with D_2O, NH_2), 6.2 (1H, d, $J_{\alpha,NH}$ = 7 Hz, changeable into s with D_2O, $C^{\alpha}\underline{H}$–NH), 7.1—7.7 (9H, m, aromatic H), 7.7—8.2 (2H, m, aromatic H), 9.4 (1H, d, $J_{\alpha,NH}$ = 7 Hz, exchangeable with D_2O, C^{α}H–N$\underline{H}$). ^{13}C-NMR (DMSO-d_6) δ: 35.6 (d), 96.7 (s), 118.5 (s), 123.4 (d), 126.8 (d), 127.5 (d), 128.2 (d), 129.1 (d), 131.8 (d), 132.9 (s), 138.6 (s), 139.3 (d), 144.3 (s), 166.2 (s).

The aqueous solution was neutralized with 5% NaOH and extracted with $CHCl_3$. The $CHCl_3$ extract was dried over Na_2SO_4 and concentrated under reduced pressure. The residue was recrystallized from benzene–acetone to give **14** as colorless crystals, mp 197—198 °C, in 13.5% yield (45.2 mg). *Anal.* Calcd for $C_{18}H_{17}N_5O_2$: C, 64.46; H, 5.11; N, 20.89. Found: C, 64.31; H, 5.17; N, 20.60. MS *m/e*: 335 (M^+). IR $\nu_{\max}^{\mathrm{KBr}}$ cm^{-1}: 3460, 3425, 3300 (NH or NH_2), 1680, 1630 (C=O). ^{1}H-NMR (acetone-d_6) δ: 4.0—4.4 (2H, br, exchangeable with D_2O, NH_2), 5.6 (1H, d, $J_{\alpha,\mathrm{NH}}=7$ Hz, changeable into s with D_2O, $C^{\alpha}\underline{H}$–NH), 6.5—8.2 (14H, m, 3 of 14H are exchangeable with D_2O, aromatic H and NH). ^{13}C-NMR (DMSO-d_6) δ: 48.6 (d), 100.6 (s), 122.9 (d), 126.3 (d), 127.5 (d), 128.0 (d), 128.9 (d), 131.1 (d), 133.9 (s), 139.0 (s), 139.3 (d), 144.2 (s), 166.1 (s), 172.0 (s).

Acid Hydrolysis of 3 in MeOH——An aqueous solution of 20% HCl (6 ml) was added dropwise to a well stirred suspension of **3** (490.5 mg, 1.5 mmol) in MeOH (10 ml), and the mixture was stirred for 2 h at room temperature. The reaction mixture was poured onto an excess of ice and extracted with $CHCl_3$. The $CHCl_3$ extract was washed with H_2O, dried over Na_2SO_4, and chromatographed on a column of SiO_2 with $CHCl_3$.

The first fraction gave **15** as colorless needles from acetone, mp 186—188 °C, in 59% yield (310 mg). *Anal.* Calcd for $C_{19}H_{18}N_4O_3$: C, 65.13; H, 5.18; N, 15.99. Found: C, 65.02; H, 5.17; N, 15.96. MS *m/e*: 350 (M^+). IR $\nu_{\max}^{\mathrm{KBr}}$ cm^{-1}: 3420, 3340, 3250 (NH or NH_2), 1740, 1640 (C=O). ^{1}H-NMR ($CDCl_3$) δ: 3.85 (3H, s, $COOCH_3$), 4.0—4.9 (2H, br, exchangeable with D_2O, NH_2), 5.6 (1H, d, $J_{\alpha,\mathrm{NH}}=7$ Hz, changeable into s with D_2O, $C^{\alpha}\underline{H}$–NH), 7.1—8.0 (12H, m, one of 12H is exchangeable with D_2O, aromatic H and NH). ^{13}C-NMR (DMSO-d_6) δ: 48.3 (d), 51.9 (q), 98.3 (s), 123.1 (d), 126.5 (d), 127.6 (d), 128.0 (d), 129.0 (d), 131.3 (d), 133.6 (s), 139.0 (s), 139.4 (d), 144.8 (s), 166.6 (s), 171.3 (s).

The second fraction gave **13** in 2.7% yield (12.8 mg).

The aqueous solution was neutralized with 5% NaOH and extracted with $CHCl_3$. The $CHCl_3$ extract was dried over Na_2SO_4 and concentrated under reduced pressure. Recrystallization of the residue from benzene–acetone gave **14** in 6.4% yield (32 mg).

5-Amino-α-benzamido-1-phenyl-1*H*-pyrazole-4-acetamide (14) (Radziszewski Reaction of 13)——A solution of 10% H_2O_2 (1 ml) was added to a well stirred solution of **13** (158.7 mg, 0.5 mmol) and 5% Na_2CO_3 (0.55 ml) in acetone (5 ml), and the whole was stirred for 16 h at room temperature. The reaction mixture was neutralized with AcOH, concentrated under reduced pressure, and extracted with $CHCl_3$. The $CHCl_3$ extract was dried over Na_2SO_4 and chromatographed on a column of SiO_2 with $CHCl_3$. The $CHCl_3$ eluate gave **14** in 34% yield (57 mg).

Reaction of 3 with NaH in DMF——Fifty milligrams of NaH was added in small portions to a soluion of **3** (654 mg, 2 mmol) in DMF (4 g) under ice cooling, and the mixture was stirred for 30 min under ice cooling. The reaction mixture was poured onto an excess of ice, neutralized with AcOH, and extracted with ethyl acetate. The insoluble crystals were collected by suction and recrystallized from MeOH to give **9**.

The ethyl acetate extract was washed with H_2O, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was chromatographed on a column of SiO_2 eluting with benzene, followed by benzene–$CHCl_3$. The first, second, third, and fourth fractions eluted with benzene gave *O*-benzoylmandelonitrile (**22**), 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile (**20**), **9**, and α,1-diphenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-ylmethyl benzoate (**21**), respectively. The eluate with benzene–$CHCl_3$ gave 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (**4**).

Yields and melting points of **4**, **9**, **20**, **21**, and **22** are as follows.

4[18]: mp 78—79 °C, colorless needles from petr. ether, yield 7.6% (30 mg).

9[9]: mp above 300 °C, yellow needles from MeOH, yield 11.7% (45.5 mg).

20[14]: mp 190—191 °C, slightly yellow needles from MeOH, yield 3.2% (14 mg).

21: mp 128—129 °C, colorless prisms from MeOH, yield 6.3% (51 mg). *Anal.* Calcd for $C_{25}H_{18}N_4O_2$: C, 73.87; H, 4.46; N, 13.99. Found: C, 73.66; H, 4.51; N, 13.77. MS *m/e*: 406 (M^+). IR $\nu_{\max}^{\mathrm{KBr}}$ cm^{-1}: 1725 (C=O). ^{1}H-NMR ($CDCl_3$) δ: 7.1—7.6 (12H, m, aromatic H and C^{α}-H), 8.0—8.3 (4H, m, aromatic H), 8.3 (1H, s, C^3-H), 8.95 (1H, s, C^6-H).

22[15]: mp 57—58 °C, colorless prisms from petr. ether, yield 4.9% (23 mg).

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