Studies on Pyrazolo[3,4-*d*]pyrimidine Derivatives. XVII.[1] Reactions of 5-Benzoyl-4,5-dihydro-6-methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile (The 6-Methylpyrazolopyrimidine Reissert Compound)

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Acid hydrolysis of the 6-methylpyrazolopyrimidine Reissert compound (6) gave the ring-opened product (12) and the oxazole (13). Alkaline hydrolysis of 6 afforded the 6-methylpyrazolopyrimidine (7) and benzoic acid (15). The anion (I) of 6 mainly underwent both aromatization and rearrangement, resulting in the formation of the 6-methylpyrazolopyrimidinecarbonitrile (20) and the 4-benzoyl-6-methylpyrazolopyrimidine (21) together with by-products such as 7, the dimer (22), the benzoate (23a), and *O*-benzoylbenzoin (24a). The anion I added to the carbonyl carbon of aromatic aldehydes (8a—c), giving the benzoates (23a—c) together with 7, the *O*-benzoylaroins (24a—c), and the *O*-benzoylcyanohydrins (27a, c).

Keywords methylpyrazolopyrimidine; Reissert compound; hydrolysis; rearrangement; aromatization; electrophilic substitution

Recently, it has been reported that the behavior of the quinazoline Reissert compound **1**[2a] on treatment with hydrochloric acid and sodium hydride was different from that of the isoquinoline Reissert compound **2**, while the 2-methylquinazoline Reissert compound **3**, in which a methyl substituent is located at the 2-position, showed similar behavior to **2** rather than **1**, as shown in Chart 1.

Moreover, we reported a one-step preparation of the 6-methylpyrazolopyrimidine Reissert compound **6** by application of Ruchirawat's method to 6-methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (**7**).[1,3] In order to establish the generality of the effect of the methyl substituent as observed in **3**,[2b] we examined the following reactions of **6**; (a) acid hydrolysis, (b) alkaline hydrolysis, (c) self-decomposition of the anion (I) of **6**, and (d) reaction with an aromatic aldehyde (**8**). In the present paper, we describe the results in detail, in comparison with those for the pyrazolopyrimidine Reissert compound (**9**).[2c,d]

Acid Hydrolysis It was reported by us that reaction of the pyrazolopyrimidine Reissert compound **9** with aqueous hydrochloric acid in dimethyl sulfoxide (DMSO) resulted in the formation of the ring-opened product, the aminopyrazoleacetonitrile (**10**), as a main product together with the aminopyrazoleacetamide (**11**).[2c]

In the case of **6** under the same conditions, the two types of reactions, the ring opening to the pyrimidine and the ring closure to the oxazole, competed with each other, giving α-benzamido-5-acetamido-1-phenyl-1*H*-pyrazole-4-acetonitrile (**12**) and 4-(5-acetamido-1-phenyl-1*H*-pyrazol-4-yl)-5-amino-2-phenyloxazole (**13**). Thus, compound **12**, corresponding to **10** as observed in the acid hydrolysis of **9**,[2c] may be formed through hydrolytic ring opening of **6**.

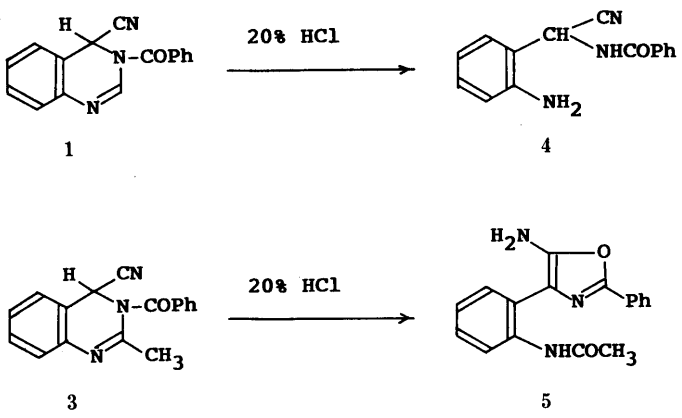


Chart 1

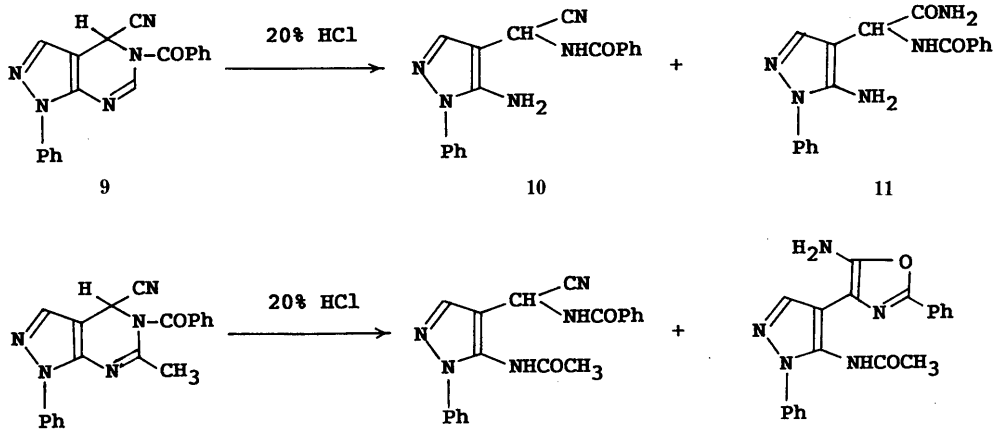


Chart 2

On the other hand, the oxazole **13** may be formed by ring closure to oxazole and hydrolytic ring opening to the pyrimidine. This is similar to a mechanism[2b] reported for oxazole ring closure (**5**) from the acid hydrolysis of the 2-methylquinazoline Reissert compound **3**.

The structural determinations of **12** and **13** were accomplished on the basis of analytical and spectral data as described in the experimental section, as well as the preparation of an authentic specimen of **12** by acetylation of **10** with acetic anhydride.

Alkaline Hydrolysis We reported that the pyrazolopyrimidine Reissert compound **9** was hydrolyzed with aqueous sodium hydroxide in methanol to give the pyrazolopyrimidine (**14**) and benzoic acid (**15**) as main products.[2c]

The 6-methylpyrazolopyrimidine Reissert compound **6** underwent similar hydrolysis under the same conditions, resulting in the formation of the 6-methylpyrazolopyrimidine **7** and benzoic acid **15**. The hydrolysis may proceed through attack of a hydroxide ion at the carbonyl carbon, followed by the ready loss of a cyanide ion, yielding **7** and **15**, as shown in Chart 3.

Self-decomposition of the Anion (I) of 6 Recently, we reported[2a,b] that the self-decomposition of the anion of Reissert compounds proceeded in two ways; one is aromatization, leading to the corresponding heteroarenecarbonitriles, and the other is rearrangement, affording the corresponding aroyl or acylheteroarenes. For example, the anion (III) of the pyrazolopyrimidine Reissert compound **9** underwent the same aromatization as observed for the anion (V) of the quinazoline Reissert compound **1**,[2a] giving the pyrazolopyrimidinecarbonitrile (**16**) as a direct reaction product.[2c] On the other hand, the anion (IV) of the isoquinoline Reissert compound **2** resulted in rearrangement, giving 1-benzoylisoquinoline (**17**).[4] Moreover, both aromatization and rearrangement occurred competitively in the self-decomposition of the anion (VI) of the 2-

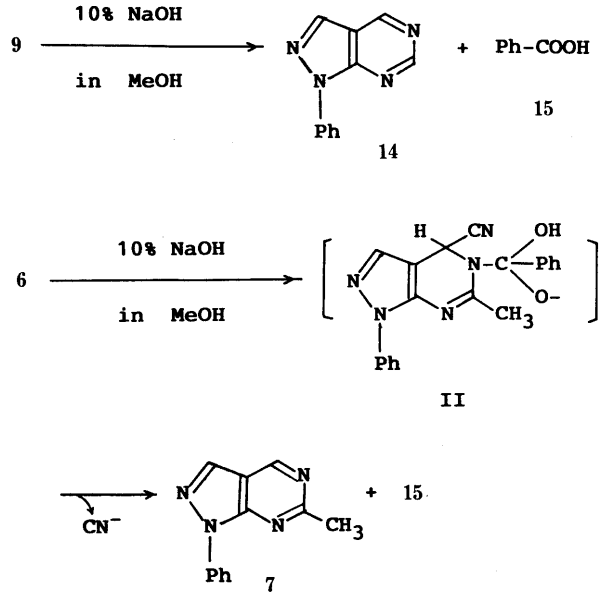


Chart 3

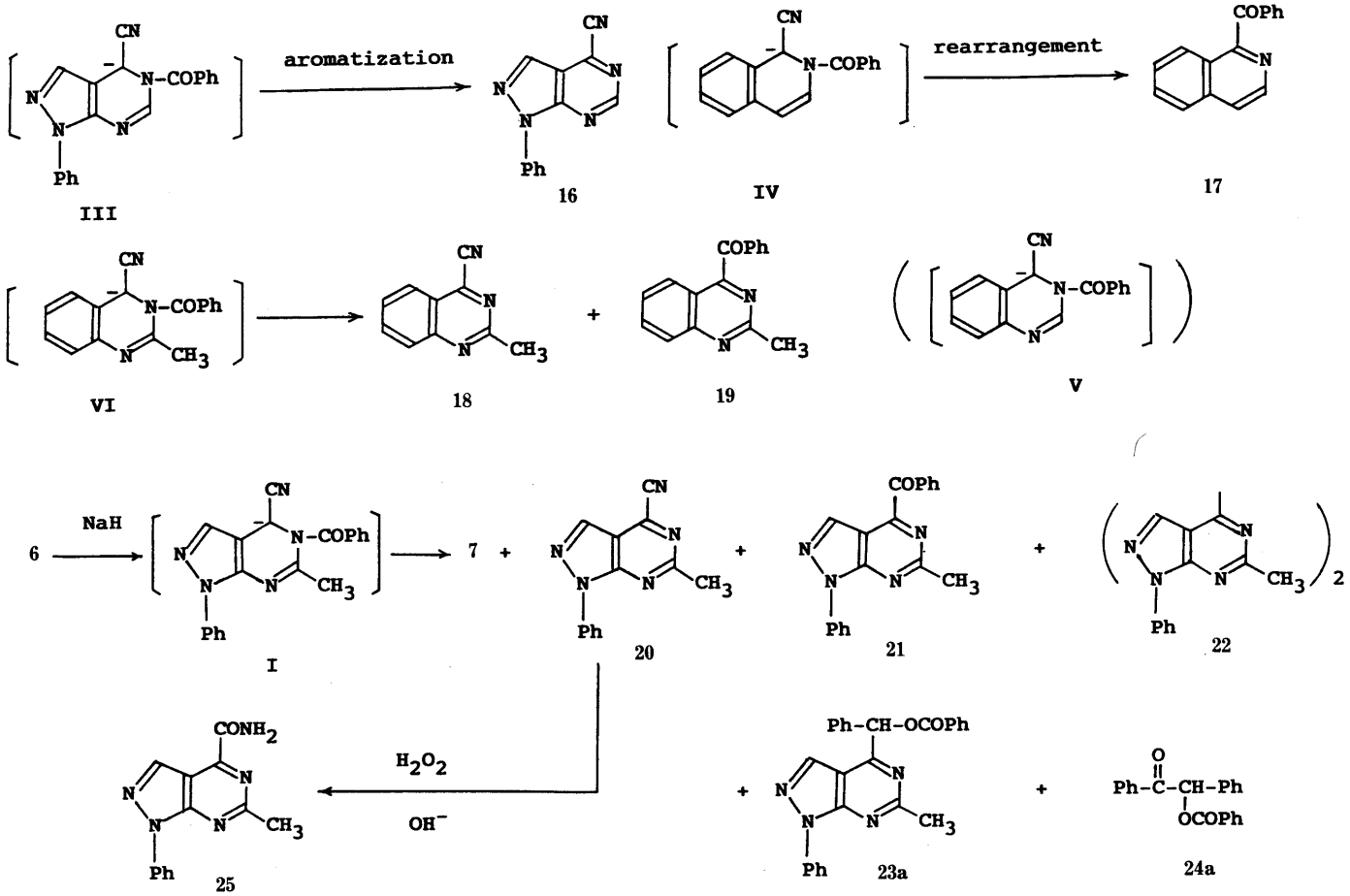
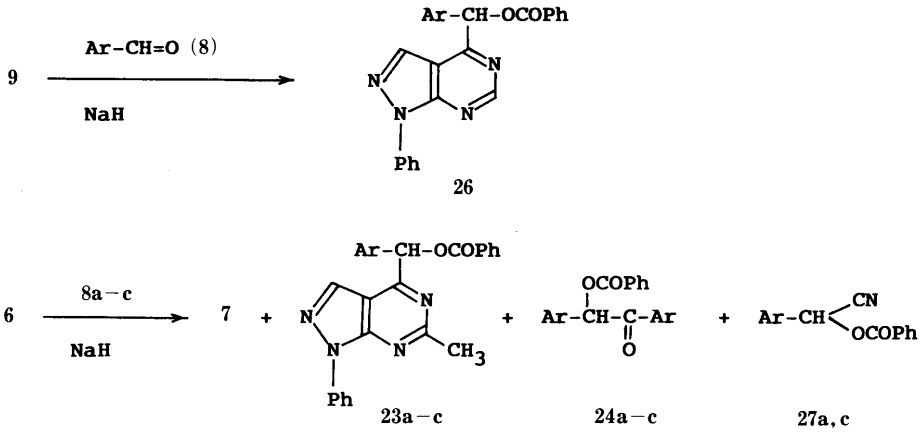


Chart 4



a : Ar=C_6H_5, b : Ar=CH_3-C_6H_4 (*p*), c : Ar=Cl-C_6H_4 (*p*)

Chart 5

methylquinazoline Reissert compound **3**, and 2-methyl-4-quinazolinecarbonitrile (**18**) and 4-benzoyl-2-methylquinazoline (**19**) were obtained.[2b]

When an equimolar mixture of the 6-methylpyrazolopyrimidine Reissert compound **6** and sodium hydride in dimethylformamide (DMF) was stirred for 30 min at 0 °C, the 6-methylpyrazolopyrimidinecarbonitrile (**20**) and the 4-benzoyl-6-methylpyrazolopyrimidine (**21**) were obtained together with the 6-methylpyrazolopyrimidine **7**, the 4,4′-bis[6-methylpyrazolopyrimidine] (**22**), the benzoate (**23a**), and *O*-benzoylbenzoin (**24a**).[5] Moreover, use of refluxing xylene instead of DMF in the above reaction resulted in the formation of similar products, **20**, **21**, **7**, **23a**, and **24a**. The nitrile **20** was easily convertible to the corresponding amide (**25**) by Radziszewski reaction[6] using aqueous hydrogen peroxide and aqueous sodium carbonate. The structures of **20**, **21**, **22**, **23a**, and **25** were suggested by the elemental analyses and mass spectra (MS), and confirmed by the infrared (IR) absorption and proton nuclear magnetic resonance (^{1}H-NMR) spectral data as described in the experimental section, as well as by the above derivation to the amide **25**.

The aromatization and the rearrangement may proceed through a mechanism similar to that proposed by us[2b] for the self-decomposition of the 2-methylquinazoline Reissert compound **3** with sodium hydride. The mechanisms for the formation of by-products, **7**, **22**, **23a**, and **24a** are assumed to be similar to the proposed mechanisms[2a] for the by-products from the quinazoline Reissert compound **1**.

Reaction with Aromatic Aldehydes (8) Recently, we have reported that the introduction of functionalized carbons at the 4-position on the 1*H*-pyrazolo[3,4-*d*]pyrimidine ring was achieved by the nucleophilic addition of the anion (III) of the pyrazolopyrimidine Reissert compound **9**, to the carbonyl carbon of aromatic aldehydes (**8**), giving the corresponding benzoates (**26**).[2d]

When the 6-methylpyrazolopyrimidine Reissert compound **6** reacted with aromatic aldehydes (**8a—c**) in the presence of sodium hydride in DMF, the benzoates **23a—c** were obtained, although the yields were not good, together with the self-decomposition products of the anion I such as the 6-methylpyrazolopyrimidine **7**, *O*-benzoylaroins (**24a**, **b**,[7] **c**[7]), and *O*-benzoylcyanohydrins (**27a**,[8] **c**[9]).

The experimental results may be summarized as follows. i) Acid hydrolysis of the 2-methylpyrazolopyrimidine Reissert compound **6** gave the pyrazoleacetonitrile **12** and the oxazole **13**, as in the case of the 2-methylquinazoline Reissert compound **3**.[2b] ii) In the self-decomposition of the anion I, the anion underwent both aromatization and rearrangement, resulting in the formation of the 6-methylpyrazolopyrimidinecarbonitrile **20** and the 4-benzoyl-6-methylpyrazolopyrimidine **21**. iii) In the reaction with aromatic aldehydes **8**, the anion I added to the carbonyl carbon of **8**, resulting in the formation of the corresponding benzoates **23** in the same way as in the reaction with the quinoline Reissert compound.[10]

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 FT-NMR spectrometer. Chemical shifts are quoted in parts per million (ppm) with tetramethylsilane as an internal standard, and coupling constants (*J*) are given in hertz (Hz). The following abbreviations are used: s=singlet, d=doublet, *t*=triplet, q=quartet, m=multiplet, and br s=broad singlet. MS were recorded on a JEOL JMS D-100 mass spectrometer. Exact mass measurement was carried out on a JEOL JMS-01SG-2 mass spectrometer combined with a JEC-6 spectrum computer. Samples were vaporized in a direct inlet system. Column chromatography was carried out on SiO_2, Wakogel C-200 (200 mesh). High-performance liquid chromatography (HPLC) was carried out on a Hitachi 655A-11 liquid chromatograph equipped with a Hitachi 655A variable-wavelength monitor. A column (20 mm×250 mm) of CHEMCOSORB 7-ODS-H was used for reversed-phase chromatography.

Acid Hydrolysis of 6 A mixture of **6** (1 mmol, 341 mg) and 20% HCl (4 ml) in DMSO (4 ml) was stirred for 30 min. The reaction mixture was poured onto a large amount of ice, and extracted with EtOAc. The extract was washed with H_2O, dried over Na_2SO_4, and concentrated to dryness. The residue was chromatographed on a column of SiO_2. The fraction eluted with $CHCl_3$ gave yellow crystals (229 mg). The yellow crystals were subjected to HPLC with 80% MeOH. The first and second fractions gave 5-acetamido-α-benzamido-1-phenyl-1*H*-pyrazole-4-acetonitrile (**12**) in 30% yield (109 mg) and 4-(5-acetamido-1-phenyl-1*H*-pyrazol-4-yl)-5-amino-2-phenyloxazole (**13**) in 23% yield (81 mg), respectively.

Compound **12** was recrystallized from ether–petroleum benzin–acetone to give colorless needles, mp 158—160 °C. *Anal.* Calcd for $C_{20}H_{17}N_5O_2$: C, 66.84; H, 4.77; N, 19.49. Found: C, 66.67; H, 4.76; N, 19.50. MS *m/z*: 359 (M^+). IR ν_{max}^{KBr} cm^{-1}: 3260 (NH), 2250 (CN), 1660 (CO). ^{1}H-NMR ($CDCl_3$): 8.87 (1H, br s, NHAc, exchangeable with D_2O), 8.29 (1H, d, *J*=8.0 Hz, CH-NH-COPh, exchangeable with D_2O), 7.00—7.90 (11H, m, aromatic H and C^3-H), 6.05 (1H, d, *J*=8.0 Hz, CH-NHCOPh, changed

into s with D_2O), 2.07 (3H, s, $COCH_3$).

Compound **13** was purified by recrystallization from benzene–MeOH, to give colorless needles, mp 206—207 °C. *Anal.* Calcd for $C_{20}H_{17}N_5O_2$: C, 66.84; H, 4.77; N, 19.49. Found: C, 66.85; H, 4.77; N, 19.24. MS *m/z*: 359 (M^+). IR ν_{max}^{KBr} cm^{-1}: 3460, 3360, 3192 (NH), 1662 (CO). ^{1}H-NMR ($(CD_3)_2SO$): 10.37 (1H, br s, NHAc, exchangeable with D_2O), 7.80—8.15 (2H, m, aromatic H), 7.10—7.80 (9H, m, aromatic H and C^3-H), 5.90 (2H, br s, NH_2, exchangeable with D_2O), 2.18 (3H, s, $COCH_3$). ^{13}C-NMR ($(CD_3)_2SO$): 22.6 (q), 93.7 (s), 123.0 (d), 125.6 (d), 126.6 (d), 126.8 (s), 128.2 (s), 129.1 (d), 129.3 (d), 130.6 (d), 135.3 (s), 137.3 (d), 138.7 (s), 144.3 (s), 157.0 (s), 170.6 (s).

Synthesis of 12 A mixture of 5-amino-α-benzamido-1-phenyl-1*H*-pyrazole-4-acetonitrile (**10**,[2c] 0.5 mmol, 159 mg) and Ac_2O (200 mg) was heated at 120 °C for 20 min. After cooling, the reaction mixture was poured onto a large amount of ice, neutralized with 5% Na_2CO_3, and extracted with $CHCl_3$. The extract was washed with H_2O, dried over Na_2SO_4, and concentrated to dryness. The residue was passed through a column of SiO_2 with benzene, to remove impurities. Compound **12**, mp 158—160 °C from petroleum benzin–acetone, was obtained in 24% yield (43 mg).

Alkaline Hydrolysis of 6 A mixture of **6** (1 mmol, 341 mg) and 10% NaOH (2 ml) in MeOH (8 ml) was stirred for 1 h at room temperature (the mixture changed into a yellowish uniform solution). The reaction mixture was neutralized with AcOH, the solvent was removed under reduced pressure, and the residue was extracted with $CHCl_3$. The extract was washed with 5% Na_2CO_3 then H_2O, dried over Na_2SO_4, and concentrated to dryness. The residue was chromatographed on a column of SiO_2. The fraction eluted with $CHCl_3$ gave 6-methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (**7**),[1] mp 99—100 °C from petroleum benzin, in 81% yield (171 mg).

The Na_2CO_3 layer was neutralized with 5% HCl and extracted with $CHCl_3$. The extract was dried over Na_2SO_4 and the $CHCl_3$ was removed under reduced pressure, to give benzoic acid (**15**) as colorless leaflets from petroleum benzin, mp 121—122 °C, in 77% yield (94 mg).

Self-decomposition of the Anion (I) of 6 i) Reaction in DMF: NaH (1 mmol, 24 mg) was added to a stirred solution of **6** (1 mmol, 341 mg) in DMF (3 ml) under ice cooling, and the whole was stirred for a further 30 min. The reaction mixture was poured onto a large amount of ice, neutralized with AcOH, and extracted with AcOEt. The extract was washed with H_2O, dried over Na_2SO_4, and concentrated to dryness. The residue was chromatographed on a column of SiO_2. The first, second, third, fourth, and fifth fractions eluted with benzene gave *O*-benzoylbenzoin (**24a**),[5] 6-methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile (**20**), 4-benzoyl-6-methyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (**21**), 4,4′-bis-[6-methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine] (**22**), and α,1-diphenyl-6-methyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-ylmethyl benzoate (**23a**) in 8% (8 mg), 6% (15 mg), 3% (10 mg), 1% (3 mg), and 7% yields (31 mg), respectively. The fraction subsequently eluted with $CHCl_3$ gave **7** in 41% yield (86 mg).

Compound **20**: mp 138—140 °C, slightly yellow needles from MeOH. *Anal.* Calcd for $C_{13}H_9N_5$: C, 66.37; H, 3.86; N, 29.77. Found: C, 66.13; H, 3.94; N, 29.47. The IR spectrum did not show any absorption due to the cyano group. This result is compatible with the reported absence of the absorption peak of a cyano group located at an electron-deficient carbon, such as in 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carbonitrile (**16**).[11] ^{1}H-NMR ($CDCl_3$): 8.27 (1H, s, C^3-H), 8.00—8.25 (2H, m, aromatic H), 7.20—7.70 (3H, m, aromatic H), 2.88 (3H, s, CH_3).

Compound **21**: mp 120—122 °C, slightly yellow needles from MeOH. *Anal.* Calcd for $C_{19}H_{14}N_4O$: C, 72.60; H, 4.49; N, 17.83. Found: C, 72.28; H, 4.58; N, 17.83. MS *m/z*: 314 (M^+). IR ν_{max}^{KBr} cm^{-1}: 1658 (CO).

Compound **22**: mp 256—258 °C, yellow needles from MeOH. MS *m/z*: 418 (M^+). MS *m/z* Calcd for $C_{24}H_{18}N_8$: 418.1654. Found: 418.1675. ^{1}H-NMR ($CDCl_3$): 9.00 (1H, s, C^3-H), 7.90—8.40 (2H, m, aromatic H), 7.00—7.80 (3H, m, aromatic H), 3.00 (3H, s, CH_3).

Compound **23a**: mp 115—116 °C, colorless needles from MeOH. *Anal.* Calcd for $C_{26}H_{20}N_4O_2$: C, 74.27; H, 4.79; N, 13.33. Found: C, 74.01; H, 4.73; N, 13.27. MS *m/z*: 420 (M^+). IR ν_{max}^{KBr} cm^{-1}: 1720 (CO). ^{1}H-NMR ($CDCl_3$): 8.30 (1H, s, C^3-H), 7.95—8.40 (4H, m, aromatic H), 7.10—7.70 (12H, m, aromatic H and C^{α}-H), 2.82 (3H, s, CH_3).

ii) Reaction in Xylene: A suspension of **6** (1 mmol, 341 mg) and NaH (1 mmol, 24 mg) in xylene was refluxed for 2 h under stirring. After cooling, the reaction mixture was poured onto a large amount of ice, neutralized with AcOH, and extracted with benzene. The same work-up of the extract gave **24a**, **20**, **21**, **23a**, and **7** in 8% (8 mg), 24% (57 mg), 10% (30 mg), 10% (42 mg), and 36% yields (76 mg), respectively.

6-Methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine-4-carboxamide (25) Acetone (4 ml) was added to a mixture of **20** (0.5 mmol, 117 mg) in 10% Na_2CO_3 until a uniform solution was obtained. Then 10% H_2O_2 (1 ml) was added dropwise to the uniform solution, and the whole was stirred for 9 h at room temperature. The reaction mixture was neutralized with AcOH, and concentrated under reduced pressure. The residue was taken up in H_2O, and extracted with $CHCl_3$. The organic layer was dried over Na_2SO_4, and concentrated to dryness. The residue was dissolved in $CHCl_3$ and passed through a column of SiO_2 to remove impurities. Recrystallization from MeOH gave **25**, mp 225—226 °C, in 82% yield (103 mg). *Anal.* Calcd for $C_{13}H_{11}N_5O$: C, 61.65; H, 4.38; N, 27.66. Found: C, 61.82; H, 4.44; N, 27.84. MS *m/z*: 253 (M^+). IR ν_{max}^{KBr} cm^{-1}: 3450 (NH), 1710 (CO).

Reaction of 6 with Aromatic Aldehydes (8) NaH (1 mmol, 24 mg) was slowly added to a stirred solution of **6** (1 mmol, 341 mg) and an aromatic aldehyde (**8a**—**c**, 1 mmol) in DMF (3 ml) under ice cooling, and the mixture was then stirred for 30 min. The reaction mixture was poured onto an excess of ice, neutralized with AcOH, and extracted with AcOEt. The extract was washed with H_2O, dried over Na_2SO_4, and concentrated to dryness. The residue was chromatographed on a column of SiO_2. The first, second, and third fractions eluted with benzene gave the corresponding *O*-benzoylcyanohydrins (**27a**, **c**), *O*-benzoylaroins (**24a**—**c**), and α-aryl-6-methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-ylmethyl benzoates (**23a**—**c**), respectively. The fraction subsequently eluted with $CHCl_3$ gave the 6-methylpyrazolopyrimidine **7**.

From the reaction with benzaldehyde (**8a**), *O*-benzoylmandelonitrile (**27a**),[8] *O*-benzoylbenzoin (**24a**),[5] α,1-diphenyl-6-methyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-ylmethyl benzoate (**23a**), and **7**[1] were obtained in 33% (79 mg), 41% (65 mg), 7% (30 mg), and 61% yields (127 mg), respectively.

From the reaction with *p*-tolualdehyde (**8b**), *O*-benzoyltoluoin (**24b**),[7] 6-methyl-1-phenyl-α-(*p*-tolyl)-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-ylmethyl benzoate (**23b**), and **7** were obtained in 14% (24 mg), 6% (27 mg), and 51% yields (107 mg), respectively. The benzoate **23b**: mp 137—138 °C, colorless needles from MeOH. *Anal.* Calcd for $C_{27}H_{22}N_4O_2$: C, 74.63; H, 5.12; N, 12.90. Found: C, 74.40; H, 5.20; N, 12.71. IR ν_{max}^{KBr} cm^{-1}: 1715 (CO). ^{1}H-NMR ($CDCl_3$): 8.30 (1H, s, C^3-H), 7.85—8.40 (4H, m, aromatic H), 6.85—7.80 (11H, m, aromatic H and C^{α}-H), 2.83 (3H, s, CH_3), 2.38 (3H, s, CH_3).

From the reaction with *p*-chlorobenzaldehyde (**8c**), *O*-benzoyl-*p*-chloromandelonitrile (**27c**),[9] *O*-benzoyl-4,4′-dichlorobenzoin (**24c**),[7] α-(*p*-chlorophenyl)-6-methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4-ylmethyl benzoate (**23c**), and **7** were obtained in 18% (50 mg), 12% (23 mg), 9% (39 mg), and 49% yields (102 mg), respectively. The benzoate **23c**: mp 154—156 °C, colorless needles from MeOH. MS *m/z* Calcd for $C_{26}H_{19}ClN_4O_2$: 454.2121. Found: 454.2119. IR ν_{max}^{KBr} cm^{-1}: 1720 (CO). ^{1}H-NMR ($CDCl_3$): 8.20 (1H, s, C^3-H), 8.00—8.40 (4H, s, aromatic H), 7.15—7.80 (11H, m, aromatic H and C^{α}-H), 2.83 (3H, s, CH_3).

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