

pressure of about 20 mm the first day of treatment, but tachyphylaxis occurred on subsequent days. The oral  $LD_{50}$  in mice, calculated by the method of Litchfield and Wilcoxon,<sup>8</sup> was 380 mg/kg. Although preliminary, these data seem to lead to conclusion that trifluoromethyl substitution does not increase the hypotensive activity, whereas it induces a certain enhancement of toxicity.

#### Experimental Section<sup>9</sup>

**4-Trifluoromethylphthalic Anhydride (I).**—A solution of 7.9 g of 4-trifluoromethylphthalic acid<sup>4,5,10</sup> in 40 ml of  $Ac_2O$  was refluxed for 2 hr. Excess solvent was removed and the residue was distilled at 80–85° (0.4 mm) to give 7 g (95%) of I which solidified on standing, mp 62–65°. The purity of the product was checked by glpc (F and M Model 5750 apparatus equipped with a flame ionization detector and Moseley recorder Model 7172A; F and M stainless steel column 1.8 m  $\times$  2 mm i.d., filled with silicone rubber UCW 98 on Diatoport S 80–100 mesh (10:100), column temperature 120°, injector temperature 160°, detector temperature 160°,  $N_2$  flow 35 cc/min), retention time 2 min 40 sec; ir, 1880 and 1790 ( $C=O$ ), 1255 ( $C-O$ ), 1330 and 1180 ( $CF_3$ ), 885  $cm^{-1}$  ( $CH$  arom).

**6-Trifluoromethyl-2,3-dihydro-1,4-phthalazinedione (II).**—A solution of 3.22 g (0.064 mole) of 98% hydrazine hydrate in 64 ml of  $AcOH$  was added slowly with cooling to a solution of 13.9 g (0.064 mole) of I in 540 ml of  $AcOH$ . The mixture was refluxed for 2 hr. After cooling the precipitate was filtered, washed with  $Et_2O$ , and crystallized from  $MeOH$  to afford 12.6 g (85%) of II: mp 298–300° dec; ir, 3300–2100 ( $N-H$  and  $O-H$ ), 1670 ( $C=O$ ), 1590 ( $C=N$ ), 1315 and 1140 ( $CF_3$ ), 870 and 820  $cm^{-1}$  ( $CH$  arom); nmr ( $DMSO-d_6$ ),  $\tau$  1.8–1.5 (multiplet 3 H,  $H$  arom), –1.6 to –2.1 (broad singlet, 2 H,  $OH$  and  $NH$ ). *Anal.* ( $C_9H_5F_3N_2O_2$ ) C, H, F, N; equiv wt: calcd, 230.14; found, 225 ( $pK_{MCS}$  = 6.8).<sup>11</sup>

**6-Trifluoromethyl-1,4-dichlorophthalazine (III).**—An intimate mixture of II (6.05 g, 0.026 mole) and 30 g of  $PCl_5$  was placed in a glass liner of a bomb tube and heated at 170–175° (oil bath temperature) for 6 hr. After cooling the reaction mixture was added to 400 g of ice-water and neutralized with concentrated  $NH_4OH$ . The solid was filtered, washed with  $H_2O$ , dried *in vacuo*, and crystallized from  $(i-Pr)_2O$  to give 5.9 g (84%) of III, mp 129–130°. The product can be sublimed at 70° (0.2 mm). Ir spectra were as expected. *Anal.* ( $C_9H_3Cl_2F_3N_2$ ) C, H, Cl, F, N.

**6-Trifluoromethyl-1(4)-chloro-4(1)-methoxyphthalazine (IV).**—A solution of 14.5 g (0.054 mole) of III in 290 ml of  $MeOH$  was added with stirring to a solution of 1.29 g (0.056 g-atom) of Na in 100 ml of  $MeOH$  at room temperature. After boiling at reflux for 45 min the solvent was evaporated *in vacuo* to dryness and the residue was extracted with  $(i-Pr)_2O$ . The extract was filtered to remove insoluble material, treated with charcoal, and concentrated to crystallization, yield 11.3 g (79%), mp 133°. An analytical specimen was recrystallized from  $(i-Pr)_2O$ , mp 135°. Ir and nmr spectra were as expected. *Anal.* ( $C_{10}H_6ClF_3N_2O$ ) C, H, Cl, F, N.

**6-Trifluoromethyl-1,4-dihydrazinophthalazine Dihydrochloride (V).**—To 15 ml of 98% hydrazine hydrate and 15 ml of absolute  $EtOH$  heated at 40°, 23 g (0.088 mole) of IV was added, and the mixture was boiled at reflux for 2 hr. When the temperature rose to 85° solution occurred. After cooling, a red precipitate was filtered, washed with a little absolute  $EtOH$ , and dried *in vacuo* at 50° yielding 8.9 g (39%), mp 152–156°. Recrystallization of a portion of this material from absolute  $EtOH$  furnished

an analytical sample of the base, mp 155–156°, ir and nmr spectra as expected. *Anal.* ( $C_9H_5F_3N_6$ ) C, H, N; F: calcd, 22.09; found, 22.76.

The dihydrochloride (V) was prepared by adding  $Et_2O-HCl$  to a solution of the base in the minimum amount of warm absolute  $EtOH$ . The precipitate was filtered and recrystallized from 95%  $EtOH-Et_2O$ , yield 5.94 g (54%), mp 212° dec, tlc (on silica gel G buffered at pH 2.2 with McIlvaine reagent, developed with  $EtOH-H_2O$  65:35, and visualized by spraying with an aqueous solution of 0.1 N J and then with concentrated  $H_2SO_4$ , or with an ammoniacal  $AgNO_3$  solution)  $R_f$  0.55, ir and nmr spectra as expected. *Anal.* ( $C_9H_5F_3N_6 \cdot 2HCl$ ) C, H, F, N,  $Cl^-$ .

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### The Synthesis of the $\alpha$ and $\beta$ Anomers of 1-(2-Deoxy-D-ribofuranosyl)-2-pyridone<sup>1</sup>

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One phase in the design of nucleoside analogs as potential anticancer agents has been directed to the preparation of deaza and deoxy models of essential metabolites.<sup>2</sup> The design of such models for the inhibition of thymidylate synthetase<sup>3</sup> prompted this study. This note describes the synthesis of 1-(2-deoxy- $\beta$ -D-ribofuranosyl)-2-pyridone (**5**, 3-deaza-4-deoxy-2'-deoxyuridine). The ribofuranosyl analog of **5** has been prepared from the  $HgCl$  salt of 2-pyridone and by application of the Hilbert-Johnson reaction<sup>4</sup> of 2-benzoyloxypyridine with the protected ribofuranosyl chloride.<sup>5</sup> A recent report from Wagner and coworkers describes the synthesis of the title compounds from the  $Ag$  salt of 2-pyridone.<sup>5b,c</sup> Similarly, 3-deazauridine has been synthesized by Robins and Currie by the silyl method.<sup>2b</sup>

The synthesis of the intermediate **3** was accomplished in low yield by application of the Hilbert-Johnson reaction of 2-benzoyloxypyridine (**1b**) with 3,5-di(*O-p*-toluyl)-2-deoxy-D-ribofuranosyl chloride (**2**).<sup>6</sup> A higher yield (6%) of the  $\alpha$  and  $\beta$  anomeric mixture (**3a** and **b**) was achieved by use of the  $HgCl$  salt **1a**. After chromatographic separation of the anomers the protected  $\alpha$  anomer (**3a**) crystallized. Transesterification of **3a** followed by silica chromatography gave crystalline 1-(2-deoxy- $\alpha$ -D-ribofuranosyl)-2-pyridone (**4**).

(1) This research was supported by Research Grant CA-5639 and Career Development Award 1K3-CA-10,739 of the National Cancer Institute, National Institutes of Health.

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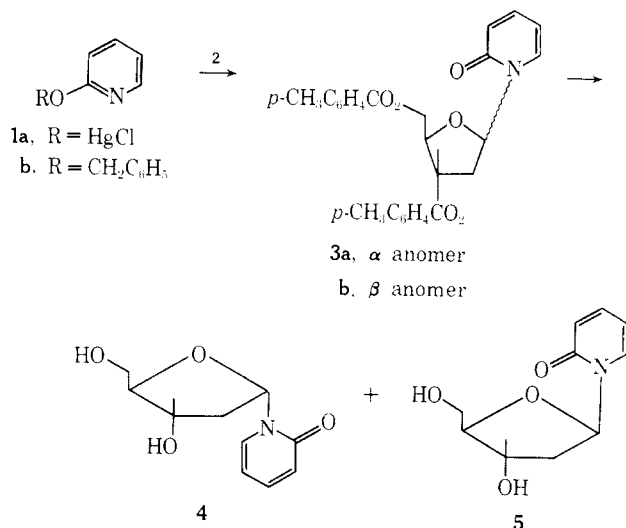
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(9) Where analyses are indicated only by symbols of the elements, analytical results for those elements were within  $\pm 0.4\%$  of the theoretical values. Melting points were determined in capillary tubes and are uncorrected. Ir spectra were determined with a Perkin-Elmer Model 137 spectrophotometer as Nujol mulls. Nmr spectra were recorded at 60 Mcps by a Varian A-60 spectrometer using TMS as internal standard (10.00 ppm) in the solvents indicated. Spectra not mentioned specifically were as expected. F analyses were performed as described by B. Cavalleri, E. Bellasio, and E. Testa, *Gazz. Chim. Ital.*, **96**, 227 (1966).

(10) When prepared according to ref 5, crude 4-trifluoromethylphthalic acid was used directly.

(11) Potentiometric titration in  $MCS-H_2O$  (4:1) solution with 0.1 N  $NaOH$ .

The  $\beta$  anomer of the protected nucleoside (**3b**) was not purified but rather was transesterified and the  $\beta$  nucleoside **5** was purified on silica to give a 2% over-all



yield for the sequence from **1a**. The uv spectra of **4** and **5** are consistent with an N-substituted 2-pyridone.

Equilibration from the furanose to the pyranose isomer was found in 3-(2-deoxy-D-ribofuranosyl)-2,6-dibenzoyloxypyridine<sup>2a</sup> where the pyranosyl isomer was found after acid equilibration; pseudouridine also equilibrated to give the pyranose derivative in base.<sup>7</sup> Similarly, equilibration could not be excluded in the title compounds since Pischel and Wagner reported the pyranosyl and the furanosyl forms in the ribose analog of **4**.<sup>5</sup> This possibility was examined by treatment in acid or base followed by neutralization and periodate oxidation at pH 5.<sup>8</sup> 2-Pyridone did not consume IO<sub>4</sub><sup>-</sup> under any conditions; similarly, **4** and **5** retained the furanosyl structure during base equilibration. Acid equilibration of **4** at 25° for 2 hr did not give any detectable amount of the pyranose; however, after 1 hr at 65° in 0.2 M HCl **4** consumed 2 molar equiv of IO<sub>4</sub><sup>-</sup> demonstrating ring opening of the sugar. Longer treatment of **4** and **5** in acid at higher temperatures gave further degradation to a product that consumed 4 equiv of IO<sub>4</sub><sup>-</sup>. From these results the furanose structure of **4** and **5** is stable in neutral and alkaline solution, or after brief treatment at 25° with dilute acid. Slow degradation in acid was also observed in the uv. Solutions of **4** in 1 M KOH and 1 M HCl at 25° were examined at time intervals. No shift in the maximum of the alkaline solution was observed. However, the maximum in acid broadened after 20 hr and stabilized after several days at 277 m $\mu$  (2-pyridone, 278 m $\mu$ ) a hypsochromic shift of 6 m $\mu$  suggesting cleavage of the sugar residue.

Wagner and coworkers<sup>5c</sup> assigned configurations **3a** and **b** from the nmr spectrum which showed a band of width 13.9 Hz for the anomeric proton of **3a** and 8.8 Hz for the anomeric proton of the  $\beta$  anomer (**3b**). The nmr spectrum of **4** (D<sub>2</sub>O) at 100 Mc exhibited first-order splitting patterns for the sugar protons at carbons 1' and 2'. By selective decoupling experiments and

analysis of the resultant patterns, a *cis* relationship between the proton on C-1' and that on C-3' was observed; this is based on the observation that the splitting constant between *cis* protons is greater than that observed for *trans* protons in ribofuranosyl systems. The appearance of the spectrum and the chemical shifts of the sugar protons of **4** resembled closely that of  $\alpha$ -thymidine reported by Lemieux.<sup>9</sup> The nmr of the  $\beta$  anomer **5** showed a sugar proton pattern much like that of thymidine; however, the anomeric proton overlapped with the pyridone protons at 6.4 ppm and could not be examined for the splitting pattern.

The optical rotatory dispersion curve of **4**, the  $\alpha$  anomer from nmr, shows a well-defined negative Cotton effect. The  $\beta$  anomer (**5**) gave a positive Cotton curve thus following the same pattern found in the pyrimidine nucleosides.<sup>10</sup>

Compounds **4** and **5** failed to inhibit thymidylate synthetase<sup>2a</sup> at a concentration ratio of inhibitor/substrate (2'-deoxyuridine 5'-monophosphate) of 200. They also did not inhibit the growth of *Bacillus cereus*; however, *Bacillus subtilis* and *Staphylococcus aureus* growth was inhibited 25% at  $1 \times 10^{-3}$  M.

### Experimental Section

Melting points were recorded from a calibrated Thomas-Hoover Unimelt unit or a microscope hot stage. Spectra were recorded on Beckman IR10, Beckman DU, Cary 14, Cary 60, Varian A-60-A, and Varian HR100 spectrometers. Microanalyses were run by Midwest Microanalytic Laboratory, Indianapolis, Ind., or on an F & M 185 C, H, N analyzer.

**1-(3,5-Di-O-*p*-toluyl-2-deoxy-D-ribofuranosyl)-2-pyridone (3a,b).**—The HgCl salt of 2-pyridone<sup>5</sup> (3.1 g, 0.01 mole) was dried by refluxing in 100 ml of PhMe using a Dean-Stark water trap. After cooling 3.7 g (0.009 mole) of 3,5-di-O-*p*-toluyl-2-deoxy-D-ribofuranosyl chloride<sup>6</sup> was added and the mixture refluxed for 1 hr. The suspension was filtered, the solid was washed with 50 ml of PhMe, and the filtrate was washed twice with 50 ml of 30% KI; toluic acid formed in the reaction was extracted with a saturated solution of NaHCO<sub>3</sub>. After drying (MgSO<sub>4</sub>) the solvent was evaporated and the anomeric mixture (**3a,b**) was purified by dry column chromatography on silica using 2% MeOH in CHCl<sub>3</sub>. The  $\alpha$  anomer **3a** crystallized from C<sub>6</sub>H<sub>6</sub>-C<sub>6</sub>H<sub>14</sub> to give 124 mg (3%), mp 152–153° (lit.<sup>5b</sup> mp 159°), nmr as expected.<sup>5c</sup> Anal. (C<sub>26</sub>H<sub>25</sub>NO<sub>5</sub>) C, H, N.

The  $\beta$  anomer **3b** was not crystallized from the residue.

**3-(2-Deoxy- $\alpha$ -D-ribofuranosyl)-2-pyridone (4).**—The diester **3a** (4.18 g, 9.2 mmoles) was dissolved in 50 ml of C<sub>6</sub>H<sub>6</sub>, 50 ml of MeOH and 5 ml of  $\sim 1$  M NaOCH<sub>3</sub> in MeOH were added, and the mixture was allowed to stand 2 days at 25°. Neutralization to pH 7 with Dowex 50 (H<sup>+</sup> form) followed by evaporation gave an oily two-phase residue. This was chromatographed on 200 g of silica eluting 25-ml fractions with 10% MeOH in CHCl<sub>3</sub>. The product **4** solidified after evaporation from fractions 41–59 to give 1.03 g (56%); mp 96–97.5° (lit.<sup>5b</sup> 132°); nmr (D<sub>2</sub>O, 100 Hz), H<sub>2</sub> $\alpha$   $\delta$  2.08 ( $J_{H2\beta}$  = 7.0,  $J_{H1\beta}$  = 1.3,  $J_{H3}$  = 1.0 Hz), H<sub>2</sub> $\beta$   $\delta$  2.81 ( $J_{H2\alpha}$  = 7.0,  $J_{H1\beta}$  = 3.6,  $J_{H3}$  = 3.0 Hz), H<sub>1</sub> $\alpha$   $\delta$  6.36 ( $J_{H2\alpha}$  = 3.6,  $J_{H2\beta}$  = 1.3 Hz); the remainder of the assignments were as expected; uv,  $\lambda_{max}$  (0.1 M HCl) 283 m $\mu$  ( $\epsilon$  5460),  $\lambda_{min}$  242 (650);  $\lambda_{max}$  (H<sub>2</sub>O) 298 (5800), 226 (5800),  $\lambda_{min}$  246 (300);  $\lambda_{max}$  (0.1 M KOH) 299 (6500),  $\lambda_{min}$  248 (1700); ORD ( $c$  0.00358, H<sub>2</sub>O, 22°),  $[\Phi]_{490}^{\circ}$  –2180°,  $[\Phi]_{317}^{\circ}$  –11,800°,  $[\Phi]_{273}^{\circ}$  +4060°,  $[\Phi]_{249}^{\circ}$  +1410°,  $[\Phi]_{243}^{\circ}$  +1940°,  $[\Phi]_{233}^{\circ}$  –2530°; CD ( $c$  0.00358, H<sub>2</sub>O)  $[\theta]_{350}^{\circ}$  0,  $[\theta]_{291}^{\circ}$  –10,600,  $[\theta]_{255}^{\circ}$  0,  $[\theta]_{250}^{\circ}$  +1240,  $[\theta]_{248}^{\circ}$  –3000,  $[\theta]_{235}^{\circ}$  +6040,  $[\theta]_{244}^{\circ}$  –9000. Anal. (C<sub>10</sub>H<sub>13</sub>NO<sub>4</sub>) C, H, N.

**3-(2-Deoxy- $\beta$ -D-ribofuranosyl)-2-pyridone (5).**—Impure **3b** was treated as described in the synthesis of **4**. Chromatography on silica gave **5** (2% from **1a**); mp 115–116° (lit.<sup>5b</sup> 115–116°); nmr as expected; uv,  $\lambda_{max}$  (0.1 M HCl) 283 m $\mu$  ( $\epsilon$  5200),  $\lambda_{min}$  242 (1050);  $\lambda_{max}$  (H<sub>2</sub>O) 298 (5750), 226 (5950);  $\lambda_{min}$  247 (690);  $\lambda_{max}$  (1 M KOH) 298 (6200),  $\lambda_{min}$  251 (1500); ORD ( $c$  0.00333, H<sub>2</sub>O,

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22°, [ $\Phi$ ]<sub>400</sub> + 1900°, [ $\Phi$ ]<sub>317</sub> + 4350, [ $\Phi$ ]<sub>279</sub> - 890°, [ $\Phi$ ]<sub>253</sub> + 1580°, [ $\Phi$ ]<sub>242</sub> + 890°, [ $\Phi$ ]<sub>231</sub> + 5450°. *Anal.* (C<sub>10</sub>H<sub>13</sub>NO<sub>4</sub>) C, H, N.

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### Fluorinated Pyrimidines. XXXVI. Synthesis of Some 2,4-Substituted 5-Trifluoromethylpyrimidines<sup>1</sup>

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In this laboratory we have been concerned for some time with 5-trifluoromethyl-2'-deoxyuridine,<sup>4</sup> which has powerful tumor inhibitory<sup>5</sup> and antiviral activities.<sup>6,7</sup> 5-Methyl-2'-deoxycytidine has been found in mammalian and polyoma viral DNA.<sup>8</sup> Even though it is known that the methylation of 2'-deoxycytidine occurs after its incorporation into DNA, we were interested in preparing the corresponding trifluoromethyl derivative and testing it for biological activity. Because of the alkaline instability of the trifluoromethyl group in 5-trifluoromethyluracil<sup>4</sup> and its nucleosides, conventional nucleoside syntheses that require alkaline deblocking were considered unsuitable. Consequently, we attempted to prepare 5-trifluoromethyl-2'-deoxycytidine enzymatically with the *trans*-N-deoxyribosylase<sup>9</sup> from *Lactobacillus helveticus* (ATCC 8018) by an exchange between 5-trifluoromethylcytosine, prepared from 2,4-dichloro-5-trifluoromethylpyrimidine,<sup>10</sup> and a suitable deoxyribonucleoside donor. This enzymatic route has been used successfully in the syntheses of the analogs, 5-trifluoromethyl-2'-deoxyuridine<sup>4</sup> and 5-nitro-2'-deoxyuridine.<sup>11</sup> Although the synthesis of 5-trifluoromethyl-2'-deoxyuridine could be accomplished with our enzyme preparation, we were not able to detect by tlc the presence of 5-trifluoromethyl-2'-deoxycytidine in any attempted enzymatic reaction between 5-trifluoromethylcytosine and either thymidine, 2'-deoxycytidine, 2'-deoxyuridine, 2'-deoxyguanosine, or 2'-deoxyadenosine. Therefore, although cytosine was readily converted to 2'-deoxycytidine with this enzyme, 5-trifluoromethylcytosine apparently had no affinity for the enzyme.

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(2) Holder of a Postdoctoral Fellowship from the Damon Runyon Memorial Fund.

(3) American Cancer Society Professor of Oncology.

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For other reasons, 4-benzylamino-2-hydroxy-5-trifluoromethylpyrimidine (VIII) was synthesized from 2,4-dichloro-5-trifluoromethylpyrimidine (I). Two isomers, 4-benzylamino-2-chloro-5-trifluoromethylpyrimidine (IV) and 2-benzylamino-4-chloro-5-trifluoromethylpyrimidine (V), were obtained which could only be separated by tlc. On treatment with NaOMe the mixture of IV and V gave the corresponding isomeric methoxy compounds, VI and VII, which could be separated by mechanical retrieval of two distinct crystal forms. The structures of the isomers were determined as follows. On hydrolysis of IV and V, as well as VI and VII, with 1 N HCl, two of the compounds were more reactive and should have had the Cl and OCH<sub>3</sub> groups in the 2 position. Furthermore, 2-benzylamino-4-chloro-5-trifluoromethylpyrimidine had a greater bathochromic shift in its spectrum relative to the 2,4-dichloro compound than did its isomer, which is in agreement with analogous studies by Boarland and McOmie.<sup>12</sup>

**Biological Activity.**—Compounds II and III did not inhibit the growth of L5178Y cells in culture<sup>13</sup> at 10<sup>-4</sup> M. However, compounds VI and VII inhibited these cells approximately 50% at 10<sup>-4</sup> M.

### Experimental Section

All melting points are corrected. All analyses are by Galbraith Laboratories, Knoxville, Tenn. All uv spectra were determined on a Cary Model 15 spectrophotometer.

**2,4-Dichloro-5-trifluoromethylpyrimidine (I)** was synthesized as described by Shen, *et al.*<sup>10</sup>

**4-Amino-2-chloro-5-trifluoromethylpyrimidine (II).**—To 1.0 g of I in a precooled steel bomb (Parr) was added 20 ml of liquid NH<sub>3</sub>. The bomb was closed and gradually allowed to come to room temperature and was then recooled. After evaporation of the NH<sub>3</sub>, the residue was extracted with 150 ml of Et<sub>2</sub>O and filtered, and the solvent was evaporated to dryness *in vacuo*. The residue was crystallized from EtOH to give 900 mg (98%) of II, mp 146°,  $\lambda_{\text{max}}^{\text{MeOH}}$  239 and 282 m $\mu$  ( $\epsilon$  21,000 and 2500). *Anal.* (C<sub>5</sub>H<sub>3</sub>F<sub>3</sub>ClN<sub>3</sub>) C, H, N.

**4-Amino-2-hydroxy-5-trifluoromethylpyrimidine (III).**—Compound II (900 mg) was refluxed in 100 ml of 0.1 N HCl until it dissolved. The solution was then evaporated *in vacuo*, and the residue was washed with EtOH and Et<sub>2</sub>O. It was then dissolved in 100 ml of 80% EtOH and 4 g of Amberlite IR-48 (OH-) was added. The mixture was stirred for 15 min until the pH reached 6.0. The resin was filtered and washed with EtOH, and the combined filtrates were evaporated to dryness *in vacuo*. The residue was dissolved in EtOH and reprecipitated with Et<sub>2</sub>O to give 500 mg (61%) of a colorless solid, which was recrystallized from EtOH; mp 250–255° dec,  $\lambda_{\text{max}}^{\text{MeOH}}$  286 m $\mu$  ( $\epsilon$  7700). *Anal.* (C<sub>5</sub>H<sub>3</sub>F<sub>3</sub>N<sub>3</sub>O) C, H, F, N.

**Isomeric Mixture of 4-Benzylamino-2-chloro-5-trifluoromethylpyrimidine (IV) and 2-Benzylamino-4-chloro-5-trifluoromethylpyrimidine (V).**—To a stirred solution of I (810 mg) in 3 ml of EtOH was added 803 mg of benzylamine in 3 ml of EtOH. The mixture was warmed; it decolorized and was kept at 40° for 15 min when a crystalline precipitate formed. The mixture was evaporated to dryness, and the residue was dissolved in 50 ml of Et<sub>2</sub>O. The ether phase was washed with three 50-ml portions of H<sub>2</sub>O, 50 ml of 0.1 N HCl, then 50 ml of H<sub>2</sub>O, and was dried (Na<sub>2</sub>SO<sub>4</sub>). The Et<sub>2</sub>O was evaporated *in vacuo* to give an oil, which solidified on standing. This material was crystallized from petroleum ether (bp 50–70°) to give 995 mg (79%) of a mixture of the two isomers IV and V. After recrystallization, an analytical sample of the mixtures was obtained, mp 101–104°. *Anal.* (C<sub>12</sub>H<sub>9</sub>F<sub>3</sub>ClN<sub>3</sub>) C, H, F.

A few milligrams of the mixture of isomers was separated by tlc (Eastman 6060) in PhH to give IV, mp 106°, and V, mp 110°. The structures of the isomers were assigned as described above.

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