solved in dilute HCl; sodium sulfamate was added to the chlorhydric solution and the precipitated barium sulfate was isolated as usual. From the amount of barium sulfate obtained (0.0243 g) a 2.1% yield in carbon dioxide was calculated.

The filtrate was then extracted by chloroform (3 × 10 ml) and the combined extracts were diluted to 50 ml. Treatment of 20 ml of this solution by ethereal picric acid gave 0.480 g (78%) of pyridinium picrate, mp and mmp 165° after recrystallization from EtOH; the benzaldehyde content of the above solution was determined by quantitative VPC analysis, which indicated a 2.5% yield.

The remaining aqueous layer was then acidified with dilute HCl and extracted with ether (3 × 10 ml). The residue from evaporation of ether was dissolved in water (10 ml) and an aqueous solution of mercuric acetate was added to the resulting solution. The mercuric salt of phenylglyoxylic acid which precipitated was isolated by filtration, washed, and dried; the salt weighed 0.86 g (70%) and had mp 167° (lit. mp[8] 165–166°).

Preparation of Methyl Phenylglyoxylate. A. By Reaction of Methyl α-Bromophenylacetate with 1 Equiv of Pyridine *N*-Oxide. A solution of pyridine *N*-oxide (5.25 g, 55 mmol) and methyl α-bromophenylacetate (11.9 g, 55 mmol) in methylene chloride (10 ml) was refluxed for 2 hr; 100 ml of a 10% HCl solution was added and the resulting mixture was extracted with ether (3 × 50 ml). Evaporation of the dried extract (Na_2SO_4) and distillation of the oily residue afforded 4.2 g (48%) of methyl phenylglyoxylate, bp 66° (0.5 mm), 2,4-DNP mp 172° (lit.[16] mp 171°).

B. By Reaction of Methyl α-Bromophenylacetate with 1.5 Equiv of Pyridine *N*-Oxide. A solution of pyridine *N*-oxide (10.5 g, 111 mmol) and methyl α-bromophenylacetate (16.8 g, 73 mmol) in methylene chloride (20 ml) was refluxed for 2 hr (TLC examination showed that methyl α-bromophenylacetate had been completely transformed); work-up as above afforded 7.86 g (65%) of methyl phenylglyoxylate.

C. By Reaction of Methyl α-Bromophenylacetate with Pyridine *N*-Oxide in the Presence of Silver Nitrate and Subsequent Decomposition by Triethylamine. A solution of methyl α-bromophenylacetate (17 g, 75 mmol) in acetonitrile (15 ml) was added dropwise, under stirring, to an ice-cooled solution of pyridine *N*-oxide (7.4 g, 78 mmol) and silver nitrate (13.3 g, 78 mmol) in acetonitrile (30 ml). Stirring was continued for an additional 2 hr; the precipitated silver bromide was eliminated by filtration and washed with acetonitrile. To the stirred filtrate, triethylamine was slowly added, and the resulting solution was then acidified by 10% HCl. Work-up as in A afforded 10.8 g (87%) of methyl phenylglyoxylate.

Registry No.—**3a**, 56943-39-6; **3b**, 56943-41-0; **3c**, 56943-42-1; **4a**, 56943-43-2; **4b**, 56943-45-4; **4c**, 56943-46-5; **5a**, 56943-47-6; **5b**, 56943-49-8; **5c**, 56943-50-1; **6a**, 56943-51-2; **6b**, 56943-53-4; **6c**, 56943-54-5; **7b**, 57031-39-7; **7c**, 56943-56-7; **8a**, 56943-57-8; **8b**, 56943-59-0; **8c**, 56943-60-3; **9a**, 56943-61-4; **9b**, 56943-63-6; **9c**, 56943-64-7; **10b**, 109-06-8; **11b**, 108-99-6; **12b**, 108-89-4; **13b**, 108-48-5; methyl α-bromoacetate, 96-32-2; *tert*-butyl α-bromoacetate, 5292-43-3; benzyl α-bromoacetate, 5437-45-6; methyl α-bromopropanoate, 5445-17-0; methyl α-bromobenzeneacetate, 3042-81-7; α-bromoacetic acid, 79-08-3; α-bromobenzeneacetic acid, 4870-65-9; pyridine *N*-oxide, 694-59-7; $AgNO_3$, 7761-88-8; picric acid, 88-89-1; sodium hydroxide, 1310-73-2; 2,4-DNP, 119-26-6; triethylamine, 121-44-8; *tert*-butyl glyoxylate 2,4-DNP, 56943-65-8; methyl phenylglyoxylate, 15206-55-0.

References and Notes

(1) (a) A. R. Katritsky and J. M. Lagowski, "Chemistry of the Heterocyclic N-Oxide", Academic Press, New York, N.Y., 1971, p 155; (b) *ibid.*, p 448.
(2) A. R. Katritzky and E. Lunt, *Tetrahedron*, **25**, 4291 (1969).
(3) E. Ochiai, M. Katada, and T. Naito, *J. Pharm. Soc. Jpn.*, **64**, 210 (1944).
(4) W. Feely, W. L. Lehn, and V. Boekelheide, *J. Org. Chem.*, **22**, 1135 (1957).
(5) A. R. Katritzky, *J. Chem. Soc.*, 2404 (1956).
(6) (a) H. N. Bojarska-Dahlig and H. J. den Hertog, *Recl. Trav. Chim. Pays-Bas*, **77**, 331 (1958); (b) H. N. Bojarska-Dahlig, *ibid.*, **78**, 981 (1959).
(7) J. M. Tien, I. M. Hunsberger and A. M. Javellana, Abstracts, 135th National Meeting of the American Chemical Society, Boston, Mass., April 1959, p 76–0.
(8) T. Cohen and I. H. Song, *J. Org. Chem.*, **31**, 3058 (1966).
(9) T. Cohen, I. H. Song, J. H. Fager, and G. L. Deets, *J. Am. Chem. Soc.*, **89**, 4968 (1967).
(10) C. Rüchardt, S. Eichler, and O. Krätz, *Tetrahedron Lett.*, 233 (1965), propose an alternative for the mechanism of this reaction; for a summary discussion see R. A. Abramovitch and E. M. Smith, "Pyridine and Its Derivatives", Vol. 14, Supplement, Part 2, R. A. Abramovitch, Ed., Wiley, New York, N.Y., 1974, p 83.
(11) T. Kato, Y. Goto, and Y. Yamamoto, *Yakugaku Zasshi*, **84**, 287 (1964).
(12) I. M. Hunsberger and J. M. Tien, *Chem. Ind. (London)*, 88 (1959).
(13) L. A. Carpino, *J. Org. Chem.*, **29**, 2840 (1964).
(14) NMR spectra of the isolated bromides **3a–6a**, **8a**, and **9a** were identical with those of the corresponding nitrates that are reported in Table I.
(15) R. L. Shriner, R. C. Fuson, and D. Y. Curtin, "The Systematic Identification of Organic Compounds", 4th ed, Wiley, New York, N.Y., 1956.
(16) C. F. H. Allen, *J. Am. Chem. Soc.*, **52**, 2955 (1930).
(17) J. C. P. Schwarz, and M. Mac Dougall, *J. Chem. Soc.*, 3065 (1956).

Lithiation of Methoxyindoles

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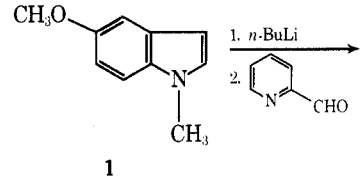
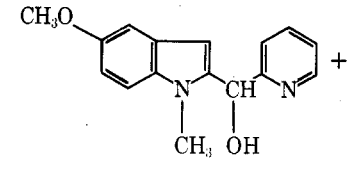
It has been demonstrated that 1-benzenesulfonylindole can be lithiated in the 2 position and that the resulting lithiated intermediate can be used for the synthesis of a variety of 2-substituted indoles.[1,2] This extends earlier studies[3] which had demonstrated selective lithiation of 1-alkylindoles, since the benzenesulfonyl group can be removed readily by hydrolysis.[1] In view of the interest in biologically active methoxyindoles we have now extended lithiation studies to 5- and 6-methoxyindole derivatives.

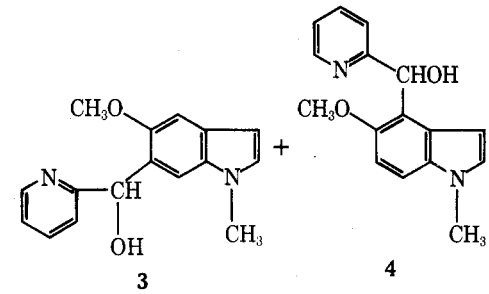
Lithiation of 1-methyl-5-methoxyindole (**1**) by *n*-butyllithium in refluxing ether was nonselective as judged by the formation of three isomeric alcohols after reaction with pyridine-2-carboxaldehyde. Two of the products (**2** and **3**) were obtained as pure crystalline compounds while a third (**4**) was obtained as an oil slightly contaminated with **2**.



1



2



3 4

Product **2** was identified as the 2-substituted product on the basis of a sharp singlet for the indole 3H proton in the NMR. The other major product was assigned structure **3** since the aromatic region reveals two prominent singlets at δ 6.96 and 7.22, indicating that both of the 5 and 6 positions of the ring are substituted. The third product was noncrystalline and a sample purified by chromatography contained ~10% **2**. However, the NMR clearly indicated that it was an isomeric product of substitution on the carbocyclic ring since the indole 3H signal appeared as a doublet and the other spectral features were those expected for a 1:1 ad-

duct. Lithiation occurred at 0° using *tert*-butyllithium in THF and the 2 position was selectively lithiated. Compound **2** was isolated in 39% yield and no **3** or **4** could be detected.

1-Benzenesulfonyl-5-methoxyindole (**5**) and 1-benzenesulfonyl-6-methoxyindole (**6**) were prepared readily by reaction of the sodium salts of the corresponding indoles with benzenesulfonyl chloride. Lithiation was effected using *tert*-butyllithium in THF. Lithiation occurred selectively at the 2 position in both compounds and good yields of adducts were obtained on reaction with carbonyl compounds such as pyridine-2-carboxaldehyde, pyridine-3-carboxaldehyde, and 4-acetylpyridine. These results indicate that the benzenesulfonyl group, in addition to serving as a removable N-protecting group, may selectively activate the 2 position of the indole nucleus toward lithiation. The reaction products were identified as 2-substitution compounds on the basis of NMR data. In particular, in each case the readily observable 3H proton appears as a singlet whereas a doublet is observed in indoles lacking 2 substituents. Lithiation of **5** by *n*-butyllithium was also selective when carried out in refluxing ether for a period of 10 hr.

Experimental Section

1-Benzenesulfonyl-5-methoxyindole (5). A solution of sodium methylsulfinylmethide was prepared in the standard way[4] from 0.40 g (1.75 mmol) of sodium hydride and 10 ml of dimethyl sulfoxide. The solution was cooled in an ice bath and 5-methoxyindole (2.0 g, 1.35 mmol) in ether was added dropwise followed by stirring for 1.5 hr. The solution was then cooled to 0° and 2.88 g (1.65 mmol) of benzenesulfonyl chloride was added followed by stirring for 0.5 hr. Water was added and the reaction mixture was extracted with methylene chloride. After drying and evaporation, recrystallization from methylene chloride–hexane gave **5** (3.29, 82%): mp 98–99°; NMR ($CDCl_3$): δ 3.75 (s, 3), 6.53 (d, 1, J = 4 Hz), and 6.8–8.0 (m, 9).

Anal. Calcd for $C_{15}H_{13}NO_3S$: C, 62.75; H, 4.53. Found: C, 62.85; H, 4.58.

1-Benzenesulfonyl-6-methoxyindole (6) was prepared in a similar manner from 6-methoxyindole,[5] mp 140–142°, 75% yield after recrystallization from methylene chloride–hexane.

Anal. Calcd for $C_{15}H_{13}NO_3S$: C, 62.72; H, 4.53. Found: C, 62.73; H, 4.55.

1-Methyl-5-methoxyindole (1) was prepared by alkylating the anion prepared as above with methyl iodide, 73% yield, mp 103–104° (lit.[6] mp 103–104°) after recrystallization from methylene chloride–hexane.

Lithiation of 1-methyl-5-methoxyindole with *n*-butyllithium was carried out by refluxing an ether solution containing **1** (0.50 g, 3.1 mmol) and 1 equiv of *n*-butyllithium for 13 hr. After cooling to room temperature an ether solution of pyridine-2-carboxaldehyde was added. The solution was stirred for 0.5 hr and poured into water. The product mixture was extracted and separated by column chromatography. Benzene eluted unreacted **1** (0.18 g, 36%). Ethyl acetate eluted a mixture (0.53 g, 74%) of **2, 3,** and **4.** The NMR spectrum of the mixture indicated that the ratio was roughly 4:5:1. Pure samples of **2** and **3** were obtained by chromatography on silica gel using 10% ether in benzene for elution followed by recrystallization. Compound **2**: mp 142–143°; NMR ($CDCl_3$) δ 3.55 (s, 3), 3.78 (s, 3), 4.75 (s, broad, 1), 5.95 (s, 1), 6.13 (s, 1), 6.7–7.8 (m, 6) and 8.55 (d, 1, J = 4.5 Hz).

Anal. Calcd for $C_{16}H_{16}N_2O_2$: C, 71.64; H, 5.96; N, 10.45. Found: C, 71.41; H, 6.00; N, 10.41.

Isomer **3**: mp 104–106°; NMR ($CDCl_3$) δ 3.65 (s, 3), 3.83 (s, 3) 6.28 (s, 1), 6.35 (d, 1, J = 3.0 Hz), 6.93 (d, 1 J = 3.0 Hz). 7.0–7.7 (m, 5), and 8.50 (d, 1, J = 4.5 Hz).

Anal. Calcd for $C_{16}H_{16}N_2O_2$: C, 71.64; H, 5.97. Found: C, 71.57; H, 5.98.

A fraction containing **2** and a third product was rechromatographed to give **4** as an oil, contaminated by ~10% **2**: NMR ($CDCl_3$) δ 3.60 (s, 3), 3.80 (s, 3), 6.22 (d, 1 J = 3.0 Hz), 6.45 (s, 1), 6.82 (d, 1, J = 3.0 Hz), 6.9–7.7 (m, 5), 8.50 (d, 1, J = 4.0 Hz).

Lithiation of 1-methyl-5-methoxyindole with *tert*-butyllithium was carried out by adding *tert*-butyllithium (1.65 mmol) to a solution of **1** (0.25 g, 1.5 mmol) in tetrahydrofuran (30 ml) at 0°. The solution was then stirred at room temperature for 45 min before being cooled again to 0°. A solution of pyridine-2-carboxaldehyde (200 mg, 1.9 mmol) was added. The usual work-up gave the crude product in which no **3** or **4** could be detected by TLC or NMR. Chromatography afforded recovered **1** (0.085 g) and **2** (0.106 g, 39%).

Lithiation of 1-benzenesulfonyl-5-methoxyindole (5) was effected by addition of 1.25 equiv of *tert*-butyllithium at 0° in THF (25–50 ml) followed by stirring at room temperature for 45 min.

Reaction with **pyridine-3-carboxaldehyde** was carried out by dropwise addition of 0.53 g (1.5 equiv) of the aldehyde in THF (5 ml) at 0° followed by stirring at room temperature for 1.5 hr. After hydrolysis and extraction, chromatography on silica gel gave recovered **5** (0.12 g) and 1-benzenesulfonyl-2-(3-pyridylhydroxymethyl)-5-methoxyindole (1.04 g, 84%): mp 157–158° after recrystallization from methylene chloride; NMR (Me_2SO) δ 3.72 (s, 3), 6.44 (s, 2), 6.7–7.1 (m, 4), 7.2–8.0 (m, 9).

Anal. Calcd for $C_{21}H_{18}N_2O_4S$: C, 63.96; H, 4.57; N, 7.11. Found: C, 63.71; H, 4.56; N, 7.02.

Reaction with pyridine-2-carboxaldehyde was done in a similar manner and gave 20% unreacted **5** and 1-benzenesulfonyl-2-(2-pyridylhydroxymethyl)-5-methoxyindole in 59% yield: mp 140–142° after recrystallization from methylene chloride–hexane; NMR (acetone) δ 3.70 (s, 3), 6.25 (s, 1), 6.65 (broad, 1), 6.85 (m, 1), 6.92 (s, 1), 7.1–8.1 (m, 10), and 8.5 (d, 1).

Anal. Calcd for $C_{21}H_{18}N_2O_4S$: C, 63.96; H, 4.57; N, 7.11. Found: C, 63.73; H, 4.64; N, 7.08.

Lithiation of 1-benzenesulfonyl-5-methoxyindole (5) with *n*-butyllithium was effected by refluxing an ether solution with an equimolar amount of *n*-butyllithium for 10 hr. An ether solution of pyridine-2-carboxaldehyde was added and the mixture stirred at room temperature for 0.5 hr. The usual work-up followed by chromatography separated unreacted starting material from **2** which was identified by its NMR spectrum. There was no evidence of formation of **3** or **4.**

Lithiation of 1-benzenesulfonyl-6-methoxyindole (6) was carried out as described for **5.** The products are described below. Reaction with **pyridine-3-carboxaldehyde** gave 1-benzenesulfonyl-2-(3-pyridylhydroxymethyl)-6-methoxyindole in 70% yield: mp 133–134° after recrystallization from ether–hexane; NMR (acetone) δ 3.82 (s, 3), 6.60 (s, 2), 6.83 (d of d, 1, J = 8.2, 1.5 Hz), 7.1–8.0 (m, 10), and 8.55 (broad, 2).

Anal. Calcd for $C_{21}H_{18}N_2O_4S$: C, 63.96; H, 4.57; N, 7.11. Found: C, 63.86; H, 4.60; N, 7.13.

From pyridine-2-carboxaldehyde, 1-benzenesulfonyl-2-(2-pyridylhydroxymethyl)-6-methoxyindole was obtained in 62% yield after recrystallization from methylene chloride–hexane: mp 123–125°; NMR (acetone) δ 3.80 (s, 3), 6.18 (s, 1), 6.63 (broad s, 1), 6.76 (d of d, J = 8.2, 2.2 Hz), 7.1–8.2 (m, 11), and 8.48 (d, 1, J = 4.5 Hz).

Anal. Calcd for $C_{21}H_{18}N_2O_4S$: C, 63.96; H, 4.57; N, 7.11. Found: C, 63.88; H, 4.59; N, 7.13.

From 4-acetylpyridine, 1-benzenesulfonyl-2-[1-(4-pyridyl)-1-hydroxyethyl]-6-methoxyindole was obtained in 58% yield after recrystallization from chloroform: mp 237–238°; NMR (Me_2SO) δ 1.84 (s, 3), 3.77 (s, 3), 5.93 (s, 1), 6.85 (d of d, 1, J = 9.0, 0.8 Hz), 7.1–7.9 (m, 10), and 8.38 (d, J = 5 Hz).

Anal. Calcd for $C_{22}H_{20}N_2O_4S$: C, 64.71; H, 4.90. Found: C, 64.71; H, 4.97.

Acknowledgment. This work was supported by National Cancer Institute Grant CA-12940.

Registry No.—**1**, 2521-13-3; **2**, 56995-09-6; **3**, 56995-10-9; **4**, 56995-11-0; **5**, 56995-12-1; **6**, 56995-13-2; 5-methoxyindole, 1006-94-6; benzenesulfonyl chloride, 98-09-9; 6-methoxyindole, 3189-13-7; *n*-butyllithium, 109-72-8; pyridine-2-carboxaldehyde, 1121-60-4; *tert*-butyllithium, 594-19-4; pyridine-3-carboxaldehyde, 500-22-1; 1-benzenesulfonyl-2-(3-pyridylhydroxymethyl)-5-methoxyindole, 56995-14-3; 1-benzenesulfonyl-2-(2-pyridylhydroxymethyl)-5-methoxyindole, 56995-15-4; 1-benzenesulfonyl-2-(3-pyridylhydroxymethyl)-6-methoxyindole, 56995-16-5; 1-benzenesulfonyl-2-(2-pyridylhydroxymethyl)-6-methoxyindole, 56995-17-6; 4-acetylpyridine, 1122-54-9; 1-benzenesulfonyl-2-[1-(4-pyridyl)-1-hydroxyethyl]-6-methoxyindole, 56995-18-7

References and Notes

(1) R. J. Sundberg and H. F. Russell, *J. Org. Chem.*, **38**, 3324 (1973).
(2) R. Besselievre, C. Thal, H.-P. Husson, and P. Potier, *J. Chem. Soc., Chem. Commun.*, 90 (1975).

(3) D. A. Shirley and P. A. Roussel, *J. Am. Chem. Soc.*, **75**, 375 (1953); F. E. Ziegler and E. B. Spitzner, *Ibid.*, **92**, 3492 (1970); J. Kebrle and K. Hoffmann, *Gazz. Chim. Ital.*, **93**, 238 (1963); J. Kebrle, A. Rossi, and K. Hoffmann, *Helv. Chim. Acta*, **42**, 907 (1959); C. R. Ganellin and H. F. Ridley, *J. Chem. Soc. C*, 1537 (1969).
(4) E. J. Corey and M. Chaykovsky, *J. Am. Chem. Soc.*, **84**, 866 (1962); **87**, 1353 (1965).
(5) Prepared from 4-methoxy-2-nitrotoluene and dimethylformamide dimethyl acetal by the method of W. Leimgruber and A. D. Batcho, as reported at the Third International Congress of Heterocyclic Chemistry, Sendai, Japan, 1971.
(6) J. W. Cook, J. D. Loudon, and P. McCloskey, *J. Chem. Soc.*, 1203 (1951).

O-Acylation of Acidic Methylene Compounds

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We would like to report the results on the O-acylation of the relatively acidic methylene compound 1,2-diphenyl-4-butyl-3,5-pyrazolidinedione (phenylbutazone). The previously reported methods[1] which involved acylation in the presence of aqueous sodium hydroxide or triethylamine gave low yields of the *O*-acyl phenylbutazones (**2**) along with some uncharacterized side products. However, these results are not surprising in view of the fact that **2a** undergoes hydrolysis at a rate approaching that of acetic anhydride.[2] Carbon as well as oxygen acylation of the enolate of phenylbutazone is also possible and the C-acylated phenylbutazone may lead to some of the side products. Therefore, it was logical that methods used for preparing mixed anhydrides and *O*-acyl enolates offered the best opportunity for preparing **2**.

A recent innovation in the preparation of mixed anhydrides has been to employ the reaction of the thallium(I) salt of the weaker of the two acids with the acid halide of the stronger acid.[3a] In general, carbon acid thallium(I) salts are insoluble in most reaction solvents and in the case of phenylbutazone the insoluble nature of the thallous salt would ensure that an excess of the acid halide was always present in the reaction medium, conditions also known to maximize O-acylated product in the related reactions of enolate anions with acylating agents.[3b,4]

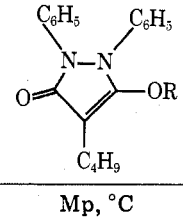
The thallium(I) salt of phenylbutazone (**1**) was a stable, nonhygroscopic white solid. Its infrared spectrum showed loss of all carbonyl bands exhibiting instead a broad absorption centered at 1500 cm^{-1} with a weak shoulder[5] at 1650 cm^{-1}. When **1** was suspended in ether and allowed to react with an acid chloride at room temperature, only one product was observed when the reaction was analyzed by TLC. The ir, uv, and NMR spectra, as well as the elemental analysis of the products were consistent with the corresponding *O*-acyl derivatives of phenylbutazone (**2**) and acid hydrolysis of several of the derivatives prepared regenerated phenylbutazone.

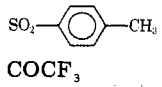
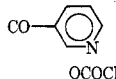
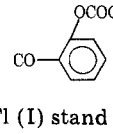
Attempts to prepare **2f** by reaction of **1**, or other salts of phenylbutazone, with nicotinoyl chloride hydrochloride in the presence or absence of an acid scavenger were unsuccessful. Apparently the intermediate acid chloride, generated in situ, preferentially reacted with itself to give an uncharacterized saltlike material faster than it reacted with **1** to give **2f**. Other amino acid chloride hydrochlorides gave similar results so that it was not possible to prepare **2** by the above route when R contained a tertiary amino group.

Therefore, an alternate synthetic scheme was investigated based on the observation of Bourne et al.[6] that 1:1 mixtures of trifluoroacetic anhydride (TFAA) and a carboxylic acid gave the corresponding mixed anhydride and pyridinium trifluoroacetate when the mixtures were allowed to react with pyridine. However, the initial reaction between phenylbutazone and TFAA did not give **2e**, but rather an incompletely characterized adduct that incorporated 1 equiv of TFAA[7] and is considered to be **3**. The NMR spectrum showed an acidic proton at δ 13.6 and loss of the methine hydrogen signal of O=C-CH-C=O centered at δ 3.4, and the ir spectrum showed two strong anhydride-like absorptions at 1830 and 1785 cm^{-1}, as well as complete loss of the carbonyl absorption at 1710 cm^{-1}.

More interesting was the fact that the initial adduct (**3**) could be equilibrated with another anhydride to give a second phenylbutazone adduct which had lost one trifluoroacetyl group and had incorporated the acyl portion of the other anhydride. Thus, **3** was equilibrated with acetic an-

Table I
***O*-Acyl Phenylbutazone Derivatives**



Compd	R	% yield	Mp, °C	Method[a]	Anal. Calcd over found
2a	$COCH_3$	54	49–51	TFAA	Experimental
2b	COC_6H_5	65	116–117.51[a]	Tl (I)	C, 75.70; H, 5.88; N, 6.79 C, 75.92; H, 5.85; N, 6.66
2c	$COC(CH_3)_3$	75	114–115	Tl (I)	Experimental
2d		33	125–126.5	Tl (I)	C, 67.51; H, 5.66; N, 6.06 C, 67.65; H, 5.74; N, 5.89
2e	$COCF_3$				
2f		78	139–141	TFAA	C, 72.62; H, 5.61; N, 10.16 C, 72.39; H, 5.64; N, 9.98
2g		54	85–871[b]	Tl (I)	C, 71.47; H, 5.57; N, 5.95 C, 71.26; H, 5.67; N, 5.82

[a] TFAA and Tl (I) stand for the basic method used as exemplified in the Experimental Section for **2a** and **2c**.