

acidified. The resulting solid was filtered off (carbon dioxide atmosphere) and recrystallized from alcohol or acetic acid. The compound forms brick-red aggregates of jagged prisms melting at 265° (dec.) and dissolving in dilute sodium hydroxide solution.

*Anal.* Calcd. for  $C_{18}H_{14}O_3N_2$ : C, 70.56; H, 4.61. Found: C, 70.62; H, 4.77.

**2-Methyl-1,4-naphthoquinone Guanyldiazone.**—To 3.0 g. of 2-methyl-1,4-naphthoquinone dissolved in 20 cc. of hot alcohol, was added a solution of 6.2 g. of aminoguanidine bicarbonate<sup>3</sup> in dilute nitric acid (3.2 cc. concentrated acid in 18 cc. of water). After refluxing for one hour, yellow crystals separated; 600 cc. of hot water was added to dissolve them, and excess ammonia then added. The solid which separated was recrystallized twice from water, as the nitrate, and then twice from alcohol as the base. The compound forms red, slender, felted needles and melts at 218° (dec.). It is soluble in dilute acetic, lactic and citric acids, but the mineral acid salts are very sparingly soluble.

*Anal.* Calcd. for  $C_{18}H_{12}ON_4$ : C, 63.12; H, 5.30. Found: C, 63.41; H, 5.56.

**2-Methyl-1,4-naphthoquinone Pyridinium Chloride Acethydrazone.**—A solution of 4.2 g. of 2-methyl-1,4-naphthoquinone, 50 cc. of alcohol, 5 cc. of acetic acid and 4.5 g. of Girard reagent P (acethydrazide pyridinium chloride)<sup>4</sup> was refluxed for one hour. The product crystallized out on cooling and was filtered off, extracted with a little hot alcohol, and then recrystallized twice from a larger volume of alcohol. The compound forms light yellow felted needles, melting at 241° (dec.). It gives a relatively stable solution in water. As it is hygroscopic and retains water obstinately, it was dried for two hours at 120° *in vacuo* for analysis.

*Anal.* Calcd. for  $C_{18}H_{14}O_2N_3Cl$ : N, 12.29; Cl, 10.38. Found: N, 12.35; Cl, 10.51.

(3) Thiele and Barlow, *Ann.*, **302**, 311 (1898).

(4) Girard and Sandulesco, *Helv. Chim. Acta*, **19**, 1095 (1936).

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### Substituted Amides of 2,4,6-Trimethylbenzoic Acid

A number of N-substituted 2,4,6-trimethylbenzamides have been prepared. The procedure of Shriner and Fuson<sup>1</sup>

(1) Shriner and Fuson, "Identification of Organic Compounds" 1st ed., John Wiley and Sons, New York, N. Y., 1935, p. 146.

was employed, 1.5 g. of the appropriate amine dissolved in 20 cc. of dry benzene being added to 0.0025 mole of 2,4,6-trimethylbenzoyl chloride dissolved in 25 cc. of dry benzene. The 2,4,6-trimethylbenzoyl chloride was identical with that previously employed for dielectric constant measurements.<sup>2</sup>

The amides obtained, none of which have been described previously in the literature, are listed in the table together with their melting points, general physical appearance and nitrogen analyses. They were crystallized from ethanol or aqueous ethanol with the exception of the last two which were crystallized from ligroin.

The author is indebted to Dr. M. Z. Fineman who furnished a sample of *o*-*t*-butylaniline used in the preparation of one of the amides. This amine was prepared by Dr. Fineman by the nitration of *t*-butylbenzene and separation of the *o*-nitro derivative by fractionation<sup>3</sup> followed by reduction,<sup>4</sup> b. p. 108–109° (14 mm.), acetyl derivative, m. p. 162°.

### 2,4,6-TRIMETHYLBENZOYLAMINES

Amine	Description	M. p., °C. (uncor.)	N analyses, %	
			Calcd.	Found
Ethylamine	Colorless plates	114.5–115.5	7.32	7.46
<i>i</i> -Propylamine	Colorless transparent plates	113.5–115	6.82	7.24
Benzylamine	Colorless plates	137.5–138.5	5.53	5.59
$\alpha$ -Phenylethylamine	Colorless needles	130 –131	5.24	5.51
<i>o</i> -Toluidine	Colorless needles	124 –125.5	5.53	5.76
<i>m</i> -Toluidine	Colorless plates	110 –111.5	5.53	5.45
<i>p</i> -Toluidine	Flat transparent needles	173 –174	5.53	5.65
<i>p</i> -Anisidine	Thin colorless needles	185	5.20	5.40
<i>p</i> -Phenetidine	Needles	171 –172	4.94	5.17
<i>o</i> - <i>t</i> -Butylaniline	Colorless needles	150.5–152	4.74	4.65
$\delta$ -Naphthylamine	Light tan	165 –166.5	4.84	5.04
Piperidine	Transparent prisms	75.5– 77	6.06	5.63
Morpholine	Colorless plates	70 – 71.5	6.01	6.32

The nitrogen analyses were performed by an improved micro-Kjeldahl method of Ma and Zuazaga.<sup>5</sup>

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(2) Kadesch and Weller, *THIS JOURNAL*, **63**, 1310 (1941).

(3) Craig, *ibid.*, **57**, 195 (1935).

(4) Shoesmith and Mackie, *J. Chem. Soc.*, 2334 (1928).

(5) Ma and Zuazaga, *Ind. Eng. Chem., Anal. Ed.*, in press.