perature, 110.1 grams (1 mole) of resorcinol with 1.5 liters of 1,2-dichloroethane was heated under reflux for 10 minutes and cooled then by means of an ice bath. To this efficiently stirred slurry at 0-5° C. was added in one portion 320 grams (2.4 moles) of anhydrous aluminum chloride followed by the dropwise addition over 2 hours of 169 ml. (252.1 grams. 1.2 moles) of trifluoroacetic anhydride. The ice bath was removed after addition of the anhydride was complete. The resulting yellow solution was allowed to warm to room temperature of its own accord with stirring for 4 hours. It was poured onto an excess of chipped ice and extracted with methylene chloride several times. The combined organic extract was washed with water, dried (anhydrous magnesium sulfate), and filtered. Concentration of the filtrate to a small volume (steam bath) afforded a light yellow solid which was purified by recrystallization from carbon tetrachloride to afford 140-150 grams (68 to 73%) of compound I, m.p. 106-108° C. [lit. (*6*), m.p. 103° C.]. This yield represents the average of four such experiments. An additional 10 to 20 grams (5 to 10%) of compound I was obtained by sublimation (100-120° C./0.5-1 mm. of Hg) of the residue from the combined filtrates. [Impure ketone (I) may develop a yellow color in storage. Recrystallization and/or sublimation yields pure, white compound.] Compound I gives a red-brown color with ferric nitrate. Methylation of this ketone according to the procedure of Whalley (*6*) gave the dimethyl ether, m.p. 51-53° C. [lit. (*6*), m.p. 52° C.].

ω-Trifluorophloroacetophenone (II). Following the above procedure, 6.3 grams (0.05 mole) of phloroglucinol with 150 ml. of 1,2-dichloroethane was heated under reflux for several minutes and cooled. The reaction was conducted then at room temperature. To the efficiently stirred mixture was added in one portion 22.7 grams (0.17 mole) of anhydrous aluminum chloride followed by the dropwise addition over 20 minutes of 8.1 ml. (12.1 grams, 0.058 mole) of trifluoroacetic anhydride. After addition, it was stirred for 1 day and poured onto ice. After one extraction with methylene chloride (to remove a small amount of tar), the aqueous layer was extracted with ether. The ether layer was separated, dried (anhydrous magnesium sulfate), and filtered. Concentration of the filtrate to dryness (steam bath) left a yellow solid which was dissolved in benzene and filtered to remove benzene-insoluble phloroglucinol. Chilling of the filtrate deposited 4.4 grams (40%) of yellow crystals, m.p. 174-177° C. dec. Anal. calcd. for $C_8H_5F_3O_4$: C, 43.3; H, 2.3; F, 25.7; mol. wt., 222. Found: C, 42.9; H, 2.6; F, 25.3; mol. wt., 213.

An alcoholic solution of this ketone with ferric nitrate gives a brown color. Infrared and nuclear magnetic resonance studies are reasonable for compound II. IR data show two distinct strong absorptions in the carbonyl region (6.07 μ and 6.20 μ). Proton magnetic resonance spectra show two absorptions. One absorption at $-2.02\ \tau$ is assigned to hydroxyl; the other at $+3.76\ \tau$ is assigned to aromatic protons (relative areas of 3 to 2). Fluorine magnetic resonance data show a single absorption at 7.26 θ^* assigned to CF_3. Both spectra were taken in a mixture composed of 50 mg. of compound II in 0.36 ml. of pyridine with 0.11 ml. of $CFCl_3$.

Alkaline Degradation of ω-Trifluorophloroacetophenone (II). A mixture of 0.5 gram (2 mmoles) of compound II, with 0.8 gram (20 mmoles) of sodium hydroxide in 10 ml. of water was heated under reflux for one-half hour and cooled (ice bath). Acidification (pH 3) afforded a white solid, m.p. 217-219° C., which was identified as phloroglucinol by undepressed mixture melting point and superimposable infrared spectra.

Both ketones failed to give a 2,4-dinitrophenylhydrazone under the usual conditions or according to a newer technique (*4*).

ACKNOWLEDGMENT

The author thanks James T. Elfstrum for his technical assistance and the molecular spectroscopy group of the 3M Company for the spectral data.

LITERATURE CITED

(1) Allen, C.F.H., *Org. Syn.* **14**, 1 (1934).
(2) Heller, G. *Ber.* **48**, 1286 (1915).
(3) Parish, R.C., Stock, L.M., *J. Org. Chem.* **30**, 927 (1965).
(4) Shine, H.J., *Ibid.*, **24**, 1790 (1959).
(5) Tedder, J.M., *Chem. Revs.* **55**, 787 (1955).
(6) Whalley, W.B., *J. Chem. Soc.* **1951** p. 665.

RECEIVED for review Febuary 7, 1966. Accepted June 3, 1966. Contribution No. 370 from Central Research Laboratories, Minnesota Mining and Manufacturing Company.

CORRECTION:

In the article "Heat of Solution, Heat Capacity, and Density of Aqueous Urea Solutions at 25° C." by Edward P. Egan, Jr., and Basil B. Luff [J. CHEM. ENG. DATA **11**, 192 (1966)], the formula given for urea is incorrect. The first sentence of the text should read, "Urea, $CO(NH_2)_2$ is used. . . "

CORRECTION:

In the article "On Latent Heat of Vaporization, Surface Tension, and Temperature," [J. CHEM. ENG. DATA **11**, 69-72 (1966)], there were errors which should be corrected as follows:

Page 69, line 8 should read, "Figure 1 shows $[\lambda\ (1 - 1/T_r)]/T_c$ plotted as a function of . . . . "

Page 71, Equation 10 should be

$$\frac{\gamma^{1/4}}{\gamma_B^{1/4}} = \frac{\lambda}{\lambda_B}\ \frac{TP_B}{T_BP}\ \frac{\rho_1\rho_g}{(\rho_1\ \rho_g)_B}$$

Page 71, Equation 11 should be

$$\gamma/\gamma_B = (\lambda/\lambda_B)^4 \tag{11}$$

Page 71, line 8 should read, "Figure 2 shows the validity of the relation $\gamma/\gamma_B = f(\lambda/\lambda_B)$ . . . . "

Page 71, line 4 should read, "$\gamma_o\ (1 - T_r)^m$ after fitting the the equation using the avaliable . . . . "

Page 71, Equation 12 should be

$$\gamma/\gamma_B = 1.02\ (\lambda/\lambda_B)^{3.16} \cong (\lambda/\lambda_B)^{3.16} \tag{12}$$