TABLE I

R_f VALUES OF SOME COMMON PHENOLS IN VARIOUS SOLVENT SYSTEMS

The solvent systems were prepared by equilibrating at room temperature (approximately 23°) the proportions by volume of the solvents indicated. The organic phase was employed as the descending solvent and the aqueous phase was placed within the chamber to maintain equilibrium conditions.

	Solvent systems		
Phenol	n-$C_5H_{11}OH$-H_2O (mutually saturated)	n-C_4H_9OH-C_6H_6-H_2O 1:9::10	n-C_4H_9OH-C_6H_6-H_2O 1:19::20
Hydroquinone	0.80	0.43	0.078
Resorcinol	.83	.54	.13
Catechol	.86	.70	.38
Toluhydroquinone	.85	..	.23
Phloroglucinol	.61	..	.00
Pyrogallol	.65	..	.025
α-Naphthol	.90	..	.83
β-Naphthol	.92	..	.85

descending solvent face of the alcohol–benzene–water system to drip from the lower boundary of the paper, greater resolution was obtained in some instances. For example, with butyl alcohol-benzene–water (1:19:20), it was found possible to resolve completely a mixture of the first six phenols listed in the table. By continuing development until the most rapidly moving component (catechol) was about to pass from the paper, the following relative rates of migration were found: (hydroquinone = 1.00), phloroglucinol, 0.00; pyrogallol, 0.33; resorcinol, 1.45; toluhydroquinone, 2.4; catechol, 4.7.

The following systems were tested with the listed phenols but found unsatisfactory because of poor resolution: cyclohexanone-water, hexane-acetonitrile, chloroform–formamide and benzene–formamide.[7] Butyl alcohol–water effected some resolution as did benzene–water but these systems were comparatively unsatisfactory for the compounds listed.

A serious limitation of the method may be the volatility of phenols of interest. For example, o-cresol completely evaporates during the interval necessary for the development and drying of the chromatogram.

Acknowledgment.—The author wishes to thank Dr. Alejandro Zaffaroni for his helpful advice.

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[7] A. C. Zaffaroni, R. B. Burton and E. H. Keutmann, *Science*, **111**, 6 (1950).

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2-Keto-4-methylcyclohexanecarboxyanilide

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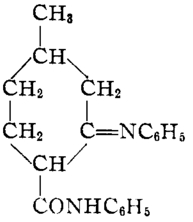
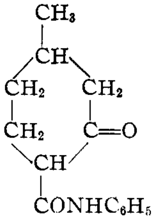
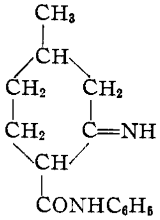
Kötz and Merkel[1] reported the preparation of the "anilinoanilid," I, by heating ethyl 2-keto-4-methylcyclohexanecarboxylate with aniline. In connection with other work,[2] we attempted to repeat their preparation. We obtained a product with the reported melting point, but the analysis corresponded to the ketoanilide, II. It was converted to the corresponding iminoanilide, III, which reacted with aniline to evolve ammonia, as expected.[3] The product of this reaction, presumably I, was not isolated in a pure state. These results are compatible with the results of Sen and Basu[4] who obtained analogs of II, but were unable to obtain any analogs of I from ethyl 2-ketocyclohexanecarboxylate, and with the work of Thomson and Wilson,[3] who prepared analogs of I under different conditions. Hence, it would appear that the analytical data on the compound reported by Kötz and Merkel was in error.


I


II


III

Experimental[5]

2-Keto-4-methylcyclohexanecarboxyanilide (II).—Equimolar amounts of ethyl 2-keto-4-methylcyclohexanecarboxylate and aniline were heated at 140° for 2 hours. The crude product was obtained in 24% yield, m. p. 123–127°. Recrystallizations from benzene–petroleum ether and ethanol gave a pure material, m. p. 129–130°.

Anal. Calcd. for $C_{14}H_{17}O_2N$: C, 72.70; H, 7.42. Found: C, 72.73; H, 7.36.

2-Imino-4-methylcyclohexanecarboxyanilide (III).—This compound was prepared according to Thomson and Wilson[3] and recrystallized from ammoniacal ethanol: m. p. 128–129°; m. p. of mixture with the above compound, 98–108°.

Anal. Calcd. for $C_{14}H_{18}ON_2$: C, 73.01; H, 7.88; N, 12.17. Found: C, 72.61; H, 7.55; N, 12.09.

When this compound was heated with aniline in petroleum ether (b. p. 100–140°), ammonia was evolved over a period of several hours. The oil which separated on cooling was difficult to crystallize. A sample which melted at 95–99° was obtained, but further attempts to purify it resulted in hydrolysis to compound II.

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[1] A. Kötz and B. Merkel, *J. prakt. Chem.*, [2] **79**, 115 (1909).

[2] R. M. Roberts and M. B. Edwards, THIS JOURNAL, **72**, 5537 (1950).

[3] J. K. Thomson and F. J. Wilson, *J. Chem. Soc.*, 111 (1935).

[4] H. K. Sen and U. Basu, *J. Ind. Chem. Soc.*, **6**, 309 (1929).

[5] Microanalyses by Clark Microanalytical Laboratory, Urbana, Ill.

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The Osmotic and Activity Coefficients of Cobalt Bromide and Cobalt Iodide

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Osmotic and activity coefficients of a high order of accuracy have been obtained for calcium chlo-