Undecyl Alcohol.[10]—From undecenyl alcohol by means of hydrogen and platinum-oxide platinum black in alcohol solution; b. p. 100–102° at 2 mm.

Undecyl Bromide.[6]—B. p. 134–137° at 18 mm.; n_D^{20}, 1.4571; d_4^{20}, 1.0521. Calcd. for $C_{11}H_{23}Br$: Br, 33.99. Found: 33.89.

TABLE VIII

β-CYCLOPENTYLETHYL ALKYL ACETIC ACIDS, $C_5H_9(CH_2)_2CH(CO_2H)R$

R =	B. p., °C.	n_D^{20}	d_4^{20}	Calcd., %		Found, %	
				C	H	C	H
H	115–118 (2.4 mm.)	1.4575	0.9849	69.18	10.33	69.01	10.30
C_2H_5	122–124.5 (1.3 mm.)	1.4590	.9602	71.68	10.95	71.71	10.90
n-C_3H_7	130–132 (1.9 mm.)	1.4595	.9533	72.66	11.19	72.22	11.08
n-C_4H_9	136–137 (1 mm.)	1.4608	.9435	73.52	11.40	73.34	11.38
n-C_5H_{11}	150–154 (1.9 mm.)	1.4610	.9360	74.27	11.58	74.03	11.57
n-C_6H_{13}	157–161 (1.9 mm.)	1.4616	.9303	74.93	11.75	74.93	11.64
n-C_7H_{15}	167–169 (2 mm.)	1.4621	.9252	75.52	11.90	75.57	11.88
n-C_8H_{17}	173–176 (1.5 mm.)	1.4629	.9210	76.05	12.02	75.80	11.94

The bacteriological work was carried out by W. M. Stanley. The same strain of bacillus was used as in previous papers in this series.

Summary

1. Two series of cyclopentyl acids have been prepared of the general formulas $C_5H_9CH(CO_2H)R$ when R varied from n-heptyl to n-undecyl and $C_5H_9(CH_2)_2CH(CO_2H)R$ when R varied from ethyl to n-octyl.
2. Bacteriological results showed those acids containing sixteen to eighteen carbon atoms were the most effective bactericides toward *B. Leprae.*
3. Comparisons of cyclopentyl and cyclohexyl substituted acids are given.

URBANA, ILLINOIS

[CONTRIBUTION FROM THE LABORATORY OF GENERAL CHEMISTRY, UNIVERSITY OF WISCONSIN]

THE SOLUBILITY OF META-NITRANILINE IN WATER

BY JAMES H. WALTON AND T. G. FINZEL

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The following solubility determinations were made as a result of solubility studies carried out by Bateman and Baechler.[1] These investigators report a marked lack of agreement among the published solubility data[2] of this compound; further preliminary experiments made at the Forest Products Laboratory showed that saturated solutions are obtained with great difficulty.

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[10] See Blaise and Guerin, *Bull. soc. chim.*, [3] **29**, 1202 (1903).

[1] Bateman and Baechler, "The Solubility of some Amino and Nitro Derivatives of Benzene in Water at 25° C." Report from the Forest Products Laboratory, Madison, Wisconsin.

[2] Vaubel, *J. prakt. Chem.*, **52**, 72 (1895); Carnelley and Thomson, *J. Chem. Soc.*, **53**, 782 (1888).

Experimental

Materials.—The *m*-nitraniline melted at 112.5°. When recrystallized from methanol the melting point remained unchanged.

Method.—Six hundred cc. of boiled water was practically saturated at 15 to 20° above the temperature at which the solubility determination was made. The solution was cooled to the desired temperature and, after several hours, samples were removed. In sampling the saturated solution, muslin or filter paper did not remove the finely suspended solid phase from the solution. A miniature Gooch asbestos filter, as well as a Mandler filter of the type used in bacteriological work, proved satisfactory. This unusual difficulty in filtration is very evidently the cause of the lack of concordant results obtained by earlier investigators. Attention is further called to the extremely slow rate of solution of the *m*-nitraniline. In approaching saturation at 25°, without previous solution at a higher temperature, the solution was stirred for at least twenty-four hours before equilibrium was established. The solution was analyzed by two different methods.

1. **The Titanium Trichloride Method**[3] depends on the reduction of the nitro group by an excess of standard titanium trichloride in a hydrochloric acid solution, the reduction being carried out in a carbon dioxide atmosphere. The excess trichloride is titrated with a standard ferric alum solution, using ammonium sulfocyanate as an indicator. In using this method, it was found that below 35° a correction was necessary for the oxygen dissolved in the sample that was being analyzed.

2. **The Sodium Nitrite Method**[4] depends upon diazotizing the amino group of the *m*-nitraniline, using standard sodium nitrite and a starch–cadmium iodide solution as an outside indicator. In each of the above methods the value of the standard solution was established by means of

Table I

The Solubility of *m*-Nitraniline in Water

Temp., °C.	Grams of *m*-nitraniline in 100 g. of solution	
	$NaNO_2$ method	$TiCl_3$ method
0	0.029	0.0302
10	.046	.0473
20	.072	.0732
25	..	.0914
35	.135	.1407
44	.211	.211
55	.309	.338
65.4	.523	.543
75	.815	.838
83.4	1.19	1.23

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[3] English, *J. Ind. Eng. Chem.*, **12,** 994 (1920).

[4] Callan, Henderson and Stafford, *J. Soc. Chem. Ind.*, **39,** 86T (1920).

pure *m*-nitraniline. The titanium trichloride method gave the more concordant results, making it especially applicable for small concentrations of *m*-nitraniline. The data obtained by this method are listed in the table.

For the analysis of samples containing larger amounts of *m*-nitraniline, the nitrite method compares favorably with the titanium trichloride method, since in this case rather large volumes of the trichloride are necessary. To give some idea of the difference in the results, the sodium nitrite data are also given. When these results are plotted, a perfectly smooth curve results between the temperatures of 0 and 83.4°. Attention is called to the fact that at the latter temperature Sidgwick and Rubie[5] obtained a solubility of 1.7%, while the average figures from the repeated determinations by the authors gave a value of 1.23%.

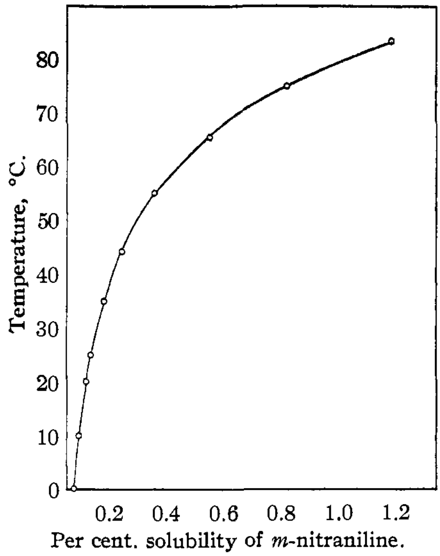

Fig. 1.

Summary

1. A study of the solubility of *m*-nitraniline in water has been made between the temperatures 0 and 83.4°.

2. In agitating *m*-nitraniline and water, it was shown that the solid phase settles with such difficulty and is so finely divided that either an asbestos or Mandler filter must be used as a sampler.

3. At 25° the rate of solution of *m*-nitraniline in water, near the saturation point, is extremely slow.

4. A comparison between the titanium trichloride method and the sodium nitrite method for the analysis of *m*-nitraniline has been made.

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[5] Sidgwick and Rubie, *J. Chem. Soc.*, **119**, 1013 (1921).