are probably due to the differences in the equilibrium conditions noted above.

COLUMBUS, OHIO

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[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF ILLINOIS]

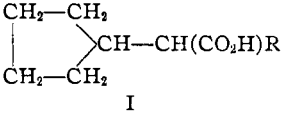
# CYCLOPENTYL ALKYL ACETIC ACIDS AND OMEGA-CYCLOPENTYLETHYL ALKYL ACETIC ACIDS AND THEIR BACTERICIDAL ACTION TOWARD B. LEPRAE. XI[1]

BY G. R. YOHE AND ROGER ADAMS[2]

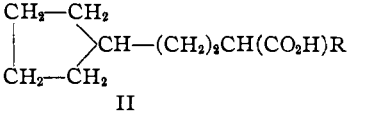
RECEIVED MARCH 26, 1928 PUBLISHED MAY 5, 1928

In previous papers six different isomeric series of acids containing cyclohexyl groups, $C_6H_{11}(CH_2)_xCO_2H$, $C_6H_{11}CH(CO_2H)R$, $C_6H_{11}CH_2CH(CO_2H)R$, $C_6H_{11}(CH_2)_2CH(CO_2H)R$, $C_6H_{11}(CH_2)_3CH(CO_2H)R$, $C_6H_{11}(CH_2)_4CH(CO_2H)R$, have been prepared and have been shown to contain members highly bactericidal *in vitro* to *B. Leprae*. Those acids with the carboxyl group at the end of the chain were not nearly as effective as the isomers with the carboxyl near the ring. This is a very fortunate circumstance since the latter are much more readily prepared and it is, therefore, possible to make a study of the effect of analogous structures with comparative ease. Acids containing a cyclopentyl group in place of the cyclohexyl group are of interest, not only because they make possible a comparison of the effect of the cyclopentyl and cyclohexyl groups upon the bactericidal activity, but because a cyclopentyl group is present in the dihydrochaulmoogric and dihydrohydnocarpic acids, and the cyclopentenyl group is present in the chaulmoogric and hydnocarpic acids.

In this research two series of acids, cyclopentyl alkyl acetic acids (I) and β-cyclopentylethyl alkyl acetic acids (II) have been prepared,



I



II

where R in (I) was *n*-heptyl to *n*-undecyl and R in (II) was ethyl to *n*-octyl. The bacteriological results are given in Tables I and II.

[1] For previous articles in this field see (a) Shriner and Adams, THIS JOURNAL, **47**, 2727 (1925); (b) Noller with Adams, *ibid.*, **48**, 1074 (1926); (c) **48**, 1080 (1926); (d) Hiers with Adams, *ibid.*, **48**, 1089 (1926); (e) Van Dyke and Adams, *ibid.*, **48**, 2393 (1926); (f) Sacks with Adams, *ibid.*, **48**, 2395 (1926); (g) Hiers with Adams, *ibid.*, **48**, 2385 (1926); (h) Adams, Stanley, Ford and Peterson, *ibid.*, **49**, 2934 (1927); (i) Arvin with Adams, *ibid.*, **49**, 2940 (1927); (j) Adams, Stanley, and Stearns, *ibid.*, **50**, 1475 (1928).

[2] This communication is an abstract of a portion of the thesis submitted by G. R. Yohe in partial fulfilment of the requirements for the Degree of Doctor of Philosophy in Chemistry at the University of Illinois.

TABLE I

BACTERIOLOGICAL TESTS TO *B. Leprae*

Cyclopentyl alkyl acetic acids, $C_5H_9CH(CO_2H)R$

| | Dilution of sodium salts in thousands | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| R = | 100 | 111 | 125 | 133 | 143 | 153 | 167 | 176 | 185 | 200 | 250 | 300 |
| *n*-$C_7H_{15}$ | ± | − | + | + | + | + | + | + | + | + | + | + |
| *n*-$C_8H_{17}$ | − | − | + | + | + | + | + | + | + | + | + | + |
| *n*-$C_9H_{19}$ | − | − | + | + | ± | + | + | + | + | + | + | + |
| *n*-$C_{10}H_{21}$ | − | − | − | − | − | + | ± | ± | ± | − | + | + |
| *n*-$C_{11}H_{23}$ | − | − | − | − | − | − | ± | ± | ± | + | + | + |

TABLE II

BACTERIOLOGICAL TESTS TO *B. Leprae*

Cyclopentylethyl alkyl acetic acids, $C_5H_9(CH_2)_2CH(CO_2H)R$

| | Dilution of sodium salts in thousands | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| R = | 10 | 20 | 30 | 40 | 50 | 60 | 70 | 80 | 90 | 100 | 110 | 120 | 130 | 140 | 150 | 160 | 170 | 180 | 190 |
| $C_2H_5$ | − | − | − | + | + | + | + | + | + | + | | | | | | | | | |
| *n*-$C_3H_7$ | − | + | + | + | + | + | + | + | + | + | | | | | | | | | |
| *n*-$C_4H_9$ | − | − | − | + | + | + | + | + | + | + | | | | | | | | | |
| *n*-$C_5H_{11}$ | − | − | − | − | ± | − | − | ± | + | + | | | | | | | | | |
| *n*-$C_6H_{13}$ | − | − | − | − | − | − | − | − | − | − | − | − | − | ± | ± | + | + | + | + |
| *n*-$C_7H_{15}$ | − | − | − | − | − | − | − | − | − | − | − | − | − | − | − | − | ± | + | − |
| *n*-$C_8H_{17}$ | − | − | − | − | − | − | − | − | − | − | − | − | − | − | − | − | − | ± | − |
| $C_5H_9(CH_2)_3CO_2H$ | + | + | + | + | + | + | + | + | + | + | | | | | | | | | |
| $C_5H_9(CH_2)_4CO_2H$ | − | − | − | ± | + | + | + | + | + | + | | | | | | | | | |
| $C_5H_9(CH_2)_5CO_2H$ | − | − | − | ± | + | + | + | + | + | + | | | | | | | | | |

As in the cyclohexyl series, the greatest bactericidal action is found in the acids containing sixteen to eighteen carbon atoms. A comparison of the two cyclopentyl series would indicate that the β-cyclopentylethyl alkyl acetic acids are slightly more effective than the isomeric cyclopentyl alkyl acetic acids. A similar slight difference could be detected in the

TABLE III

COMPARISON OF BACTERICIDAL ACTION OF VARIOUS ACIDS

| Acids | No. | R = | Maximum bactericidal dilutions in thousands |
|---|---|---|---|
| $C_6H_{11}CH(CO_2H)R$ | 1a | $C_8H_{17}$ | 110 |
| | 1b | $C_9H_{19}$ | 190 |
| | 1c | $C_{10}H_{21}$ | 180 |
| $C_5H_9CH(CO_2H)R$ | 2a | $C_9H_{19}$ | 111 |
| | 2b | $C_{10}H_{21}$ | 143 |
| | 2c | $C_{11}H_{23}$ | 153 |
| $C_6H_7CH(CO_2H)R$ | 3a | $C_9H_{19}$ | 150 |
| | 3b | $C_{10}H_{21}$[a] | 167 |
| | 3c | $C_{11}H_{23}$[a] | 125 |
| $C_6H_{11}(CH_2)_2CH(CO_2H)R$ | 4a | $C_6H_{13}$ | 160 |
| | 4b | $C_7H_{15}$ | 220 |
| | 4c | $C_8H_{17}$ | 320 |
| $C_5H_9(CH_2)_2CH(CO_2H)R$ | 5a | $C_7H_{15}$ | 170 |
| | 5b | $C_8H_{17}$ | 180 |

[a] These two acids will be described in a subsequent paper in this series.

cyclohexyl series. It is also interesting to note that the cyclopentyl nonyl acetic acid and cyclopentylethyl heptyl acetic acid are isomers of dihydrohydnocarpic acid, and are far more bactericidal than the latter compound.

Comparisons of cyclopentyl with isomeric cyclohexyl compounds and of cyclopentyl with cyclopentenyl compounds are given in Table III. By comparing isomers 1a, 1b, 1c, with 2a, 2b, 2c, and 4a, 4b, with 5a, 5b, it appears that there is no significant difference in bactericidal effect between the cyclohexyl and cyclopentyl compounds of equal molecular weight or of equal length side chain, though the figures appear to favor the cyclohexyl compounds. The difference between 2a, 2b, 2c, and 3a, 3b, 3c, shows the effect of the olefin linkage. It is obvious that the bactericidal action is not affected markedly by the presence of the double bond.

The acids were prepared by saponification of cyclopentyl alkyl and cyclopentylethyl alkyl malonic esters. The raw material, cyclopentyl bromide, was made from cyclopentanol which, in turn, was made by the catalytic reduction of cyclopentanone. Cyclopentyl ethanol for the second series of compounds was made by the action of ethylene oxide upon cyclopentyl magnesium bromide.

## Experimental Part

**Cyclopentanol.**—This was prepared by the reduction of cyclopentanone by means of hydrogen and platinum-oxide platinum black[3] similar to the procedure described by Noller and Adams,[1c] differing in that 95% ethanol was used as a solvent in place of methanol. A few new observations may be noted about this reaction: (1) catalyst made directly from C. P. chloroplatinic acid was not as active as that from reworked catalyst; (2) the same catalyst could be used several times, though its activity diminished each time; for example, 0.5 mole of cyclopentanone in 150 cc. of 95% ethanol with 1 g. of catalyst required seven hours for the first reduction, eleven hours for the second and twenty-five hours for the third; (3) in several runs in which relatively small amounts of the solvent alcohol were used (100 cc. of alcohol or less to 1 mole of cyclopentanone) and the platinum oxide was reduced to platinum black in the presence of the ketone, considerable reduction to cyclopentane and water was noted.[4]

**Cyclopentyl Bromide.**[1c]—From cyclopentanol as previously described. It is preferable to distil this product under diminished pressure, as under ordinary pressure a small amount of decomposition to cyclopentene and hydrogen bromide is sometimes encountered; b. p. 56° at 45 mm.

**Cyclopentylethanol.**—The same general procedure was used for preparing this as was described by Hiers and Adams[1d] for preparing cyclohexylethanol from cyclohexyl bromide. The only modification was to reflux the reaction mixture for one to two hours after the ethylene oxide had been added to the Grignard solution, then to effect the rearrangement by allowing to stand for one to two days at room temperature. The crude cyclopentylethanol was obtained in 25–35% yields. It was difficult to purify from the ethylene bromohydrin formed as a side product,[5] so the fraction boiling at 85–95° at

[3] Shriner and Adams, THIS JOURNAL, **45**, 2171 (1923); Tuley and Adams, *ibid.*, **47**, 3061 (1925).

[4] See Vavon, *Compt. rend.*, **155**, 287 (1912).

[5] Blaise and Haller, *Compt. rend.*, **134**, 552 (1902).

22 mm. was converted directly to cyclopentylethyl bromide which could be easily purified.

The pure cyclopentylethanol was prepared by converting the bromide to the acetate and then saponifying with dilute methyl alcoholic potassium hydroxide; b. p. 96.5–97° at 24 mm.; $n_D^{20}$, 1.4577; $d_4^{20}$, 0.9180. Calcd. for $C_7H_{14}O$: C, 73.66; H, 12.39. Found: C, 73.09; H, 12.18.

**β-Cyclopentylethyl Bromide.**—From the crude alcohol using the hydrobromic acid and sulfuric acid[6] method, the yields were 60–65%. The phosphorus tribromide method gave essentially the same results; b. p. 75–77° at 19 mm.; $n_D^{20}$, 1.4863; $d_4^{20}$, 1.2860. Calcd. for $C_7H_{13}Br$: Br, 45.14. Found: 45.30.

**Cyclopentylbutanol.**—Prepared from cyclopentylethyl bromide in a similar manner to the preparation of cyclopentylethanol from cyclopentyl bromide. Decomposed with 30% sulfuric acid and distilled, the product was readily obtained pure in 70–75% yields; b. p. 88–92° at 2 mm.; $n_D^{20}$, 1.4613; $d_4^{20}$, 0.9033. Calcd. for $C_9H_{18}O$: C, 75.98; H, 12.77. Found: C, 75.64; H, 12.41.

**δ-Cyclopentylbutyl Bromide.**—From the alcohol and hydrobromic acid and sulfuric acid.[6] The yield was 60–65%; b. p. 110–111° at 17 mm.; $n_D^{20}$, 1.4820; $d_4^{20}$, 1.1872. Calcd. for $C_9H_{17}Br$: Br, 38.77. Found: 38.65.

**δ-Cyclopentylbutyl Cyanide.**—From twenty hours' refluxing of the bromide and 25% excess of potassium cyanide in 75% alcohol, a yield of 80–85% was obtained b. p. 124–126.5° at 17 mm.; $n_D^{20}$, 1.4542; $d_4^{20}$, 0.8887. Calcd. for $C_{10}H_{17}N$: C, 79.39; H, 11.34. Found: C, 79.33; H, 11.29.

**δ-Cyclopentyl Pentanoic Acid.**—From twenty-four hours' heating on a steam cone of the cyanide and excess sodium hydroxide in 60% alcohol, a yield of 80–85% was obtained; b. p. 124–128° at 2 mm.; $n_D^{20}$, 1.4594; $d_4^{20}$, 0.9752. Calcd. for $C_{10}H_{18}O_2$: C, 70.53; H, 10.65. Found: C, 70.21; H, 10.58.

**Diethyl δ-Cyclopentylbutyl Malonate.**—From the bromide and malonic ester by the usual procedure, a yield of about 40% was obtained; b. p. 154–160° at 2.2 mm.; $n_D^{20}$, 1.4493; $d_4^{20}$, 0.9934. Calcd. for $C_{16}H_{28}O_4$: C, 67.55; H, 9.93. Found: C, 67.08; H, 9.75.

**δ-Cyclopentylbutyl Malonic Acid.**—From the ester by the procedure described by Adams, Stanley and Stearns,[1i] a yield of 85% was obtained. From benzene it was readily purified to a m. p. 121–124° (uncorr.). Calcd. for $C_{12}H_{20}O_4$: C, 63.11; H, 8.84. Found: C, 63.10; H, 8.90.

**ε-Cyclopentyl Hexanoic Acid.**—From the malonic acid by heating for two hours at 160–180°, a yield of 75% was obtained; b. p. 133–135° at 1.8 mm.; m. p. 33–33.5°; $n_D^{35}$, 1.4549; $d_4^{35}$, 0.9518. Calcd. for $C_{11}H_{20}O_2$: C, 71.68; H, 10.95. Found: C, 71.65; H, 10.83.

**Diethyl β-Cyclopentylethyl Alkyl Malonates.**—These were all prepared by the usual procedure, condensing β-cyclopentylethyl bromide with the sodium derivatives of the various diethyl alkyl malonates; yields, 50–60%.

**Diethyl Cyclopentyl Alkyl Malonates.**—Cyclopentyl malonic ester[7] (b. p. 115–117° at 2 mm.; $n_D^{20}$, 1.4440; $d_4^{20}$, 1.0325) was dissolved in benzene, the calculated amount of sodium dissolved in this solution and the calculated amount of the proper alkyl halide introduced;[1i] yields, 50–60%.

**β-Cyclopentylethyl Alkyl Malonic Acids and Cyclopentyl Alkyl Malonic Acids.**—These were prepared by heating with excess alcoholic potassium hydroxide overnight and working up as previously described.[1h] All which could not be purified readily were decomposed in crude form to the monobasic acid.

[6] "Organic Syntheses," John Wiley and Sons, Inc., New York, Vol. I, p. 1.

[7] Verwey, *Ber.*, **29**, 1996 (1896).

**β-Cyclopentylethyl Alkyl Acetic Acids and Cyclopentyl Alkyl Acetic Acids.**—From the malonic acids by heating for two hours at 160–180°, essentially quantitative yields of monobasic acids were obtained.

**Alkyl Halides.**—Previous articles have described most of those used.[1h] Decyl bromide and undecyl bromide were prepared from the corresponding alcohols and hydrobromic acid and sulfuric acid.[4]

**Decyl Alcohol.**[8]—From nonyl magnesium bromide and formaldehyde[9] a 60% yield of product was obtained; b. p. 115–120° at 15 mm.

TABLE IV

DIETHYL CYCLOPENTYL ALKYL MALONATES, $C_5H_9C(CO_2C_2H_5)_2R$

| R = | B. p., °C. | $n_D^{20}$ | $d_4^{20}$ | Calcd., % C | Calcd., % H | Found, % C | Found, % H |
|---|---|---|---|---|---|---|---|
| $n$-$C_7H_{15}$ | 143–146 (1 mm.) | 1.4548 | 0.9749 | 69.88 | 10.52 | 69.56 | 10.23 |
| $n$-$C_8H_{17}$ | 160–165 (1 mm.) | 1.4553 | .9659 | 70.53 | 10.66 | 70.52 | 10.67 |
| $n$-$C_9H_{19}$ | 152–155 (0.6 mm.) | 1.4567 | .9617 | 71.13 | 10.81 | 71.00 | 10.65 |
| $n$-$C_{10}H_{21}$ | 169–171 (1 mm.) | 1.4571 | .9560 | 71.68 | 10.95 | 71.69 | 10.88 |
| $n$-$C_{11}H_{23}$ | 186–189 (1 mm.) | 1.4580 | .9522 | 72.02 | 11.24 | 72.38 | 11.16 |

TABLE V

DIETHYL β-CYCLOPENTYLETHYL ALKYL MALONATES, $C_5H_9(CH_2)_2C(CO_2C_2H_5)_2R$

| R = | B. p., °C. | $n_D^{20}$ | $d_4^{20}$ | Calcd., % C | Calcd., % H | Found, % C | Found, % H |
|---|---|---|---|---|---|---|---|
| H | 125 (2 mm.) | 1.4478 | 1.0082 | 65.52 | 9.50 | 65.39 | 9.39 |
| $C_2H_5$ | 126–129 (1.9 mm.) | 1.4511 | .9924 | 67.55 | 9.93 | 67.37 | 9.89 |
| $n$-$C_3H_7$ | 134–135 (1.7 mm.) | 1.4510 | .9873 | 68.40 | 10.14 | 67.99 | 10.01 |
| $n$-$C_4H_9$ | 136–140 (1.8 mm.) | 1.4523 | .9783 | 69.17 | 10.33 | 68.95 | 10.30 |
| $n$-$C_5H_{11}$ | 148–150 (1.1 mm.) | 1.4526 | .9688 | 69.88 | 10.51 | 69.71 | 10.38 |
| $n$-$C_6H_{13}$ | 157–162 (1 mm.) | 1.4531 | .9624 | 70.53 | 10.66 | 70.42 | 10.65 |
| $n$-$C_7H_{15}$ | 172–174 (2 mm.) | 1.4541 | .9563 | 71.13 | 10.81 | 70.91 | 10.79 |
| $n$-$C_8H_{17}$ | 182–184 (1.2 mm.) | 1.4548 | .9524 | 71.68 | 10.95 | 71.42 | 10.97 |

TABLE VI

β-CYCLOPENTYLETHYL ALKYL MALONIC ACIDS, $C_5H_9(CH_2)_2C(CO_2H)_2R$

| R = | M. p., °C. (uncorr.) | Calcd., % C | Calcd., % H | Found, % C | Found, % H |
|---|---|---|---|---|---|
| H | 126.5 | 59.96 | 8.06 | 59.97 | 8.07 |
| $C_2H_5$ | 141–143 | 63.11 | 8.84 | 63.72 | 8.79 |
| $n$-$C_3H_7$ | 137–138 | 64.42 | 9.16 | 64.43 | 9.21 |
| $n$-$C_4H_9$ | 139–140.5 | 65.52 | 9.50 | 65.40 | 9.44 |
| $n$-$C_5H_{11}$ | 124–127 | 66.62 | 9.70 | 66.04 | 9.64 |
| $n$-$C_6H_{13}$ | 129.5–130 | 67.55 | 9.93 | 67.19 | 9.95 |

TABLE VII

CYCLOPENTYL ALKYL ACETIC ACIDS, $C_5H_9CH(CO_2H)R$

| R = | B. p., °C. | M. p., °C. (uncorr.) | $n_D^{20}$ | $d_4^{20}$ | Calcd., % C | Calcd., % H | Found, % C | Found, % H |
|---|---|---|---|---|---|---|---|---|
| $n$-$C_7H_{15}$ | 155–160 (1.4 mm.) | ..... | 1.4594 | 0.9312 | 74.27 | 11.58 | 74.39 | 11.64 |
| $n$-$C_8H_{17}$ | 166–169 (2 mm.) | ..... | 1.4609 | .9279 | 74.93 | 11.75 | 74.63 | 11.67 |
| $n$-$C_9H_{19}$ | 177–178.5 (1.4 mm.) | 37–37.5 | .... | ... | 75.52 | 11.90 | 75.46 | 11.85 |
| $n$-$C_{10}H_{21}$ | 189–190.5 (1.7 mm.) | 34.5–36 | .... | ... | 76.05 | 12.02 | 76.08 | 12.03 |
| $n$-$C_{11}H_{23}$ | 193–197 (1.3 mm.) | 43.5–45.5 | .... | ... | 76.52 | 12.14 | 76.83 | 12.16 |

[8] Y. Talvitie, *Ann. Acad. Sci. Fennicae*, **16**, 26A, 1 (1927).

[9] Wood and Scarf, *J. Soc. Chem. Ind.*, **42**, 13T (1923).

**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., % C | Calcd., % H | Found, % C | Found, % 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

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[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

RECEIVED MARCH 27, 1928 PUBLISHED MAY 5, 1928

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.

[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).