

uct was collected and crystallized from ethyl alcohol, from which it separated as long narrow orange colored plates, m. p. 247–248°. The yield was 2.8 g. or 75%.

*Anal.* Calcd. for  $C_8H_5O_2NBrI$ : N, 3.98. Found: N, 4.02, 4.15.

Repeated attempts to prepare 5-bromo-7-iodoisatin by the action of iodine monochloride on 5-bromoisatin resulted only in the recovery of 5-bromoisatin. This is in accord with the previous observation that iodine cannot be introduced directly into the isatin nucleus in position 7.<sup>1</sup>

**2-Amino-3-bromo-5-iodobenzoic Acid.**—This compound was obtained when the solution resulting from the addition of 3% hydrogen peroxide to an alkaline solution of 5-iodo-7-bromoisatin was rendered acidic. The compound crystallized from ethyl alcohol as colorless needles, m. p. 226–227°.

The same compound was prepared by the action of bromine on an alcoholic solution of 2-amino-5-iodobenzoic acid. The identity of the two preparations was established by melting point methods.

*Anal.* Calcd. for  $C_7H_5O_2NBrI$ : N, 4.10. Found: N, 4.23, 4.33.

(1) Sumpter and Amundsen, *THIS JOURNAL*, **54**, 1917 (1932).

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#### Methylnepentylacetic Acid (2,4,4-Trimethylpentanoic Acid), its Methyl Ester, Amide and Acetanilide

1. Methylnepentylacetic acid obtained in 6% yield by the oxidation of 160 moles of diisobutylene<sup>1</sup> with sodium dichromate and sulfuric acid, was purified by repeated refractionation through a column of 15 theoretical plates until constant index material was obtained. The **methyl ester**, prepared by treating the acid with an excess of methanol saturated with hydrogen chloride, on fractionation gave material  $n^{20}_D$  1.4124 (Valentine), b. p. 162.25° at 730 mm. (Cottrell),  $d^{20}$  0.8706.

The **methylnepentylacetic acid** obtained by saponification of the ester, on fractionation gave  $n^{20}_D$  1.4233 (Valentine), b. p. 217.40° at 730 mm. (Cottrell),  $d^{20}$  0.9028.

(1) Cf. Whitmore and co-workers, *THIS JOURNAL*, **53**, 3137 (1931); **54**, 3707 (1932); **56**, 1397 (1934).

*Anal.* Calcd. for  $C_8H_{16}O_2$ : C, 66.6; H, 11.1. Found: C, 66.5; H, 11.5.

2. Neopentylethylene, prepared by the addition of *t*-butylmagnesium chloride to a cold ether solution of allyl bromide, was treated with hydrogen bromide in the presence of an antioxidant, diphenylamine, to give 4,4-dimethyl-2-bromopentane, b. p. 56–60° at 29–39 mm.,  $n^{20}_D$  1.4460. This was caused to react with magnesium in anhydrous ether in 66.6% yield. The Grignard reagent was cooled in a salt-ice-bath and saturated with carbon dioxide. On decomposition and fractionation, methylnepentylacetic acid was obtained in 51.6% yield, distilling at 108° at 14 mm.,  $n^{20}_D$  1.4228–1.4230.

3. Methylnepentylcarbinol, b. p. 136–136.5° at 730 mm.,  $n^{20}_D$  1.4180–1.4186, was treated with anhydrous hydrogen chloride under pressure for twenty-two weeks. The oil layer obtained was washed with water and 10% potassium carbonate solution, then dried over anhydrous potassium carbonate and fractionated through the 15-plate column. The methylnepentylcarbinyl chloride, b. p. 63–65° at 85 mm.,  $n^{20}_D$  1.4178–1.4187, was treated with magnesium in anhydrous ethyl ether, and the cooled Grignard reagent was saturated with carbon dioxide. On decomposition and fractionation, the yield of methylnepentylacetic acid b. p. 109° at 14 mm.,  $n^{20}_D$  1.4233, was 50% based on the chloride.

4. **Methylnepentylacetamide** was prepared by thionyl chloride followed by treatment of an ether solution of the acid chloride with anhydrous ammonia. On recrystallization from petroleum ether the derivative gave m. p. 123°. No depression in m. p. was shown by mixtures with the amides from the oxidation and synthetic acids.

*Anal.* Calcd. for  $C_8H_{17}ON$ : N, 9.80. Found: N, 9.68.

5. **Methylnepentylacetanilide** was prepared by adding a solution of aniline in benzene to methylnepentylacetyl chloride, washing with dilute hydrochloric acid and carbonate solution, and recrystallizing from a mixture of petroleum ether and benzene, m. p. 117.5°; mixed melting points showed no depression.

*Anal.* Calcd. for  $C_{14}H_{21}ON$ : N, 6.40. Found: N, 6.60.

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