chloric acid, and the solution was then evaporated to dryness *in vacuo*. Water was added to the residue, and the evaporation repeated, this operation being done several times. The glue-like mass was treated with a minimum of warm water for solution (5 ml.). It was found that the amino acid could best be isolated from this solution by titration with lithium hydroxide, and making use of the solubility of lithium chloride in alcohol. The solution was treated dropwise with concentrated lithium hydroxide solution as long as a test portion did not give a precipitate with several volumes of ethanol (*p*H 2.5–3). Ten volumes of ethanol then were added slowly during 20 minutes, and the liquid filtered and left to stand, when the amino acid gradually separated as crystalline grains. After five hours, the product was filtered, washed with ethanol and dried (weight 1.65 g.). The filtrates, on standing overnight, gave an additional 150 mg. The total yield was 1.80 g. or 74%. This was the best yield obtained in several runs, the difficulty being in adjusting the amount of lithium hydroxide used. If too much of the alkali is employed, an amorphous high-melting product precipitates before the amino acid separates, while if too little lithium hydroxide is added, some of the amino acid remains in solution after alcohol is added. The amorphous product may be the monolithium salt of the β-methylglutamic acid.

The methylglutamic acid was recrystallized by adding 3–4 volumes of alcohol and seed to its concentrated aqueous solution. The highest m.p. observed, after several recrystallizations, was 169.5–170.5°.

Anal. Calcd. for $C_6H_{11}NO_4$: C, 44.72; H, 6.83. Found: C, 44.58; H, 6.72.

Both the amino acid and the high-melting substance gave purple colors on warming with a dilute aqueous acetic acid solution of ninhydrin.

N-Benzoyl-β-methylglutamic Acid Monohydrate.—The amino acid was benzoylated by the procedure of Bullerwell, Lawson and Morley,[6] as used for glutamic acid, giving a white, crystalline, hydrated, benzoyl derivative. This compound was recrystallized several times from water for analysis. On heating, it melted at 114–116°, with partial solidification above 120°, forming the anhydrous acid which melted about 142°.

Anal. Calcd. for $C_{13}H_{17}NO_6$: C, 55.12; H, 6.01. Found: C, 54.82; H, 5.83.

N-Acetyl-α-carbethoxyglutamic Acid Diethyl Ester.—The addition of ethyl acrylate to ethyl acetamidomalonate was carried out in a manner similar to that used for the crotonate. The reactants were mixed more slowly and the product was extracted by ether after the steam distillation. Evaporation of the ether solution left a pasty solid; 43.4 g. (0.2 mole) of the acetamidomalonate gave 51 g. of product or 80.4% yield. This ester was best recrystallized from ether–petroleum ether (2:1). After three recrystallizations, it had m.p. 81–82.5°. The analysis indicated that a carbethoxy group was not lost in the condensation.

Anal. Calcd. for $C_{14}H_{23}NO_7$; C, 52.997; H, 7.26. Found: 52.67; H, 6.77. Calcd. for $C_{11}H_{19}NO_5$ (decarbethoxylated product): C, 53.88; H, 7.76.

(6) R. A. F. Bullerwell, A. Lawson and H. V. Morley, *J. Chem. Soc.*, 3283 (1954).

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An Explosion during the Preparation of Neopentyl Alcohol

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The preparation of neopentyl alcohol as recently described[1] has been repeated successfully many times in this Laboratory. Recently, however, a violent explosion occurred during a run. Investigation revealed that the explosion undoubtedly took place after the acetone peroxide which had been removed by suction filtration was allowed to be sucked dry on the funnel. The suction flask was unharmed but much damage resulted from the explosion. Accordingly, when this preparation is carried out as described,[1] care should be taken that the solid peroxide be kept moist and destroyed with care. Preferably, an alternate procedure which avoids the formation of acetone peroxide should be used.

(1) J. H. Hoffman and C. E. Boord, THIS JOURNAL, **77**, 3139 (1955).

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Preparation of Modified Squalenes

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There is considerable evidence that cholesterol plays an important, if ambiguous, role in the development of atherosclerosis.[1] Recent progress in the elucidation of the biogenesis of cholesterol has demonstrated that the hydrocarbon squalene is the most efficient precursor to cholesterol yet discovered.[2] It was our purpose to prepare modified squalenes in the hope that these compounds would act as metabolic antagonists to the endogenous syntheses of cholesterol. The reduction of the serum cholesterol concentration should have a beneficial effect on the severity of the atherosclerotic lesions.

It was shown recently that the hydrocarbon regenerated from squalene hexahydrochloride has a different structure from the natural squalene.[3] This can be differentiated from the natural material in biological systems.[4] We have investigated the preparation of squalene hexahydrobromides and particularly the effect of peroxide on the mode of addition of hydrogen bromide. It was hoped that materials more closely analogous to natural squalene could be regenerated from squalene hexahydrobromides of approxpiate structure. For example, a "squalene" differing from natural squalene in the position of one of the double bonds possibly could function as a metabolic antagonist to squalene utilization. We repeated the experiment of Heilbron and co-workers[5] and found, as these workers had foreshadowed, that a mixture of hexahydrobromides results when squalene is treated with hydrogen bromide in acetone solution. This mixture is rather readily separated by fractional crystallization into isomers melting at 112–114, 135–138, and 151–153°. Schmidt[6] reports the isolation of two hexahydrobromides, m.p. 116–118° and 136–138° from synthetic squalene. The infrared spectra of these isomers were similar, but distinctly different. When the hydrogen bromide reaction was run in the presence of 0.02 mole % of ascaridole a different result was obtained. A single hexahydrobromide

(1) I. H. Page, *Circulation*, **10**, 1 (1954).
(2) R. G. Langdon and K. Bloch, THIS JOURNAL, **74**, 1869 (1952).
(3) W. G. Dauben, H. L. Bradlow, N. K. Freeman, D. Kritchevsky and M. Kirk, *ibid.*, **74**, 4321 (1952).
(4) R. G. Langdon and K. Bloch, *J. Biol. Chem.*, **202**, 77 (1953).
(5) I. M. Heilbron, E. D. Kamm and W. M. Owens, *J. Chem. Soc.*, 1630 (1926).
(6) J. Schmidt, *Ann.*, **547**, 115 (1941).