N-(4-Carbethoxyphenyl)-*dl*-α-alanine

Into a test-tube were placed 3.3 g. of ethyl *p*-aminobenzoate and 1.5 g. of *dl*-α-bromopropionic acid. The mixture was heated to 135° for one hour, after which the brown melt was cooled and extracted with ten 10-ml. portions of ether. The ether solution was extracted with three 25-ml. portions of 10% hydrochloric acid, then the ether solution was dried over anhydrous sodium sulfate for 12 hours. The sodium sulfate was filtered off, and the ether evaporated to give 2.0 g. of brown crystals melting at 120–124°. The crude product was next dissolved in 10% sodium bicarbonate, treated with decolorizing charcoal and filtered. The pale yellow filtrate was brought to *p*H 2 with hydrochloric acid, and the solid product was filtered off. This material was recrystallized from 100 ml. of water to give 1.2 g. (51% of theory) of N-(4-carbethoxyphenyl)-*dl*-α-alanine melting at 133–135°.

Anal. Calcd. for $C_{12}H_{15}O_4N$: N, 6.0. Found: N, 5.9.

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9,9,10,10-Tetramethyldihydroanthracene and 9,9,10,10-Tetramethylperhydroanthracene

9,9,10,10-Tetramethyldihydroanthracene (I) was prepared by the procedure of Hugel and Lehrer[1] for introducing alkyl groups into the 9- and 10-positions of anthracene. Seventeen grams (0.08 mole) of 9,10-dimethylanthracene, 7 g. (0.03 mole) of sodium, 25 g. (0.2 mole) of dimethyl sulfate and 750 ml. of anhydrous ether yielded white plates which were recrystallized from benzene–ethanol. A yield of 7 g. (37%), melting at 168°, was obtained.

Anal. Calcd. for $C_{18}H_{20}$: C, 91.54; H, 8.46. Found: C, 91.41; H, 8.74.

Hydrogenation of 8 g. of I in a 125-ml. rotating autoclave with 2 g. of nickel on kieselguhr catalyst,[2,3] 25 ml. of cyclohexane and 100 atm. initial hydrogen pressure for 7 hours at 120° yielded 9,9,10,10-tetramethylperhydroanthracene (II). Purification by molecular distillation gave 5 g. (60%) of a clear colorless liquid insoluble in 95% ethanol, but readily soluble in hydrocarbons, $n^{20}D$ 1.5186, d^{20}_4 0.969. This compound had no absorption in the ultraviolet region.

Anal. Calcd. for $C_{18}H_{32}$: C, 87.03; H, 12.97; MR_D, 78.7. Found: C, 86.85; H, 12.87; MR_D, 77.8.

Dehydrogenation of II over 5% platinum on alumina catalyst at 300° yielded I.

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(1) G. Hugel and M. Lehrer, *Compt. rend.*, **195**, 249 (1932).

(2) V. N. Ipatieff and B. B. Corson, *Ind. Eng. Chem.*, **30**, 1039 (1938).

(3) H. Pines and V. N. Ipatieff, THIS JOURNAL, **61**, 1076 (1939).

(4) Universal Oil Products Company Predoctorate Fellow (1947–1949).

(5) Universal Oil Products Company, Riverside, Illinois.

n-Butylsulfamic Acid

(A).—Sulfur trioxide (6.5 g.), generated by heating 65% oleum, was passed into a solution of 10.0 g. of dioxane in 70 ml. of carbon tetrachloride with cooling and stirring (anhydrous conditions). A white precipitate of dioxane–sulfur trioxide addition-compound formed, and the mixture was stirred for 30 minutes. A solution of 14.6 g. of *n*-butylamine in carbon tetrachloride was added dropwise, and the mixture stirred for one hour more. After 48 hours in the cold-room the mixture was filtered and the precipitate washed with ether. A 70% yield (12.8 g.) of *n*-butylammonium *n*-butylsulfamate, white silky flakes, m.p. 127–128° (cor.) was obtained.

To 13.4 g. of the salt, dissolved in 20 ml. of water at 0–5°, 3.3 ml. of concentrated sulfuric acid was added slowly with stirring. The white flaky precipitate of *n*-butylsulfamic acid was collected, washed and dried, giving 4.98 g. (55%, based on ammonium salt) of **n-butylsulfamic acid**. The product was recrystallized from ethanol–benzene (1:1), giving colorless plates, m.p. 177–178° (cor.) with decomposition. It gave positive sodium-fusion tests for nitrogen and sulfur.

Anal. Calcd. for $C_4H_{11}NO_3S$: N, 9.14; neut. equiv., 153. Found[1]: N, 9.21; neut. equiv., 150.

(B).—A mixture of 0.5 g. of sulfamic acid and 3.0 g. of *n*-butylamine was heated in a sealed tube at 185–190° for 200 minutes. After filtration, the tube contents were diluted with ether. The resulting precipitate was collected, washed with ether and dried, giving 0.52 g. (44%) of *n*-butylammonium *n*-butylsulfamate. The free sulfamic acid was liberated as above.

(1) Nitrogen by method of A. Friedrich, *Z. physiol. Chem.*, **216**, 68 (1933). Analysis by Mr. R. S. Pyke.

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Cyclohexyl *n*-Octadecyl Ketone and Cyclohexyl-*n*-octadecylcarbinol

The Grignard reagent was formed in the usual manner from 186 g. (0.56 mole) of *n*-octadecyl bromide, 14.4 g. (0.59 g. atom) of magnesium and 400 ml. of ether. To the cooled Grignard reagent was added 52 g. (0.28 mole) of cadmium chloride and the ether was removed by distillation. Benzene (550 ml.) was added to the residue and the mixture heated to reflux and stirred while a solution of 80.6 g. (0.36 mole) of cyclohexanecarboxylic acid chloride in 100 ml. of benzene was added slowly. The resulting mixture was held at reflux temperature for one hour and then added to an excess of a mixture of ice and hydrochloric acid. The organic layer was washed with dilute sodium carbonate and water and the benzene removed by distillation. The residual oil was distilled to give 125 g. (61% yield) of cyclohexyl *n*-octadecyl ketone boiling at 208° at 0.8 mm. and melting at 47–47.5°.

Anal. Calcd. for $C_{25}H_{48}O$: C, 82.4: H, 13.2. Found: C, 82.3; H, 13.0.

The residue remaining after distillation was 5.5 g. of solid melting at 72–76°. Recrystallization from petroleum ether gave a product melting at 75–76°. A mixed melting point between this compound and an authentic sample of hexatriacontane (m.p. 76–77°)[1] showed no depression.

A 2,4-dinitrophenylhydrazone derivative of cyclohexyl *n*-octadecyl ketone was prepared in the usual manner. After three recrystallizations from ethanol and two from ethyl acetate, the product melted at 70–71°.

Anal. Calcd. for $C_{31}H_{52}N_4O_4$: N, 10.30. Found: N, 10.50.

Cyclohexyl-*n*-octadecylcarbinol.—A solution of 40 g. (0.11 mole) of cyclohexyl *n*-octadecyl ketone in 100 ml. of ether was added to a stirred solution of 15 g. (0.4 mole) of lithium aluminum hydride in 300 ml. of ether. After ten hours of stirring, 15 ml. of water was added dropwise and the mixture poured into excess ice–sulfuric acid mixture. The ether layer was separated, washed thoroughly with water, and cooled. There separated 39 g. (97% yield) of carbinol melting at 61–62°. Recrystallization from acetone and ethanol did not increase the melting point.

Anal. Calcd. for $C_{25}H_{50}O$: C, 82.0; H, 13.7. Found: C, 81.9; H, 13.7.

A 3,5-dinitrobenzoate derivative of the above carbinol was prepared by heating to 75° equimolar amounts of carbinol and 3,5-dinitrobenzoyl chloride in pyridine solution, pouring into excess water, and recrystallizing the precipitated solid from acetone and ethanol. The product melted at 75.5–76.5°.

Anal. Calcd. for $C_{32}H_{52}N_2O_6$: N, 5.00. Found: N, 5.45.

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(1) D. A. Shirley and W. H. Reedy, THIS JOURNAL, **73**, 4886 (1951).