Anal. Calcd. for $C_{21}H_{17}O_6N_5$: C, 57.93; H, 3.94. Found: C, 57.93; H, 3.75.

Treatment of the sodio derivative from 2.44 g. of di-*t*-butyl benzylmalonate and 0.29 g. of sodium hydride with 1.50 g. (0.008 mole) of *o*-nitrobenzoyl chloride as described above gave 1.44 g. (71% yield) of crude **o-nitrophenyl β-phenylethyl ketone**. Purification of the oil was effected by distillation (b.p. 163.5–166° (0.5 mm.) with some decomposition) followed by chromatography on alumina. The ketone was eluted by benzene and was then evaporatively distilled at 85–95° (0.02–0.04 mm.); $n^{25}D$ 1.5833.

Anal. Calcd. for $C_{15}H_{13}O_3N$: C, 70.58; H, 5.13. Found: C, 70.03; H, 4.90.

Treatment of the sodio derivative from 1.22 g. of di-*t*-butyl benzylmalonate and 0.15 g. of sodium hydride with 0.67 g. (0.004 mole) of cinnamoyl chloride as described above gave 0.75 g. (79% yield) of **styryl β-phenylethyl ketone**, m.p. 49–52°. Recrystallization from petroleum ether (b.p. 90–100°) raised the m.p. to 52.5–53.8° (reported 53°,[12] 53–54°[13]).

Treatment of the sodio derivative from 1.32 g. of di-*t*-butyl *n*-octylmalonate and 0.15 g. of sodium hydride with 0.65 g. (0.004 mole) of caprylyol chloride as described above gave after crystallization from methanol 0.59 g. of ***n*-heptyl *n*-nonyl ketone**, m.p. 40.8–41.6° (reported,[8] 42°). A second crop amounting to 0.07 g., m.p. 37–39.5°, was obtained, raising the yield to 65%. The hydantoin, 5-heptyl-5-nonylhydantoin, prepared from the ketone by the procedure of Henze and Speer,[14] crystallized from methanol in clusters of white needles, m.p. 116.5–117.2° (reported,[8] 123°).

Anal. Calcd. for $C_{19}H_{36}O_2N_2$: N, 8.64. Found: N, 8.41.

Treatment of the sodio derivative from 1.20 g. of di-*t*-butyl cyclohexylmalonate and 0.15 g. of sodium hydride with 0.56 g. (0.004 mole) of benzoyl chloride as described above afforded an oil which was converted to the **2,4-dinitrophenylhydrazone of phenyl cyclohexylmethyl ketone**. The crude derivative, m.p. 145–150°, was obtained in 56% yield. Recrystallization from benzene–petroleum ether raised the m.p. to 148.3–148.6°.

Anal. Calcd. for $C_{20}H_{22}O_4N_4$: N, 14.65. Found: N, 15.00.

Treatment of the sodio derivative from 2.44 g. of di-*t*-butyl benzylmalonate and 0.29 g. of sodium hydride with 1.24 g. (0.008 mole) of *o*-toluyl chloride as described above gave ***o*-tolyl β-phenylethyl ketone**[15] in 70% yield. The product was purified by evaporative distillation at 125–130° (0.05 mm.), $n^{25}D$ 1.5720.

Anal. Calcd. for $C_{16}H_{16}O$: C, 85.67; H, 7.19. Found: C, 85.26; H, 7.36.

Treatment of the sodio derivative from 14.95 g. of di-*t*-butyl cyclohexylmalonate and 1.6 g. of sodium hydride with 7.75 g. (0.05 mole) of *o*-toluyl chloride as described above afforded 6.02 g. (56% yield) of ***o*-tolyl cyclohexylmethyl ketone**, b.p. 126–130° (0.5 mm.), $n^{25}D$ 1.5278. An analytical sample was obtained by redistillation; b.p. 109.5–110° (0.04 mm.), $n^{25}D$ 1.5290.

Anal. Calcd. for $C_{15}H_{20}O$: C, 83.28; H, 9.32. Found: C, 83.16; H, 9.06.

An attempt to prepare the 2,4-dinitrophenylhydrazone of this ketone was unsuccessful.

Di-*t*-butyl Benzylmesitoylmalonate.—The sodio derivative from 4.88 g. (0.016 mole) of di-*t*-butyl benzylmalonate and 0.6 g. (0.025 mole) of sodium hydride, was heated at 80° in 150 ml. of benzene with 2.92 g. (0.016 mole) of freshly distilled mesitoyl chloride for 20 hours. The gelatinous precipitate was removed by filtration and evaporation of the filtrate under reduced pressure gave a brown oil which was dissolved in petroleum ether (b.p. 90–100°) and chromatographed on activated alumina. Development with petroleum ether eluted a small amount (1.33 g.) of a yellow oil. Further development with a 10% solution of chloroform in petroleum ether yielded from the least strongly adsorbed fraction, after extrusion of the column and elution with chloroform, a crystalline product contaminated with traces of oil. The oil was washed from the crystals with a small amount of cold petroleum ether (b.p. 40–60°) to yield 1.86 g. (26%) of almost pure di-*t*-butyl benzylmesitoylmalonate, m.p. 104.5–107.5°. Three recrystallizations from petroleum ether (b.p. 90–100°) gave glistening white crystals, m.p. 106.8–107.6°.

Anal. Calcd. for $C_{28}H_{36}O_5$: C, 74.31; H, 8.02. Found: C, 74.39; H, 7.98.

Mesityl β-Phenylethyl Ketone.—A solution of 4.60 g. (0.01 mole) of di-*t*-butyl benzylmesitoylmalonate in 35 ml. of propionic acid containing a trace of anhydrous *p*-toluenesulfonic acid was refluxed for about ten hours, and 95% of the calculated amount of gas was evolved. The reaction mixture was worked up as described above to yield 2.13 g. (84% yield) of mesityl β-phenylethyl ketone, b.p. 140–141° (0.05 mm.), $n^{25}D$ 1.5547 (reported[16] b.p. 167–169° (1.5 mm.), $n^{25}D$ 1.5520.)

Anal. Calcd. for $C_{18}H_{20}O$: C, 85.67; H, 7.98. Found: C, 85.42; H, 7.99.

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On the Infrared Spectrometry of N^{15}-Labeled Phthalyl Glycine Ethyl Ester

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The analysis of deuterium-containing compounds by means of infrared spectrometry has been described recently.[1,2] In the course of a study on the spectra–structure correlation in simple peptides, we observed, that phthalyl glycine ethyl ester labeled with N^{15} exhibited a characteristic shift of its spectrum to the right in the region, from 1430 to 1350 cm.$^{-1}$ if compared to the N^{14} control (see graph).

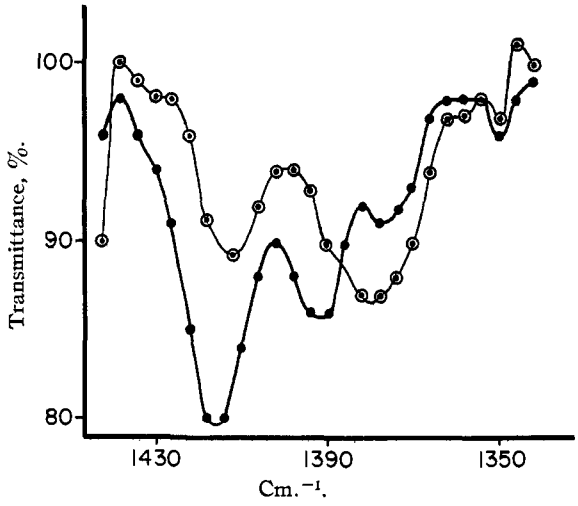

Fig. 1.—Absorption for 5 mg. of phthalyl glycine ethyl ester dissolved in 1 ml. of CCl_4, examined at one-half maximum gain: ●, N^{14} containing compound; ⊙, N^{15} containing compound.

Hence, especially in physiological investigations, infrared spectrometry may be of value in the detection and identification of compounds labeled with N^{15}.

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