tane with phosgene and reacting the resulting isocyanate with aniline.

Prolonged Hydrogenation of 2-Isocyanatomethylbicyclo-[2.2.1]heptane. The above experiment was repeated, with the exception that the catalyst used was 5% palladium-on-alumina, and the reduction was continued until the hydrogen uptake decreased to a rate of less than 1 p.s.i. in two hours (Figure 1). The total quantity of hydrogen consumed was approximately 1.26 mole per mole of olefin, or 0.26 mole more than the quantity required to saturate the double bond. Following removal of the catalyst by filtration and the solvent by evaporation, the liquid residue was refined by vacuum distillation, as described above. Two liquid fractions and a solid kettle residue were obtained. Fraction 1, representing about 61% of the kettle charge, boiled from 56° to 128° C. at 1.4 to 1.0 mm. and was identified as principally the saturated isocyanate (IIIB). Fraction 2, representing about 26% of the total charge, boiled at 132° C. at 0.85 mm. (n^{30}_D, 1.5002) and was indicated by infrared spectrum (amide C=O bands at 6.0 and 6.5 microns and an N—H at 3.05 microns) and microanalysis to be *N*-(2-norbornylmethyl)-formamide.

Anal. Calcd. for $C_9H_{15}NO$: C, 70.59; H, 9.80; N, 9.15.
Found: C, 70.73; H, 9.75; N, 9.10.

The distillation residue, comprising about 13% of the kettle charge, was an impure yellow solid melting from 105° to 153° C. Two recrystallizations from ethyl acetate with decolorizing charcoal present afforded a white crystalline product with a melting point of 176–8° C. Admixture of this product with an authentic sample of *N,N'*-(2-norbornylmethyl) urea caused no depression in melting point.

Anal. Calcd. for $C_{17}H_{28}N_2O$: C, 73.83; H, 10.15; N, 10.15.
Found: C, 73.84; H, 10.22; N, 9.90.

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Alkyl Benzyl Ketones and Hydantoin Derivatives

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Ten alkyl benzyl ketones were prepared by the interaction of phenylacetyl chloride and dialkyl cadmium. Hydantoin derivatives of these ketones were prepared.

TEN ALKYL BENZYL KETONES were synthesized by the interaction of phenylacetyl chloride and the requisite dialkyl cadmium, the synthesis being modeled after a published procedure (*2*). Hydantoin derivatives were prepared from these ketones by the method of Henze and Speer (*3*).

Table I lists the yields of the alkyl benzyl ketones prepared by means of dialkyl cadmiums as well as data on the hydantoin derivatives.

EXPERIMENTAL

Reactants were obtained commercially and used without further purification. Elemental analyses were performed by Huffman Microanalytical Laboratories, Wheatridge, Colo. Melting points were determined in a silicone oil bath and are corrected. The following examples illustrate the synthesis of the alkyl benzyl ketones and the 5-alkyl-5-benzylhydantoins.

1-Phenyl-2-heptanone. A mixture of 40 ml. of anhydrous ether and 6.1 grams (0.25 gram-atom) of magnesium was stirred under reflux while 37.8 grams (0.25 mole) of pentyl bromide in 140 ml. of anhydrous ether was added over a 3-hour period; stirring under reflux was continued for an additional hour. The reaction mixture was cooled with an ice bath and 22.4 grams (0.134 mole) of powdered anhydrous cadmium chloride was added over a 5- to 10-minute period, warmed to room temperature, and refluxed on a steam cone for 1 hour. Ether was removed by distillation on a steam cone. To the residue was added 100 ml. of anhydrous benzene and the distillation was continued until about 50 ml. more of distillate was collected. Again 100 ml. of anhydrous benzene was added, the flask was cooled in an ice bath, and 30.9 grams (0.2 mole) of phenylacetyl chloride in

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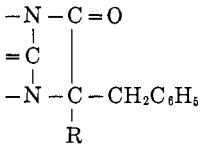
Table I. Yields of Alkyl Benzyl Ketones and Hydantoin Derivatives

Alkyl Benzyl Ketones, $C_6H_5CH_2COR$			5-Alkyl-5-benzylhydantoins, 			
					Analysis, % N	
R	% yield	B.p., ° C./mm.	M.p., ° C.	% yield	Calcd.	Found
CH_3[a]	58	74–76/3	227–28[k]	65		
C_2H_5[b]	51	78/3	218–19[l]	54		
n-C_3H_7[c]	58	97/3	205–06	70	12.06	12.02
Iso-C_3H_7[d]	41	83/2	243–44	42	12.06	12.15
n-C_4H_9[e]	60	97/2	211–12	71	11.37	11.22
Iso-C_4H_9[f]	69	90/2	251–52	64	11.37	11.39
sec-C_4H_9[g]	48	82/2	220–22	25	11.37	11.41
n-C_5H_{11}[h]	76	103/2	203–04	95	10.76	10.96
Iso-C_5H_{11}[i]	65	99/2	248–49	73	10.76	10.90
n-C_6H_{13}[j]	68	120/2	199–200	99	10.21	10.16

[a] 102.5°/19 mm. (*10*). [b] 113.5°/17 mm. (*10*). [c] 243.5–44.0°/757 mm. (*10*). [d] 113–16°/14.5 mm. (*7*). [e] 130–31°/12 mm. (*5*). [f] 250.5°/76 mm. (*8*). [g] 127–28°/17 mm. (*9*). [h] 121–22°/14 mm. (*6*). [i] 267° (*1*). [k] 130–31°/1 mm. (*6*). [k] 227–28° (*4*). [l] 217–18 (*4*).

75 ml. of anhydrous benzene was added with stirring over a period of approximately 10 minutes. The reaction mixture was warmed to room temperature and refluxed with stirring on a steam cone for 1 hour. The flask was again cooled in an ice bath and the reaction mixture decomposed by the addition of a solution of 25 grams of ammonium chloride in 200 ml. of cold water. The organic phase was separated, washed, and dried over anhydrous sodium sulfate. The benzene was removed by flash distillation and the ketone distilled under reduced pressure. There was thus obtained 28.9 grams (76%) of 1-phenyl-2-heptanone, b.p. 103° (2 mm.).

5-Benzyl-5-pentylhydantoin. A mixture of 4.5 grams (0.04 mole) of ammonium carbonate, 1.3 grams (0.02 mole) of potassium cyanide, and 1.0 gram (0.0038 mole) of 1-phenyl-2-heptanone in 50 ml. of a 50% ethanol-water solution was placed in a flask fitted with an air condenser. The flask was placed in a 60° to 70° water bath for 4 hours, carefully acidified with concentrated hydrochloric acid, and cooled, whereupon the hydantoin precipitated and was removed by filtration. After recrystallization from a 50% ethanol-water solution there was obtained 1.3 grams (95%) of 5-benzyl-5-pentylhydantoin, m.p. 203–04°.

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