bromine was completed, 49 g. (0.50 mole) of maleic anhydride was added and the stirring was continued for one hour while the mixture was still immersed in the dry ice-methanol bath. The flask was then removed and, as the solution warmed to room temperature, it turned dark red. After two hours of boiling under reflux, the ether was evaporated, leaving a gummy red solid. Recrystallization removed the heavy red oil that resulted from decomposition of the bromination product. From acetic anhydride, the solid product, ***sym*-octahydrophenanthrene-9,10-dicarboxylic anhydride (IV)**, separated as long colorless needles; from ethyl acetate as colorless platelets, m. p. 312° (Maquenne block) (lit., 310°).[5] The over-all yield was 10 g. (16%).

Anal. Calcd. for $C_{16}H_{16}O_3$: C, 74.98; H, 6.29; mol. wt., 256; sap. eq., 128. Found: C, 75.12; H, 6.45; mol. wt., 285; sap. eq., 130.

The identity of the anhydride (IV) was further established by decarboxylation with soda lime followed by dehydrogenation with selenium to yield phenanthrene, which was identified by melting point and mixed melting point.

From an experiment identical with the above except that the bromine was omitted, the starting materials, 2-cyclohexylidenecyclohexanone and maleic anhydride, were recovered.

NOYES CHEMICAL LABORATORY
UNIVERSITY OF ILLINOIS
URBANA, ILLINOIS
RECEIVED AUGUST 7, 1943

Identification of Alkylbenzenes. III. The Acetamino and Benzamino Derivatives of Isobutylbenzene

BY V. N. IPATIEFF AND LOUIS SCHMERLING

In previous papers[1] the characterization of all but one of the monoalkylbenzenes from toluene through the eight amylbenzenes was described. The mono- and diacetamino and monobenzamino derivatives of the exception, isobutylbenzene, have since been prepared and are reported here in order to make the series complete.

Isobutylbenzene was prepared in 30% yield by the Fittig reaction between phenyl bromide and isobutyl bromide and in 10% yield by the reaction between benzylmagnesium chloride and isopropyl bromide (Table I). That very little or no isomerization occurred during the reactions was shown by the fact that both preparations yielded identical derivatives.

The derivatives were prepared by the procedure described in the previous papers. Their properties are given in Table II. Both the mono- and the diaminoisobutylbenzene could be isolated from a single nitration product, separation being effected as usual by extracting the tin salt of the monoamine with ether.

TABLE I

PREPARATION OF ISOBUTYLBENZENE

Method	Reactants, g.	Conditions Temp.	Conditions Time, hr	Isobutylbenzene Yield, g.	Isobutylbenzene B. p., °C.	Isobutylbenzene $n^{20}D$
A	C_6H_5Br,[a] 31; *i*-C_4H_9-Br, 27; Na, 9	Room	24	8	164–170	1.4912
B	$C_6H_5CH_2MgCl$,[b] 75; *i*-C_3H_7Br, 62	Reflux	120	7	166–170	1.4891

[a] Dissolved in 50 cc. benzene. [b] Dissolved in 275 cc. ether.

TABLE II

DERIVATIVES OF ISOBUTYLBENZENE

Derivative	M. p., °C. (uncor.)	Crystalline appearance	Nitrogen, % Calcd.	Nitrogen, % Found
i-$C_4H_9C_6H_4NHCOCH_3$[a]	127–128[b]	Pearly flakes	7.33	7.23
i-$C_4H_9C_6H_3(NHCOCH_3)_2$	210–211[c]	White	11.29	11.54
i-$C_4H_9C_6H_4NHCOC_6H_5$	128–129[d]	Pearly flakes	5.53	5.52

[a] Hickinbottom and Preston, *J. Chem. Soc.* 1566 (1930); see, also, Hennion and Auspos, THIS JOURNAL, **65**, 1605 (1943). [b] Mixed m. p. (1:1 mixture) with monoacetamino-*s*-butylbenzene (m. p. 126°), 108–112°. [c] Mixed m. p. (1:1 mixture) with diacetamino-*t*-butylbenzene (m. p. 210°) 190–195°; with diacetamino-*n*-butylbenzene (m. p. 214°), 204–206°. [d] After recrystallization from hexane. The crude derivative was rather difficultly purified. Recrystallization from alcohol usually yielded material melting at about 118–122°.

RESEARCH LABORATORIES
UNIVERSAL OIL PRODUCTS COMPANY
RIVERSIDE, ILL.
RECEIVED SEPTEMBER 4, 1943

---

[1] Ipatieff and Schmerling, THIS JOURNAL, **59**, 1056 (1937); **60**, 1476 (1938).

The Preparation of 4′-Aminobenzanilide

BY CARL E. SPENCER

During recent synthetic work, 4′-aminobenzanilide was required. Methods previously described for its preparation using tin and hydrochloric acid[1] or iron and acetic acid[2] proved unsatisfactory because of undesirable by-products. It was found that it could be prepared readily in excellent yield and high purity from 4′-nitrobenzanilide by reduction with hydrogen using platinum black as a catalyst.

The 4′-nitrobenzanilide was prepared from *p*-nitroaniline (Eastman Practical grade) by the method of Lettré and Barnbeck[3] with a yield of 72%, m. p. 197–198°. Twenty-four grams of it in 100 ml. of ethanol with 1 ml. of 0.1 *M* ferrous sulfate was added to platinum black prepared by the reduction of 0.1 g. of platinum oxide in 100 ml. of ethanol by hydrogen at 50 lb. pressure. The reaction

---

[1] Hübner, *Ann.*, **208**, 295 (1881); Rivier and Zeltner, *Helv. Chim. Acta*, **20**, 691–704 (1937).

[2] Morgan and Micklethwait, *J. Chem. Soc.*, **87**, 933 (1905).

[3] Lettré and Barnbeck, *Ber.*, **71**, 1227 (1938).