acid,[2] m. p. 249–251° (reported melting point 255–256°). This establishes the para relationship of the vinyl and sulfonamide groups, as well as the relationships of the corresponding groups in its two parent compounds, although there is no reason to believe that the crude sulfonyl chloride does not contain appreciable amounts of the other isomers.

Polymerization.—A benzene solution of the monomer after a week in the ice box had deposited a white, powdery polymer which was not appreciably soluble in any of a wide range of solvents such as chloroform, water, alcohol and ethanolamine. When the decanted benzene was evaporated on the steam cone, the residue was found to consist principally of a similarly insoluble polymer from which smaller amounts of soluble polymer were separated by successive extractions with hot dioxane and hot benzene; these soluble portions were left as glassy solids upon evaporation of the solvents. The density of the insoluble polymer was approximately 1.25.

When the solid monomer was heated to 70° for five minutes, it first melted and then polymerized to a yellow, vitreous solid. This polymer had a softening point of 190–210°. It was insoluble in acetone, amyl acetate or benzene swelled in chloroform; and dissolved slowly in nitromethane. The molecular weight of the polymer, calculated from the relative viscosity of the nitromethane solution by means of the Kemp–Peters–Staudinger equation,[3] was 20,900.

Anal. Calcd. for $C_{10}H_{13}O_2SN$: C, 56.8; H, 6.2; N, 6.6; S, 15.2. Found: C, 55.8; H, 6.0; N, 6.2; S, 15.4.

Copolymerization with Butadiene.—The monomer copolymerized readily with butadiene in the following formula.

Butadiene	15.0	g.
p-N,N-Dimethylsulfonamidostyrene	5.0	
Water	36.0	
Soap[a]	1.0	
Potassium persulfate	0.06	
OEI[b]	0.10	

[a] Procter and Gamble Soap Flakes (silica free). [b] A commercial product containing a mixture of straight chain mercaptans of 10–16 carbon atoms.

When this emulsion recipe was placed in a four-ounce polymerization bottle and rotated end over end for six hours at 50°, a 76% conversion of copolymer was obtained. This copolymer was 84% soluble in benzene, and its dilute benzene solution had an intrinsic viscosity of 1.59. Elementary analysis of the copolymer indicated that it contained 29.4% by weight of *p*-N,N-dimethylsulfonamidostyrene. The calculated *alpha*-value[4] of 2.96 indicates that the substituted styrene entered the copolymer much more rapidly than did butadiene.

Anal. Calcd. for 29.4% substituted styrene in copolymer: C, 79.5; H, 9.7; N, 2.0; S, 4.5. Found: C, 79.6; H, 9.4; N, 2.0; S, 4.5.

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(2) de Jong, *Verslag Akad. Wetenschappen Amsterdam*, **32**, 14 (1923).

(3) A. R. Kemp and H. Peters, *Ind. Eng. Chem.*, **34**, 1097 (1942).

(4) F. T. Wall, THIS JOURNAL, **63**, 1862 (1941).

NOYES CHEMICAL LABORATORY
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RECEIVED APRIL 5, 1947

Some *s*-Butyl- and 1,4-Di-*s*-butylbenzene Derivatives

BY D. I. LEGGE[1]

A number of new derivatives of *s*-butyl- and 1,4-di-*s*-butylbenzene have been prepared. 1,4-Di-*s*-butylbenzene-2-sulfonyl chloride, like the corresponding diisopropylbenzene derivative,[2] loses an alkyl group on nitration. There is evidence pointing to partial loss of a *s*-butyl group when 1,4-di-*s*-butylbenzene reacts with chlorosulfonic acid, since after hydrolysis of the resulting sulfonyl chloride, the second crop of sulfonic acid sodium salt contains a rather high per cent. of sodium.

Experimental Part

Nitration of sulfonyl chlorides was performed with a large excess of 96% nitric acid, at 1 to 4° for thirty minutes, finally warming in the atmosphere to 25° and pouring immediately onto ice. Other derivatives were prepared by the methods already described.[3]

***s*-Butylbenzene-4-sulfonanilide.**[4]—The oily crude product (87%) was triturated with petroleum ether and recrystallized from the same solvent. It gave white crystals (25%), m. p. 65.0–66.0°.

Anal. Calcd. for $C_{16}H_{19}O_2NS$: C, 66.4; H, 6.6. Found: C, 66.6; H, 6.5.

2-Nitro-*s*-butylbenzene-4-sulfonamide.—The pale yellow nitrosulfonyl chloride, an oil (61%), gave a slightly yellow sulfonamide (95%). This gave a red solution in 5% isopropyl alcohol from which it crystallized as silvery white flakes, m. p. 111.2–112.0°.

Anal. Calcd. for $C_{10}H_{14}O_4N_2S$: C, 46.5; H, 5.5. Found: C, 46.7; H, 5.4.

1,4-Di-*s*-butylbenzene-2-sulfonic Acid Sodium Salt.—The pale yellow liquid sulfonyl chloride (40 g.) was hydrolyzed with 100 ml. of 20% sodium hydroxide, some insoluble material, probably a sulfone, was filtered off, and the sodium salts were crystallized in two fractions; (a) 92%, (b) 8%.

Anal. Calcd. for $C_{14}H_{21}O_3SNa$: Na, 7.8. Found: (a) Na, 7.8. Calcd. for $C_{14}H_{21}O_3SNa \cdot 4H_2O$: H_2O, 19.7. Found: (a) H_2O, 19.8.

Both (a) and (b) were reconverted to sulfonyl chlorides using phosphorus pentachloride, and other derivatives were prepared from (a).

Anal. Calcd. for $C_{14}H_{21}O_3SCl$: Cl, 12.3. Found: Cl (by hydrolysis) (a), 12.3; (b), 12.6.

Sulfonamide.—The crude product (85%) from 16 g. of sulfonyl chloride crystallized slowly from the initially formed oil. Recrystallization from a large volume of 1% isopropyl alcohol gave flat, white plates (57%), m. p. 63.2–64.1°.

Anal. Calcd. for $C_{14}H_{23}O_2NS$: C, 62.4; H, 8.5. Found: C, 62.5; H, 8.4.

Sulfonanilide.—The crude oily solid (91%) from 14 g. of sulfonyl chloride, gave 66% of white crystals, m. p. 105.4–106.3° when recrystallized from 90% isopropyl alcohol.

Anal. Calcd. for $C_{20}H_{27}O_2NS$: C, 69.6; H, 7.9. Found: C, 69.5; H, 7.8.

4-Nitro-*s*-butylbenzene-2-sulfonyl Chloride.—Nitration of 1,4-di-*s*-butylbenzene-2-sulfonyl chloride gave 89% of white oily solid which turned yellow on standing. It crystallized from petroleum ether in buff colored plates (38%), m. p., 71.4–72.0°.

Anal. Calcd. for $C_{10}H_{12}O_4NSCl$: Cl, 12.8. Found: Cl (by hydrolysis), 12.8.

Sulfonamide.—White flakes (91%) from isoöctane, m. p. 171.4–171.7°.

Anal. Calcd. for $C_{10}H_{14}O_4N_2S$: C, 46.6; H, 5.5. Found: C, 46.4; H, 5.3.

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[1] Present address: Anglo Transvaal Consolidated Investment Co., Ltd., Johannesburg, South Africa.

(2) Newton, THIS JOURNAL, **65**, 2439 (1943).

(3) Legge, *ibid.*, **69**, 2078, 2086 (1947).

(4) For orientation, *cf.* Huntress and Autenrieth, *ibid.*, **63**, 3446 (1941).

Sulfonanilide.—White crystals (89%) from isoöctane, m. p., 133.5–134.2°.

Anal. Calcd. for $C_{16}H_{18}O_4N_2S$: C, 58.0; H, 5.3. Found: C, 57.8; H, 5.1.

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RECEIVED JULY 25, 1946

Production of Radioactive Carbon Monoxide from Barium Carbonate

BY JOSEPH T. KUMMER[1]

It has been found possible to prepare conveniently a supply of radioactive carbon monoxide of a specific activity and sufficient in quantity for use in the study of many catalytic reactions. This is done by exchanging, over a hot tungsten filament, the C^{14} in a small amount of carbon dioxide obtained from barium carbonate of high specific activity (about 0.5% of the carbon in the barium carbonate was C^{14}) with the carbon in a large amount of normal carbon monoxide. Since the apparatus and procedure are extremely simple, and since such exchange experiments have not previously been described for a tungsten surface,[2] they are described below in conjunction with Fig. 1. It is hoped that the method will be useful to those wishing to employ carbon monoxide for tracer studies.

The system is completely evacuated, before the exchange run, to 10^{-5} mm. or better. Then, with stopcock A closed, the bulb containing 85% H_3PO_4 is rotated and the acid allowed to react with a few mg. of the radioactive barium carbonate. When stopcock A is opened, the carbon dioxide is allowed to pass through the Dry Ice trap into trap X, cooled in liquid nitrogen. The acid–carbonate mixture is evacuated and warmed to drive all of the carbon dioxide out of the solution and into trap X. This carbon dioxide is next allowed to evaporate into the electric light bulb and is diluted with the required amount of carbon monoxide, manometer C being used for estimating approximately the amount of carbon monoxide added. If the light bulb is run at 60–80 volts overnight (sixteen hours) the exchange will be complete. No experiments have been made as to the rate of exchange or length of time it would take if the bulb were run at 110 volts. Carbon filament bulbs were originally tried but were found to be unsatisfactory because their filaments burned out in an atmosphere of carbon monoxide within a few hours. After a year of use, the tungsten filament showed no deterioration. When the exchange is complete, the radioactive carbon monoxide is pumped into the storage reservoir by means of a Töpler pump through a liquid nitrogen trap, to remove the carbon dioxide.

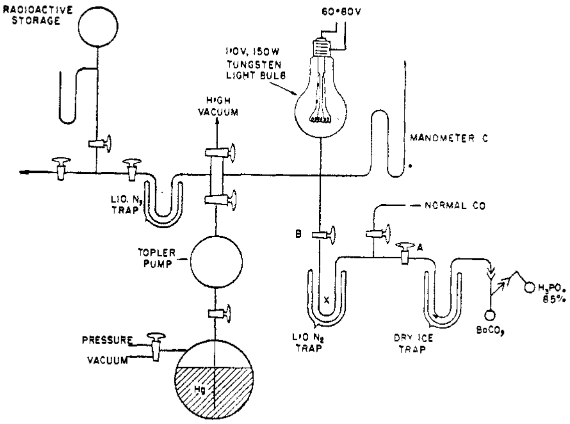

Fig. 1.

Below are data for a particular test run using a new, 150-watt, 110-volt Westinghouse tungsten filament light bulb.

A sample of 2.6 mg. of BaC^*O_3[3] was taken; it was capable of producing approximately 10^7 disintegrations per minute. The C^*O_2 in trap X was flushed into the light bulb by 200 cc. of normal carbon monoxide (to a total pressure of about 300 mm.) after the liquid nitrogen was removed from trap X. After the C^*O_2 and carbon monoxide had been mixed by a few strokes of the Töpler pump, a 1-cc. sample was removed for analysis (*without* interposing a liquid nitrogen trap); this sample showed 42,000 disintegrations per minute per cc. of gas.

A similar sample taken *through* a liquid nitrogen trap for removing carbon dioxide showed 190 disintegrations per minute per cc. of gas.

After the filament in the bulb had been operated at 70 volts for sixteen hours, a 1-cc. sample removed *through* a liquid nitrogen trap showed a count of 42,100 disintegrations per minute per cc. of gas. Apparently, therefore, the exchange over the tungsten filament was complete in this period of time.

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[1] Gulf Research & Development Company Fellowship, Mellon Institute of Industrial Research, Pittsburgh, Pa.

[2] Brandner and Urey, *J. Chem. Phys.*, **13**, 351 (1945), have studied the kinetics of C^{13} exchange between CO and CO_2 over quartz, Au, and Ag.

[3] An asterisk is used to designate C^{14}.

RECEIVED JULY 10, 1947

Thiophene-Containing Antihistaminic Agents

BY L. P. KYRIDES, F. C. MEYER AND F. B. ZIENTY

The high order of antihistaminic activity recently reported for the thiophene analog (I)[1,2,3] of Pyribenzamine was observed[4] on (I) prepared in this Laboratory and tested prior to Dr. Weston's disclosure. In addition, several other

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[1] Weston, THIS JOURNAL, **69**, 980 (1947).

[2] Clapp, Clark, Vaughan, English and Anderson, *ibid.*, **69**, 1549 (1947).

[3] Roth, Richards and Shepperd, *Federation Proc.*, **6**, 366 (1947).

[4] Lee, Dinwiddie and Chen, *J. Pharmacol. Exptl. Therap.*, **90**, 83 (1947).