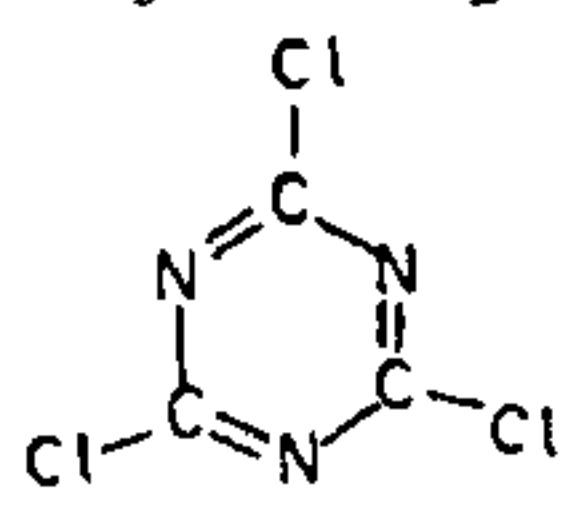


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Table 1 *p*-Toluene sulphones 3 from reagent 1 and alkyl halides 2

Alkyl halide 2	Reaction time (hr)	Yield (%)	M. P. (°C)
CH ₃ -I	6	98	89
CH ₃ -CH ₂ -I	7	90	56
CH ₃ -CH ₂ -CH ₂ -I	6	95	51
CH ₂ =CH-CH ₂ -Br	2	89	54
C ₆ H ₅ -CH ₂ -Cl	5	99	92
C ₆ H ₅ -CO-CH ₂ -Br	2	98	108
<i>p</i> -Br-C ₆ H ₄ -CO-CH ₂ -Br	2	95	105
C ₂ H ₅ O-CO-CH ₂ -Cl	10	94	90
CH ₃ -CO-CH ₂ -Cl	5	90	74
	10	92	78

POLYMER SUPPORTED REAGENTS: AN IMPROVED SYNTHESIS OF SULPHONES USING ANION EXCHANGE RESINS

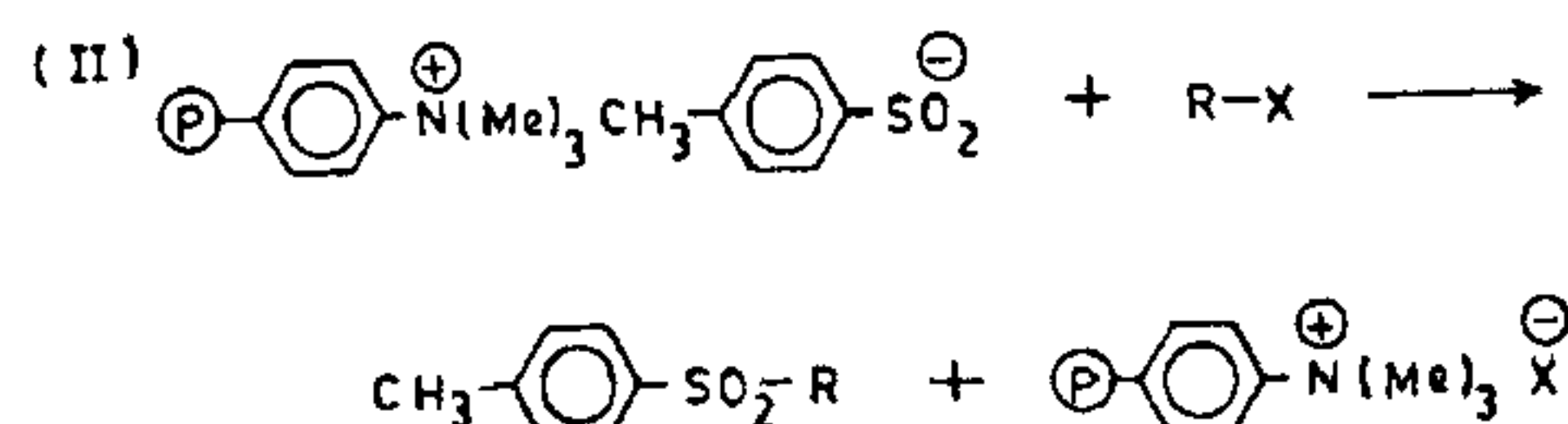
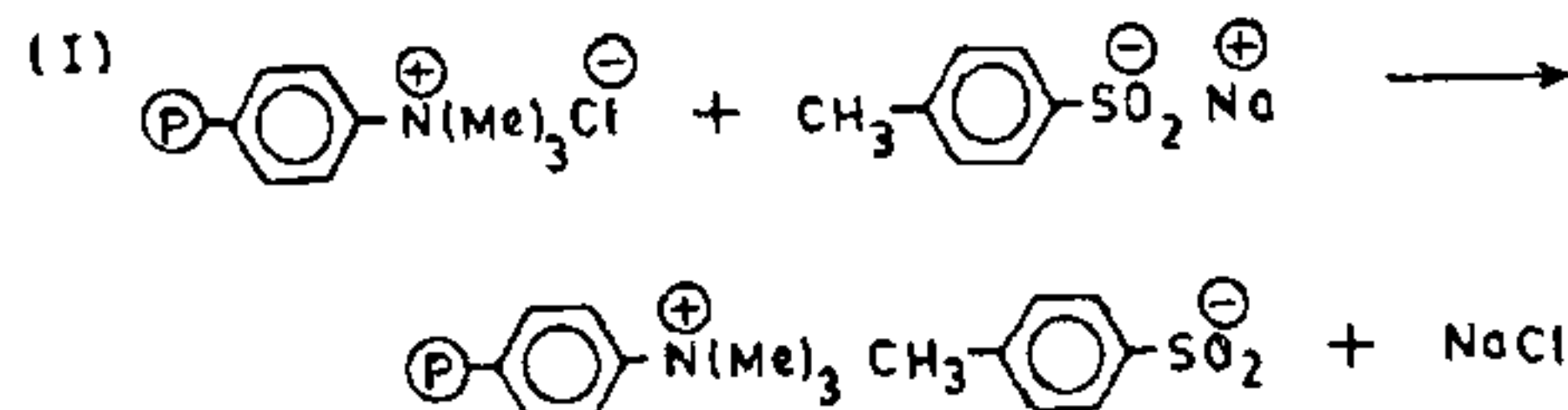
J. G. DESHMUKH, M. H. JAGDALE,
R. B. MANE, M. M. SALUNKHE and
P. P. WADGAONKAR*

Department of Chemistry, Shivaji University,
Kolhapur 416 004, India.

*Polymer Division, National Chemical Laboratory,
Pune 411 008, India.

SULPHONES are important drug intermediates¹ and are generally prepared either by oxidation of the corresponding sulphides or by a displacement reaction using sodium sulphinate as the nucleophile. A drawback of the former method is that the foul smelling thiols are the basic starting materials while the sulphinate method requires rather long reaction times and usually results only in moderate yields of sulphones^{2,3}.

Recently the procedure has been modified by using tetrabutylammonium-*p*-toluenesulphinate in the nucleophilic displacement reaction with organic halides⁴ which gives satisfactory yields. However, the preparation of tetrabutylammonium-*p*-toluenesulphinate from sodium-*p*-toluenesulphinate and tetrabutylammonium bromide was not effective, since a two-fold excess of sodium-*p*-toluenesulphinate was required and the reagent effectively used in



the alkylation reaction was only 50% pure.

As part of our work to demonstrate the applicability of polymer supported reagents in organic synthesis⁵⁻⁷ we now report a more convenient method for the synthesis of sulphones by alkylation of *p*-toluenesulphinate anion supported on IRA 400, a strong anion exchange resin containing the quaternary ammonium group. The products obtained in high yields were essentially pure and characterized by their NMR, IR and comparison with authentic samples.

A typical procedure for the preparation of Amberlite IRA 400 *p*-toluenesulphinate: A 0.25 molar aqueous solution of the sodium salt of *p*-toluenesulphinic acid was slowly percolated through a column filled with Amberlite IRA 400 in chloride form, until a negative test for chloride ion in the eluate was obtained. The resin was then

successively washed with water, ethanol and ether and was finally dried in vacuo at 50°C over P₂O₅ for 10 hr. The exchange capacity was determined by passing aqueous 1 M sodium chloride solution (100 ml) through the resin (0.3 g) in a column. The amount of sulphinate in the eluent was titrated with 0.01 N hydrochloric acid using methyl orange as indicator.

A typical procedure for synthesis of methyl phenyl sulphone: Amberlite IRA 400 *p*-toluenesulphinate form 5 g; (capacity 1 mmol sulphinate anion/g of dry resin was stirred with alkyl halide (4.9 mmol) in refluxing benzene (15 ml) for 6 hr. The resin was then filtered off, washed with dichloromethane and the solvent was removed in vacuo. Distillation of the crude product gives the sulphone.

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GLYCOGEN METABOLISM IN LIVER IN PERCHLORATE-TREATED RATS

P. SANGAN and D. B. MOTLAG

Department of Biochemistry, University of Madras, Madras 600 025, India.

PERCHLORATE is one of the toxic effluents of Space Research Centre. Perchlorate as potassium or

ammonium salts caused decreased food intake and loss of weights in rats¹⁻³. It was observed in our laboratory⁴ that the blood glucose level decreased significantly in perchlorate-treated rats when compared to control rats. It was also observed that the specific activities of aldolase and lactate dehydrogenase were increased whereas glucose-6-phosphatase activity decreased in perchlorate-treated rats⁴. Subsequent to these findings it is proposed to study the glycogen metabolism in liver of rats treated with perchlorate. The muscle glycogen synthetase and muscle phosphorylase have also been estimated, both in the control and in the perchlorate-treated rats to find out the alteration if any in these parameters.

Weanling male albino rats derived from the Wistar strain were purchased from Veterinary College, Madras. Rabbit liver glycogen, phosphoenolpyruvate, uridine 5'diphosphoglucose, uridine 5'diphosphate were obtained from Sigma Chemical Company, St. Louis, MO, USA. Glucose-1-phosphate and glucose-6-phosphate were purchased from centre for biochemicals (CSIR), V. P. Chest Institute, Delhi. Bovine serum albumin was the product of Fluka Buchs, SG, Switzerland. Cysteine hydrochloride, dinitrophenyl hydrazine, ethylene diamine tetra acetic acid were obtained from British Drug House, Poole, England. All other chemicals used were of analytical grade. Pyruvate kinase used in the present investigation was prepared from rabbit muscle according to the method of Davidson⁵.

The animals were fed with commercial rat feed with paired feeding and water *ad libitum* along with an oral administration of a chronic dosage (500 mg/kg body weight/day) of potassium or ammonium perchlorate for 45 days. The dosage was selected based on the report of Spreca *et al*⁶. The animals were then sacrificed by stunning and decapitating. The liver, kidney and muscle were dissected out immediately and a portion of the liver was kept in Rossman's fluid⁷ for histochemical studies of glycogen with periodic acid Schiff's reagent⁸. Another portion of the liver and the kidney were used for the estimation of glycogen according to the method of Morales *et al*⁹. The rest of the liver and a portion of muscle were washed with ice cold saline and appropriate amounts of the tissues were homogenized in 0.1 M Tris-HCl buffer pH 7.4. The homogenates were centrifuged at 2500 rpm for 10 minutes at 4°C. The supernatants were used to