

strated by isolation and synthesis by alternative routes the nature of the coupling products in several instances and established the structural requirements for the occurrence of the reaction. Polonovski and Pesson have usefully extended these observations, but have failed to mention the considerable use to which the reaction already has been put in synthetic work.^{4,5,6,7,8}

Experimental

2-(*p*-Chlorophenylthio)-4-hydroxypyrimidine.—Five grams of *p*-chloroaniline in 100 ml. of *N* hydrochloric acid was diazotized with 3.0 g. of sodium nitrite at 0°. Seven and one-half grams of sodium bicarbonate was added to the solution and then the whole was added rapidly with stirring to a solution of 5 g. of 2-thiol-4-hydroxypyrimidine in 4 l. of water. When the addition of the diazonium solution was complete, 10 ml. of a 15% solution of sodium carbonate was added. The solution turned pinkish in color, some gas was evolved and a solid began to form. After standing two hours the solution was neutralized with acetic acid and the solid filtered off. The pinkish powder (5.4 g.), after recrystallization from alcohol containing a little pyridine gave colorless octahedra, m. p. 243–245°.

Anal. Calcd. for C₁₀H₇ON₂SCl: C, 50.4; H, 3.0; N, 11.7. Found: C, 50.4; H, 2.9; N, 11.5.

2-(*p*-Chlorophenylthio)-4-hydroxy-6-methylpyrimidine.—To a solution of 5 g. of 2-thiol-4-hydroxy-6-methylpyrimidine in 3 l. of water was added a diazonium solution prepared from 8.9 g. *p*-chloroaniline (2 moles), followed by 16.6 ml. of a 15% solution of sodium carbonate. After standing for two hours, the solution was neutralized with acetic acid and the reddish solid was filtered off (8.3 g.). After washing with warm ethanol to remove some red material the substance was recrystallized from boiling ethanol. It formed colorless rectangular prisms, m. p. 223.5°.

Anal. Calcd. for C₁₁H₉ON₂SCl: N, 11.1. Found: N, 11.3.

Hydrolysis of *p*-Chlorophenylthioethers with Hydrochloric Acid.—One gram of each of the above compounds was refluxed with 25 ml. of 6 *N* hydrochloric acid for three hours. Steam distillation gave *p*-chlorothiophenol in about 75% yield, m. p. 53–54°; benzoyl derivative, m. p. 74–74.5°. The acid solution on evaporation gave uracil or 6-methyluracil, according to the starting material, in about 80% yield. The compounds were identified by melting points, ultraviolet absorption spectra and analyses.

Anal. Calcd. for C₆H₄N₂O₂: C, 42.8; H, 3.6. Found: C, 42.5; H, 3.6. Calcd. for C₈H₈N₂O₂: C, 47.7; H, 4.8. Found: C, 47.6; H, 4.4.

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(6) Todd, *ibid.*, 647 (1946).

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The Surface Area of Vermiculite

By B. L. HARRIS

The surface area of vermiculite before and after exfoliation was measured by adsorption of nitrogen at -195° by the method of Brunauer, Em-

mett and Teller¹ in order to ascertain whether the area was large enough that the material might be used as a catalyst support. The area of the unexfoliated sample was measured by nitrogen adsorption and found to be 0.52 sq. m./g., giving a calculated average thickness of the platelets of 1.8 microns when the theoretical density of 2.13 is assumed. This indicates that the material was thoroughly fissured before exfoliation.

A sample of the vermiculite was exfoliated by heating for five minutes at 950°, resulting in a silvery-white accordion-like structure. The loss in weight on exfoliation was 19.6%. The surface area of this sample was 10.35 sq. m./g., giving a calculated platelet thickness of 0.091 micron. The structure of vermiculite consists of sheets of (OH)₄Mg₆(Si, Al)₈O₂₀ of 9.26 Å. thickness with alternate layers of 8 H₂O spaced the order of 4.8 Å.² It was hoped that the platelet spacing might approximate this order of magnitude, resulting in a very large surface area. The smaller area, corresponding to a thickness some 200 times as great, agrees with the conclusion of Gruner that the structure collapses above 750° and that exfoliation is mechanical, due to the formation of steam.

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(2) J. W. Gruner, *Am. Mineral.*, 19, 557 (1934).

DEPARTMENT OF CHEMICAL ENGINEERING
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The Interaction of Purified Antibody with Homologous Hapten. Antibody Valence and Binding Constant

By HERMAN N. EISEN¹ AND FRED KARUSH²

As part of a study of the relation between protein structure and specificity we are investigating the properties of soluble complexes formed between purified antibody and homologous haptens. Rabbits were immunized with sheep serum coupled with diazotized *p*-arsanilic acid. Purified antibody, homologous to *p*-azophenylarsonic acid (R), was obtained³ by treating pooled antisera with human erythrocyte stromata coupled with diazotized *p*-arsanilic acid (R-stromata). After several washings with 0.16 *M* sodium chloride, the antibody was eluted from R-stromata by acidification (*pH* 3.8) with acetic acid. The R-stromata was removed by centrifugation and the supernate, which contained the antibody, was neutralized. In the antibody solutions thus prepared at least 90% of the protein was specifically precipitable with R-stromata, in agreement

(1) Research Fellow, National Institute of Health.

(2) Investigation conducted during tenure of a Fellowship in Cancer Research of the American Cancer Society, recommended by the Committee on Growth of the National Research Council.

(3) K. Landsteiner and J. van der Scheer, *J. Exp. Med.*, 63, 325 (1936).