pressure; gas absorption was complete in 2.5 hr. The catalyst was removed by filtration and the solvent evaporated on the steam bath. The residue had $n_D^{25°}$ 1.4790 and $[\alpha]_D^{25°}$ +15.2°. The dibenzylidene derivative had the m.p. 124–125°.

(+)*cis-8-Methylhydrindane* (X). The ketone (IX) (3.5 g.) was refluxed for two hr. at 135° with 4.0 g. of potassium hydroxide, 80 ml. of diethyleneglycol, and 7.0 g. of 85% hydrazine hydrate. The water was distilled off and then the temperature was raised to 200° for 4 hr. The mixture was cooled, taken up in water, extracted with pentane, and the pentane layer washed with concentrated sulfuric acid until the acid no longer became colored. The pentane solution was washed with water, dried over magnesium sulfate, and the pentane removed, leaving two grams of an oil whose infrared spectrum showed no carbonyl band and was identical with that given in ref. 7. The oil crystallized on cooling, m.p. 5°, $[\alpha]_D^{26°}$ +3° (6% in chloroform).

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A New Synthesis of *cis*-1,2-Cyclohexanediol

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cis-1,2-Cyclohexanediol (I) has found considerable use as a model compound for synthetic and mechanistic studies. In discussing methods of preparing I, Criegee and Stanger[2] have recommended a four-step method starting with cyclohexene. As pointed out by Winstein, Hess, and Buckles,[3] however, its success is dependent on the reaction conditions of the replacement reaction.

We have developed a much simpler two-step synthesis of I from cyclohexene which requires about a day's time and which is amenable to large scale work. The method employed is that developed[4] for *cis*-hydroxylation in the synthetic steroid series. It involves the interaction of an olefin with iodine, silver acetate, and wet acetic acid to give, by way of a neighboring group replacement reaction,[3] *cis*-hydroxy acetate in one operation. Subsequent hydrolysis yields the free diol.

Using this technique, we have obtained after one recrystallization quite pure I in 66% yield. Considerable variation of the reaction conditions did not improve the yield. The use of iodine monochloride in place of iodine decreased the yield of I to 32%.

EXPERIMENTAL[5]

cis-1,2-Cyclohexanediol (I). To a slurry of 16 g. (0.096 mole) of silver acetate in 150 ml. of glacial acetic acid in a three-neck flask equipped with a condenser, thermometer, and stirrer was added 3.42 g. (0.0416 mole) of freshly distilled cyclohexene, b.p. 83–85°. Accompanied by vigorous stirring, 11.7 g. (0.046 mole) of powdered iodine was added over a 30-min. period at room temperature. Finally, 0.67 g. (0.042 mole) of water was added and the reaction mixture was heated with vigorous stirring for 3 hr. at 90–95°. After the reaction mixture was cooled, filtered, and the silver iodide precipitate washed well with hot benzene and ethyl acetate, the combined filtrates were evaporated at the water pump to give a yellow viscous oil which was taken up in methanol and filtered. The filtrate was neutralized with a few ml. of alcoholic potassium hydroxide, treated with 3.5 g. of potassium hydroxide in 20 ml. of methanol and hydrolyzed by refluxing for 1.5 hr. (darkening occurred). After evaporation of the methanol at the water pump, the residue was taken up in 500 ml. of warm diethyl ether and filtered. The filtrate was evaporated yielding 3.92 g. (81%) of crude glycol. Recrystallization from carbon tetrachloride yielded 3.2 g. (66%) of white *cis*-1,2-cyclohexanediol, m.p. 97–98°, lit.[2] m.p. 96–98°.

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[1] Thanks are due the Du Pont Co. for a Summer Faculty Fellowship.

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[5] All melting points are uncorrected.

Some 4-Substituted Thiourazoles[1]

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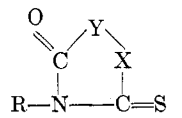
It was shown that 3-substituted rhodanines (I) possess pronounced antimicrobial activity.[3-5] The present work deals with the synthesis of some related 4-substituted thiourazoles (II). The thioura-



I, X = S, Y = CH_2
II, X = Y = NH

$RNH—C(=S)—NHNH—C(=O)—NH_2$

III

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[2] Monsanto Chemical Co. Fellow 1956–1957.

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