semicarbazone prepared from our cyclo-octanone out of cyclohexanone and diazomethane as well as that obtained from Professor Ruzicka.

The reaction of the main portion of this fraction with piperidine did not yield homogeneous derivatives of the piperidino alcohol.

Summary

The action of diazomethane on cyclohexanone in the presence of methyl alcohol yields as the main product cycloheptanone, and in smaller quantities cyclo-octanone and an oxide isomeric with cycloheptanone.

Diazomethane with cyclopentanone leads (through cyclohexanone) to cycloheptanone as chief product, and cyclo-octanone as by-product. In this case, the oxide formed was not identified.

The reaction may be advantageously applied to the preparation of cycloheptanone and cyclo-octanone.

UNIVERSITY, VIRGINIA

NOTES

Note on Catechol Sulfonephthalein.—Catechol sulfonephthalein, first mentioned by Moir,[1] was prepared by substantially the method of Lubs and Clark,[2] condensing at temperatures under 100 two moles of catechol with one mole of symmetrical dichloro derivative of *o*-sulfobenzoic acid, but omitting the use of zinc chloride as condensing agent. The resulting product was analyzed for sulfur, giving 95.71 and 95.76% of the calcd. for the formula $C_6H_4\begin{cases}C:(C_6H_3(OH)_2)_2\\ :O\\ SO_2\end{cases}$, or 100.40 and 100.45% of the calcd. for the formula $C_6H_4\begin{cases}C:(C_6H_3(OH))_2:O\\ :O\\ SO_2\end{cases}$. The product is amorphous, of a very deep purple color (practically black), solid but not brittle at ordinary temperatures, and moderately hygroscopic. When warmed to about 60° it is softened sufficiently to drop from a small stirring rod. Its solubilities are: miscible in all proportions with water; readily soluble in methanol, ethanol, acetone, glacial acetic acid and ethyl acetate; slightly soluble in ethyl acetoacetate, acetic aldehyde and ethyl ether; insoluble in benzene, toluene, xylene, petroleum ether, carbon disulfide, carbon tetrachloride, chloroform and acetic anhydride. Attempts at crystallization, using the first-named solvents, have been unsuccessful.

The aqueous solution of this product is found to give colors of but a fraction, about one-tenth, of the intensities of colors of other sulfonephthaleins. A noteworthy feature of several of these colors, and one not mentioned by Moir, is their tendency to change, in some cases in only a

[1] J. Moir, *J. So. Afr. Assoc. Anal. Chem.*, **3**, 6 (1920); *C. A.*, **14**, 3607 (1920).

[2] H. A. Lubs and W. M. Clark, *J. Wash. Acad. Sci.*, **5**, 609 (1915).

few minutes. In the following table are listed the colors exhibited by 0.2 cc. of a 0.5% aqueous solution of catechol sulfonephthalein when added to 10 cc. of the solutions listed in the first column.

	Immediate color	After 5 minutes	After 20 minutes	After 1 hour	After 18 hours	After 36 hours
1.0 *N* HCl	Red	Red	Red	Red	Red	Red
0.01 *N* HCl	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow
*P*H 5	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow
*P*H 6	Yellow	Yellow	Yellow	Yellow	Yellow, slightly greenish	Yellowish-green
*P*H 7	Yellow, slightly greenish	Yellow, slightly greenish	Yellow, slightly greenish	Yellow, more greenish	Yellowish-green	Yellowish-green
*P*H 8	Purple, then indigo	Rich blue	Blue	Blue	Pale, inky	Yellowish-green
*P*H 9	Pinkish-purple	Pinkish-purple	Pinkish-purple	Pinkish-purple	Pinkish-purple	Yellowish-green
*P*H 10	Pinkish-purple	Pinkish-purple	Darker, surface cloudy	Less red, somewhat brownish	Light green	Yellowish-green
0.01 *N* NaOH	Indigo, becoming cloudy	Clear, fainter purple	Purple, surface green	Purple, surface green	Grass green	Pale green
1.0 *N*	Grass green	Grass green	Grass green	Grass green	Green, less intense	Pale green

The colors given by products of condensation of mixtures containing larger porportions (2.2 moles, 2.5 moles) of catechol are much the same as those shown in the table, save that the greens are much more intense.

It has been found possible to prepare a satisfactory series of color shades in the range *P*H 0 to *P*H 1.5.

Products condensed at temperatures of 130 and 160° were found to be incompletely soluble in water; these soluble portions give relatively very faint acid colors and comparatively more intense alkaline colors. Acidified aqueous solutions of the low temperature product, on vigorous boiling, showed similar color differences.

The product condensed at the lower temperature appears to be fairly pure, dilute and concentrated aqueous solutions remaining clear for many days, and no material is insoluble in sodium hydroxide[2] or in sodium bicarbonate[3] developing on long standing.

TAKOMA PARK CYRUS B. WOOD
WASHINGTON, D. C.

RECEIVED FEBRUARY 21, 1930
PUBLISHED AUGUST 5, 1930

[3] W. R. Orndorff and F. W. Sherwood, THIS JOURNAL, **45**, 486 (1923).