4,4-Didodecyl-2,6-dimethylmorpholinium Chloride.—A mixture of didodecylamine (35.3 g., 0.10 mole), bis-(1-chloro-2-propyl) ether (20.6 g., 0.12 mole), sodium carbonate (12.7 g., 0.12 mole) and butyl alcohol (200 ml.) was stirred and refluxed for 72 hours. It was cooled, filtered and evaporated under reduced pressure. The residue was dissolved in hot methanol (400 ml.); this solution was chilled in ice and filtered, giving 16.7 g. of unreacted amine. The filtrate was distilled and its residue was crystallized from ethyl acetate, giving 8.8 g. of waxy, white solid (34% yield, 18% conversion). This was recrystallized from ethyl acetate; m.p. (after drying *in vacuo*), 175–180°.

Anal. Calcd. for $C_{30}H_{62}ClNO$: C, 73.79; H, 12.80; Cl, 7.26. Found: C, 73.85; H, 13.03; Cl, 7.18.

2,6-Dimethyl-4,4-dioctadecylmorpholinium Chloride.—A mixture of dioctadecylamine (84.1 g., 0.16 mole), bis-(1-chloro-2-propyl) ether (33.0 g., 0.19 mole), sodium carbonate (20.5 g., 0.19 mole), and butyl alcohol (400 ml.) was stirred and refluxed for 20 hours. It was diluted to one liter with butyl alcohol and filtered while hot. The filtrate was chilled and filtered again, yielding 64.4 g. of unreacted amine. The filtrate was evaporated, leaving 27.0 g. of residue. This was dissolved in hot methanol (400 ml.), cooled, and filtered to remove a small amount of solid. The filtrate was evaporated to dryness. The residue was recrystallized from ethyl acetate, giving 16.3 g. (65.5% yield, 15.3% conversion) of white solid; m.p., after another recrystallization and drying *in vacuo*, 172–175°.

Anal. Calcd. for $C_{42}H_{86}ClNO$: Cl, 5.38; N, 2.13. Found: Cl, 5.31; N, 2.46.

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Vapor Phase Catalytic Isomerization of *m*-Xylene

BY E. R. BOEDEKER AND W. E. ERNER

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The production of certain of the synthetic fibers requires *p*-xylene as one of the starting materials. The separation of *p*-xylene from a xylene mixture by low temperature crystallization yields a high purity *p*-xylene fraction and a fraction containing a preponderance of *m*-xylene. Additional quantities of *p*-xylene are potentially available from the isomerization of the *m*-xylene-containing fraction to a xylene mixture, the composition of which approximates thermodynamic equilibrium. With this in mind, the vapor phase isomerization of a *m*-xylene-rich fraction over a synthetic silica–alumina cracking catalyst (Houdry Type S-45) was cursorily investigated.

The charge stock used in the present study contained 84% *m*-xylene, 9% *p*-xylene, 5% *o*-xylene and 2% ethylbenzene. The charge stock was obtained from a xylene fraction by freezing out a portion of the material.

The experimental conditions and results are summarized in Table I. The products were distilled to obtain a xylene and lighter overhead fraction; the bottoms from the distillation were C_9-aromatics. The overhead fraction from the distillation was analyzed by infrared absorption spectrometry. In all cases, liquid recoveries were in excess of 98% and were corrected to a no-loss basis.

The effect of operation at reduced pressure is very clearly shown. At atmospheric pressure, the isomerization of *m*-xylene proceeds simultaneously with a disproportionation reaction. In the disproportionation reaction, two molecules of xylene apparently are adsorbed on sites which are located sufficiently close to each other to allow a shift of a methyl group, thereby forming toluene and a C_9-aromatic. If, on the other hand, the operating pressure is reduced so that the catalyst surface is more sparsely covered, the isomerization reaction proceeds to the exclusion of the disproportionation reaction.

TABLE I

Conditions				
Temperature, °C.	515	515	515	
Pressure, mm., Abs.	~760	90	90	Charge
Vol. liq. feed/vol. cat./hr.	0.7	0.6	1.2	Stock
Product analyses, mole %				
Benzene	1.2	..	..	..
Toluene	13.0	1.1	1.0	..
m-Xylene	32.3	59.8	60.3	84.0
p-Xylene	18.9	18.1	20.6	9.0
o-Xylene	13.0	9.2	7.0	5.0
Ethylbenzene	5.1	11.5	11.1	2.0
C_9-Aromatics	16.5	0.3	..	..

Doubling the liquid hourly space velocity has no significant effect on the distribution of the xylene isomers.

Data have been published on the equilibrium composition of a xylene mixture.[1] Based on these data, the composition of an equilibrium mixture of xylenes at 515° is as follows: 47% *m*-xylene, 22.5% *o*-xylene, 21.5% *p*-xylene, and 9% ethylbenzene. Comparison of these figures with those obtained experimentally at 90 mm. pressure shows that the products contain approximately equilibrium amounts of *p*-xylene and ethylbenzene; the *m*-xylene content, however, is higher than and the *o*-xylene content lower than the calculated thermodynamic equilibrium.

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Thiosemicarbazones of Some Vanillin Derivatives

BY R. P. PERRY

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The purpose of the work described in this paper was to prepare a series of substituted vanillin thiosemicarbazones which might be useful as antifungal agents and provide some basis for determining the relation of structure to antimicrobic action. Since the introduction of Tibione (acetylaminobenzaldehyde thiosemicarbazone) by Domagk and his associates,[1–3] many changes have been effected in the structure of the molecule, particularly as regards the subordinate groups *para* to the thiosemicarbazone moiety and in only a few cases have thiosemicarbazones with poly-substituted benzenoid residues been studied.[4,5] Moreover, the thiosemicarbazones thus obtained have been screened generally only for their antitubercular activity.

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[3] R. Behnisch, P. Mietzsch and H. Schmidt, *Am. Rev. Tuberc.*, **61**, 1 (1950).

[4] J. Bernstein, H. L. Yale, K. Losee, M. Holsing, J. Martins and W. A. Lott, THIS JOURNAL, **73**, 906 (1951).

[5] D. J. Drain, *et al.*, *J. Pharm. Pharmacol.*, **1**, 784 (1949).