

12. Earle, F. R., Mikolajczak, K. L., Wolff, I. A. and Barclay, A. S. *J. Am. Oil Chemists' Soc.* **41** (1964) 345.
13. Barclay, A. S. and Earle, F. R. *Econ. Botany* **19** (1965) 33.
14. Hegnauer, R. *Chemotaxonomie der Pflanzen*, Birkhäuser, Basel und Stuttgart 1964, Vol. 3, p. 562.
15. *Chem. Centr.* **70 I** (1899) 706; *Jahresbericht, E. Merck* **1898** 25.
16. Allen, E. K. and O. N. *Proc. 25th Biol. Colloq. (1964) on Microbiology and Soil Fertility*, Oregon State University Press, Corvallis 1965, p. 77.

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On the Action of *t*-Butyl Hypochlorite on *m*-Cresol

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In a paper by Ginsburg,¹ the action of *t*-butyl hypochlorite on *m*-cresol is reported. In this reaction, a compound with m.p. 55–56.5°C was obtained and it was stated to be 2-chloro-3-methylphenol since the melting point agreed with that reported by Gibson² in his preparation of the same compound in an unambiguous way.

In our search for a convenient method to prepare 2-chloro-3-methylphenol, we attempted the chlorination of *m*-cresol with *t*-butyl hypochlorite, and after fractionation of the reaction mixture, we also obtained a product with m.p. 57°C. However, as the NMR spectrum indicated, this product was not homogeneous but a mixture of 4- and 6-chloro-3-methylphenol which were separable by recrystallization from petroleum ether.

Gas chromatographic and infrared analysis of the crude chlorination mixture proved the main product to be 4-chloro-3-methylphenol. The mixture contained only about 15% 2-chloro-3-methylphenol. Thus, Ginsburg's statement¹ that the main product obtained by the action of *t*-butyl hypochlorite on *m*-cresol is 2-chloro-3-methylphenol, is not correct.

However, the result is in good agreement with that obtained in the chlorination of *m*-cresol with chlorine.³

Experimental. Melting points were determined on a Kofler hot-stage microscope and are corrected. A Perkin-Elmer model 337 spectrophotometer was used for the IR-absorption measurements. The gas chromatographic analyses were performed on a Perkin-Elmer 800 instrument using a $78 \times \frac{1}{4}$ in column packed with Apiezon L (5%) on HMDS-treated Chromosorb W (80–100 mesh).

Chlorination of m-cresol. This was performed according to the procedure of Ginsburg.¹ After evaporation of carbon tetrachloride and *t*-butanol the crude residue was analysed by gas chromatography giving the following composition: 49% 4-chloro-3-methylphenol, 44% 2- and 6-chloro-3-methylphenol and 7% unreacted *m*-cresol. As the 2- and 6-chloro-*m*-cresols could not be separated by gas chromatography they were determined by infrared analysis using the peaks at 605 and 709 cm⁻¹. The ratio of 2- to 6-chloro-*m*-cresol was about 1:2.

Samples of chloro-*m*-cresols for identification of the peaks in the gas chromatograms and the infrared spectra were obtained as follows:

2-Chloro-3-methylphenol. This compound was prepared by standard methods by the sequence: 2-methylaniline → 2-methyl-6-nitroaniline → 2-chloro-3-nitrotoluene → 2-chloro-3-methylaniline → 2-chloro-3-methylphenol. After recrystallization from petroleum ether the m.p. was 50–51°; lit.² 55–56°.

6-Chloro-3-methylphenol. Prepared from 6-chloro-3-methylaniline by diazotization and replacement of the diazonium group by hydroxyl. Recrystallization from petroleum ether, m.p. 46–46.5°; lit.³ 45–46°.

4-Chloro-3-methylphenol. A commercial product was recrystallized from petroleum ether, m.p. 66°; lit.⁴ 66°.

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1. Ginsburg, D. *J. Am. Chem. Soc.* **73** (1951) 2723.
2. Gibson, G. P. *J. Chem. Soc.* **1926** 1425.
3. Hodgson, H. H. and Moore, F. H. *J. Chem. Soc.* **1926** 2038.
4. Sah, P. P. T. and Anderson, H. H. *J. Am. Chem. Soc.* **63** (1941) 3164.

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