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Titrimetric Determination of Cresols

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A simple, rapid and sensitive titrimetric method with amplification has been worked out for the determination of 50–2000 μg of *o*-cresol, *m*-cresol or *p*-cresol. It is based on bromination of these compounds with bromine water to form the corresponding hypobromites, which liberate an equivalent amount of iodine when treated with iodide. The sensitivity of the method has been increased using the Leipter amplification procedure. The coefficient of variation does not exceed 1.5% for above 500 μg of the cresol, but increases to 2.2% at the 50 μg -level.

KEY WORDS: Cresols, bromination, amplification.

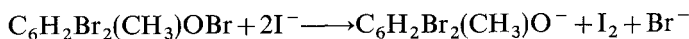
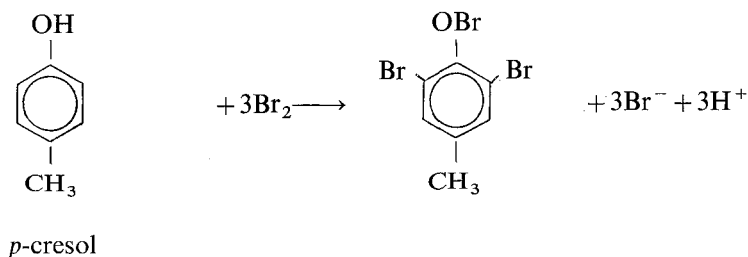
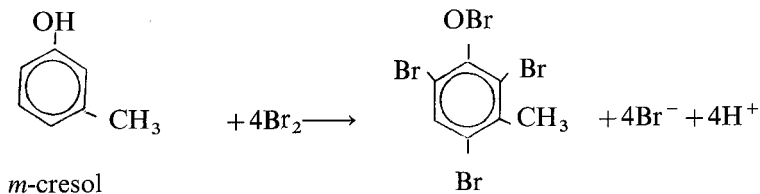
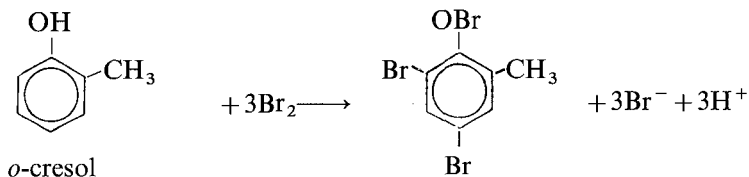
INTRODUCTION

Cresols occur in the middle and heavy oil fractions of coal-tar. They are used for preserving purposes, as disinfectant and in the manufacture of resins and plasticisers.¹ For this reason, their determination is important.

Kolthoff² found that freshly prepared *m*-cresol takes up to 3 bromine atoms, but the *o*-cresol and *p*-cresol seem to be able to substitute 2 atoms of bromine. A bromate-bromide² solution has been used for determination of cresols. A direct titration of *o*-cresol and *p*-cresol with iodine has been reported.³ However, these methods are not sensitive and the bromate-bromide solution is only used in highly acidic medium; while the iodine solution needs frequent standardization.

The present paper describes a method which is based on the bromination of *o*-cresol, *m*-cresol and *p*-cresol with an excess of

saturated bromine solution to form the corresponding hypobromites, which react with iodide (after removal of excess bromine with formic acid) to liberate equivalent amounts of iodine.



In an attempt to increase the sensitivity of the method, the "so-called" Leipert⁴ procedure has been applied, which involves the extraction of the liberated iodine into chloroform, reduction to iodide, followed by oxidation of the latter to iodate by bromine water to give a 6-fold amplification method.

EXPERIMENTAL

Reagents

All chemicals used were of A.R. grade.

Standard solutions of *o*-cresol, *m*-cresol and *p*-cresol, 1 mg/ml of each were prepared in 0.01 N sodium hydroxide solution. Less concentrated solutions were prepared by dilution. The compounds were purified by simple distillation.

Sodium thiosulphate solutions, 0.001 and 0.01 N were standardized against potassium iodate solutions of similar normality.

Solutions of bromine water (saturated), formic acid (80%), potassium iodide solution (5%) and sodium sulphite solution (1%), were also prepared.

Procedure

In a 50-ml separating funnel, place a suitable volume (1–2 ml) of sample solution containing 50–2000 μg of *o*-cresol, *m*-cresol, or *p*-cresol, 10 ml of water and 4 ml of bromine water. Stopper the flask and shake for 5 min (10 min for *o*-cresol) using the electric shaker. Destroy the excess bromine by dropwise addition of formic acid. Add 1 ml of 5% potassium iodide solution, stopper the flask and shake for 5 min. Extract the liberated iodine with two 10 ml portions of chloroform. Collect the extracts in another funnel, and shake with 10 ml of water containing 1 ml of sodium sulphite solution. Transfer the aqueous (upper) layer, containing the iodide, into a 50-ml conical flask, oxidize the iodide with 3 ml of bromine water by shaking for 3 min, then remove the excess of bromine by shaking with 2 ml of formic acid. Add 2 ml of 5% KI solution, and titrate the liberated iodine with 0.01 N thiosulphate solution using starch as indicator (for less than 500 μg of the cresol, use 0.001 N thiosulphate solution). Run a blank determination.

1 ml of 0.01 N thiosulfate = 90.12 μg of *o*-cresol,
m-cresol or *p*-cresol.

RESULTS AND DISCUSSION

The effect of bromination time of cresols was first studied, and it was found that in case of *m*-cresol and *p*-cresol, the bromination goes to completion within about 5 min, while *o*-cresol requires 10 min, which may be due to the steric effect of the methyl group.

Complete bromination of the entitled compounds requires 3–5 ml of saturated bromine water, the excess of which was destroyed by the dropwise addition of formic acid.

Reduction of the hypobromites was achieved using 1–1.5 ml of 5% potassium iodide solution. Higher excesses of iodide retards the extraction of iodine with chloroform. Complete liberation of iodine takes place within about 5 min; the time could be reduced to 2 min using concentrated phosphoric or hydrochloric acid, but the blank value increases by about 2-fold.

The working procedure finally developed was applied successfully to the determination of 50–2000 μg of the cresol. Table I shows the recoveries and the coefficient of variation for the studied compounds.

TABLE I
Titrimetric determination of Cresols.

Compound	Weight, μg		Coefficient of variation, %
	Taken	Found†	
<i>m</i> -Cresol	50	49.1	1.8
	500	498	1.1
	2000	1982	0.6
<i>p</i> -Cresol	50	49.0	2.0
	500	495	1.2
	2000	1980	0.6
<i>o</i> -Cresol	50	48.7	2.2
	500	494	1.5
	2000	1977	0.8

†Average of 5 determinations.

Interferences:

Compounds that could be brominated under the experimental conditions, are expected to interfere (e.g. phenols, toluidines... etc.).

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