

Analysis of Low Condensed Components in Condensates of Mixtures of β -Naphthalenesulfonic and β -Methylnaphthalenesulfonic Acids with Formaldehyde

ABSTRACT

The low condensed components in the condensates of mixtures of β -naphthalenesulfonic acid and β -methylnaphthalenesulfonic acid with formaldehyde were analyzed by high speed liquid chromatography. The main components of monomer and dimer were β -naphthalenesulfonic acid and the dimeric condensate of β -naphthalenesulfonic acid, respectively.

INTRODUCTION

The sodium salt of the condensates of naphthalenesulfonic acids and formaldehyde has been used widely as a dispersing agent for organic and inorganic powders. Previous investigations on the analysis of the condensates have dealt almost exclusively with the condensates of β -naphthalenesulfonic acid (NS) and formaldehyde. Paper chromatography (1), gel chromatography (2), and salting-out chromatography (3) have been reported as analytical methods for these types of condensates. These methods, however, can-

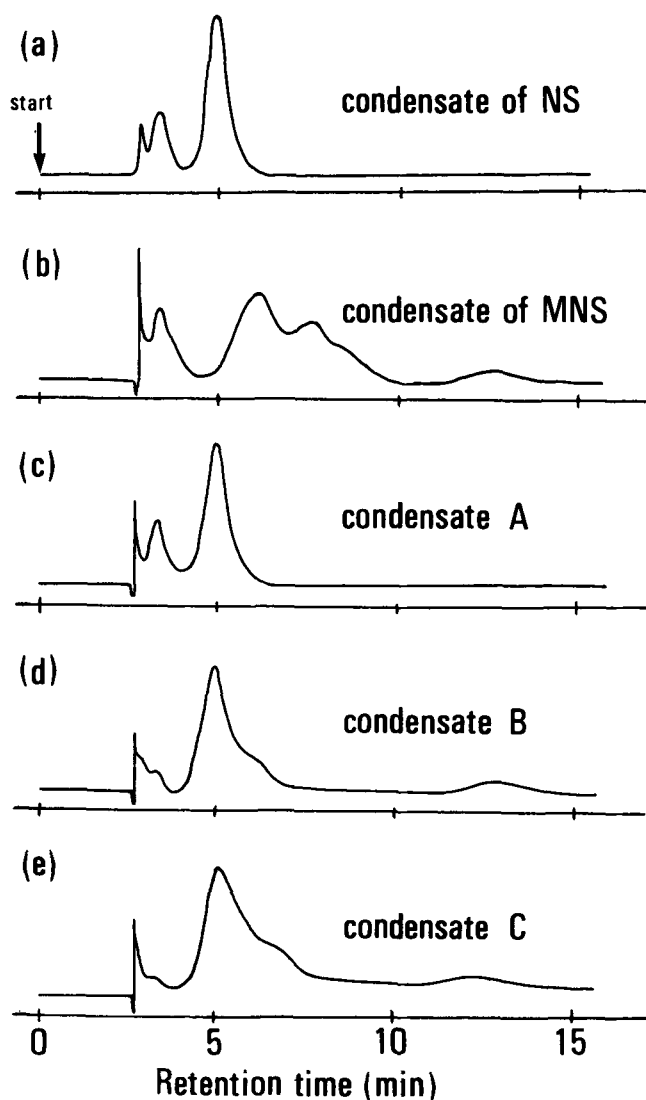


FIG. 1. Chromatograms of monomer. Conditions: 1 m x 2.1 mm column packed with Zipax SAX; mobile phase, 0.005 M, NaClO₄.

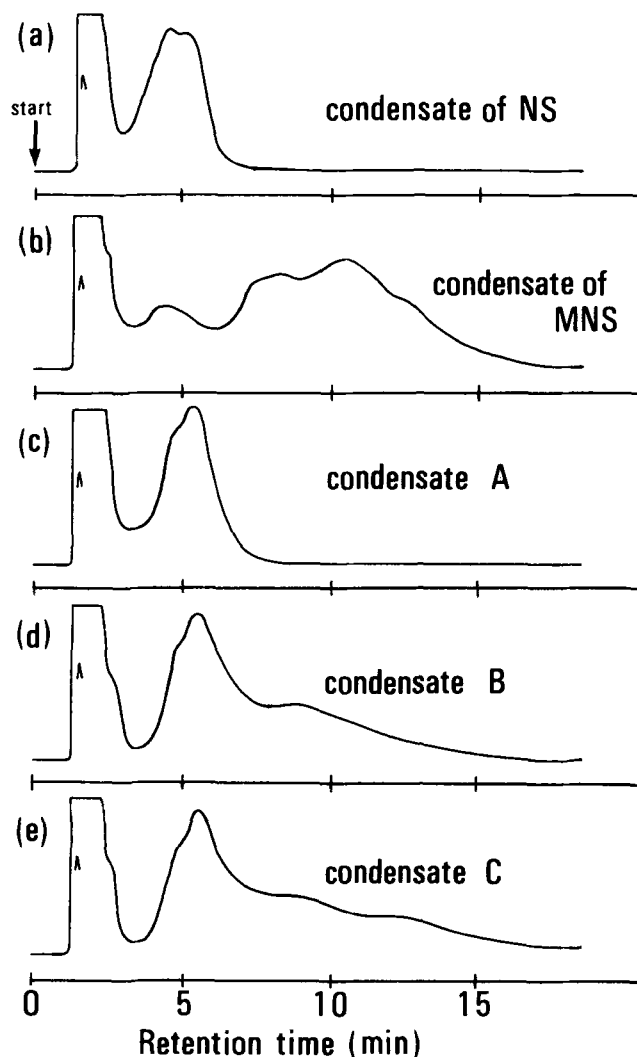


FIG. 2. Chromatograms of dimer. Conditions: 1 m x 2.1 mm column packed with Zipax, SAX; mobile phase, 0.5 M, NaClO₄.

not be utilized in the analysis of the condensates of the mixtures of NS and β -methylnaphthalenesulfonic acid (MNS) with formaldehyde because of the difficulty of separation of NS and MNS and also their condensates into their components.

This article describes the analysis of low condensed components, monomer and dimer, in the condensates of mixtures of NS and MNS with formaldehyde by high speed liquid chromatography.

EXPERIMENTAL PROCEDURES

The following three kinds of condensates of the mixtures of NS and MNS with formaldehyde, prepared by mixing both sulfonic acids in certain proportions, were synthesized in our laboratory: condensate A, NS-MNS, 3:1 (mole ratio); condensate B, NS-MNS, 1:1; and condensate C, NS-MNS, 1:3. The condensates of NS or MNS alone with formaldehyde were prepared as standards. The conditions of synthesis of these condensates were as follows: sulfonation of naphthalene or methylnaphthalene with sulfuric acid was carried out for 5 hr at 150-160 C. Then, formaldehyde (0.95-1 mole sulfonic acids) was added to the sulfonic acids during the next 2 hr, maintaining the temperature at 80-90 C. Condensation was continued for 3 hr at 100 C.

The fractionation of monomer and dimer of the condensates was carried out by salting-out chromatography using CM-Sephadex C-50 and 0.005 M magnesium chloride aqueous solution as stationary and mobile phases, respectively, according to the procedure described before (3). Each fractionated effluent containing magnesium chloride was analyzed by a high speed liquid chromatograph (model 830, du Pont de Nemours and Co., Wilmington, Del.) equipped with an UV detector (wave length, 254 nm) for monitoring the column effluent. The column used in this study was 1 m long with an internal diameter of 2.1 mm and was packed with Zipax SAX, a strong anion exchange resin.

RESULTS

Figure 1 shows the high speed liquid chromatograms for the monomer fractionated from each condensate. The first two small peaks (retention time, T_R ; 2.9 min, 3.4 min) in front of the main peak in each chromatogram are assumed to be due to both the elution of magnesium salt and other impurities in the fraction. The main peak in Figure 1A is based upon the elution of NS. In Figure 1B, the last peak (T_R ; 12.8 min) is due to the elution of dimethylnaphthalenesulfonate (DMNS) as an impurity. The main peak of condensate A (NS/MNS, 3/1) was assigned to NS. When

increasing the proportion of MNS in the mixture, other small peaks appeared in the chromatograms, showing the presence of MNS and DMNS. However, not only the main component of condensate A, but also that of the condensate B and C were NS, regardless of the ratios of NS and MNS.

The chromatograms for the dimer are shown in Figure 2. The first peak in each chromatogram is due to the elution of magnesium salt analogous to that in Figure 1. In Figure 2A, two peaks (T_R ; 4.5 min, 5.4 min) are assumed to be due to the position isomers of the dimeric condensates of NS. As can be seen in Figure 2, it is apparent that each main component of the condensate A, B, and C was the dimeric condensate of NS alone (NS-NS type). Condensates other than the NS-NS dimer (probably NS-MNS or MNS-MNS type) were found in the chromatograms as the proportion of MNS in the mixture increased (Figure 2D and 2E).

DISCUSSION

On the basis of these data, it seemed reasonable to assume that the main components of monomer and dimer in the condensates of mixtures of NS and MNS with formaldehyde were NS and the dimeric condensate of NS-NS, respectively, regardless of the ratios of NS and MNS. This result suggests that the reaction rate of condensation of MNS is larger than that of NS.

In this study, the mixtures of sulfonic acids were allowed to condense with formaldehyde for 3 hr, but the reaction time for commercially produced condensates for use as dispersing agents is usually much longer. Accordingly, it is thought that MNS or its dimeric condensates scarcely remain in the commercial product.

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New Quaternary Benzyl Chloride Salts for Inclusion in Hair Cream Rinse Formulations

ABSTRACT

Quaternary ammonium salts have been prepared from benzyl chloride and amino amides and an amino ester of a long chain fatty acid. These benzyl salts were incorporated in simple hair conditioner formulations containing the quaternary salt, anhydrous lanolin, cetyl alcohol, and water. Preliminary evaluations indicated that the formulations were effective in the

removal of tangles and snarls following washing with ordinary shampoo. The dried hair was easily manageable and had good luster.

INTRODUCTION

Although many new surface-active compounds have been reported in the past few years, many closely related compounds that could have desirable properties for specialty applications have not been prepared. Specifically, the

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