NOTE

Further Studies on an Improved Micro Method for Determination of Vicinal Diketones in Beer

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ABSTRACT

Studies on an improved micro method for determination of vicinal diketones in beer are reported. The concentration of K_2HPO_4 in the reaction mixture was found to affect the intensity of color. Use of water instead of K_2HPO_4 solution for adjusting the final volume resulted in a good linear relation between the concentration of vicinal diketones and the absorbance, and it improved the precision of the method. The chromogen was stable for at least 30 min.

Key words: *Beer, Colorimetry, Diacetyl, Vicinal diketone*

In the improved micro method (1) for determination of vicinal diketones in beer, the volume of the colored reaction mixture is adjusted with K_2HPO_4 as in the original micro method (2). However, when reaction mixtures containing a constant concentration of diacetyl were made up to volume with different volumes of 33% K_2HPO_4 solution and appropriate volumes of water, the intensity of color was found to vary with the volume of K_2HPO_4 solution added (Fig. 1). The difference in absorbance between a reaction mixture with 1.0 ml of K_2HPO_4 solution and a mixture without K_2HPO_4 solution was 0.009. This reading would correspond to 0.02 mg/L of diacetyl when a 40-ml portion of beer was analyzed. In practice, as large a sample of beer as possible is preferable for analysis so that the absorbance reading will be high and more nearly accurate. The larger the volume of beer sample, however, the larger will be the volume of distillate that accumulates in the trap. Consequently only a small volume of K_2HPO_4 solution will be used to make up the volume, compared to the 1 ml or more of K_2HPO_4 solution used to make a calibration curve. This will lead to a higher reading for vicinal diketone concentration with the sample beer.

To permit larger volumes of distillate to be analyzed, thus obtaining a high absorbance reading and reducing the extent of variation, the effect of omitting K_2HPO_4 solution was examined. The advantages of using K_2HPO_4 solution for making up the volume are supposedly that it gives a suitable pH for the color reaction and stabilizes the color. To test this, the stabilities of the color in reaction mixtures with and without added K_2HPO_4 solution were compared (Fig. 2). The pH values of these reaction mixtures were 10.24 and 10.10, respectively.

The intensity of color decreased more rapidly in the reaction mixture without added K_2HPO_4, but the decrease started at almost the same time in the two mixtures and was later than the time limit (30 min) prescribed for the measurements. The color intensity remained unchanged for 30 min even when the volume of the reaction mixture was 6.0 ml. The relation between the absorbance reading and diacetyl concentration without K_2HPO_4 solution was linear. The accuracy of the method was thus improved, and the number of reagents required for color development was reduced to four.

Several procedures were also found to produce a more reliable determination. 1) For efficient concentration of distillates after oxime formation reaction, the heating bath should be partially covered at the later stage of heating. 2) The rate of decrease in color

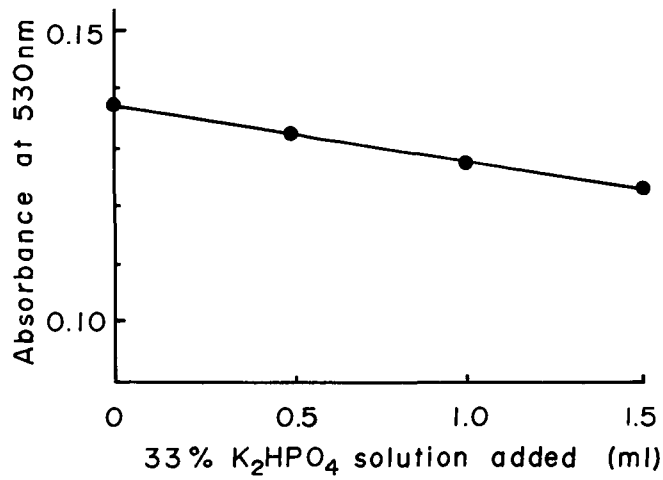

Fig. 1. Absorbance vs K_2HPO_4 concentration in the reaction mixture.

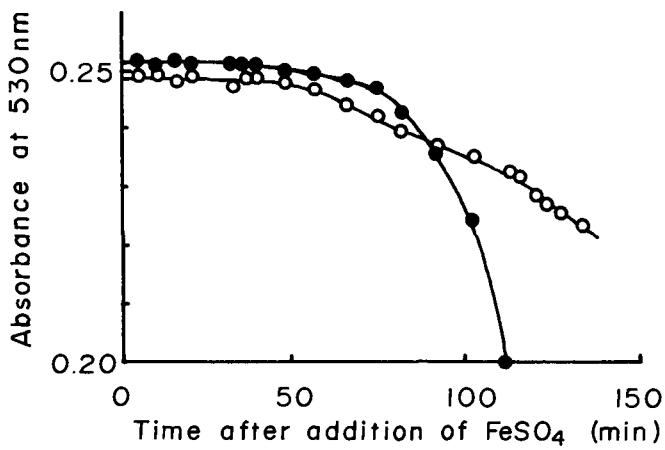

Fig. 2. Stability of color in the reaction mixture made up to a final volume of 5 ml with water (•) or with K_2HPO_4 solution (o).

intensity increases with decrease in the concentration of ammonia in the tartrate-ammonium hydroxide reagent. Some crystals of tartrate salt often form at the mouth of the reagent bottle, making the bottle difficult to plug tightly. However, the bottle should be tightly plugged and strong enough not to explode from the inner pressure of ammonia. 3) Because the recovery percentage of vicinal diketones varies with the type of gas-stripping instrument used and/or the gas-stripping condition employed, the percentage should be taken into account in calculation.

LITERATURE CITED

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