CHAPTER IV

DISCUSSION

Saccharin is the major non-caloric sweetener currently in use on a sucrose sweetener equivalent basis. It has been used as a sweetener in a wide variety of food products for many years. In spite of the limitation on the use of saccharin by the U.S. FDA and its removal from GRAS lists, its annual consumption in 1972 exceeded 4 million pounds (NAS, 1974).

The literature contains numerous reports on the determination of saccharin in foods and beverages, various techniques were used. For the qualitative determination of saccharin in food products, saccharin was converted to sodium salicylate and allowed to react with ferric chloride to give a blue-violet compound. However, the color resulting from reaction with salicylic acid is not specific for saccharin. Most phenols give an intense color reaction with ferric chloride; this is usually blue or violets, a carboxyl group ortho to hydroxyl group give violet color (Veibel, 1972).

Gnadinger (1917) used a gravimetric procedure for quantitative determination. An AOAC quantitative method in which interfering benzoates were removed by sublimation before determination was used in 1947, but the method lacks needed sensitivity and

specificity. A polarographic method for determination of saccharin was used by Lasheen (1966). A quantitative gas-liquid chromatographic procedure has been developed and applied to soft drink analyses by Conacher and O'Brien (1970). Korbelak (1969) studied a thin-layer chromatographic method which was adopted as official first action by the AOAC for the qualitative separation and identification of saccharin and other artificial sweeteners. DiPasquale and Corigliano (1968) reported a colorimetric procedure using ferroin (orthophenanthroline) for the determination of saccharin level as low as 2-6 ppm, but it lacks specificity.

Fernandez-Flores et al. (1973) modified and adopted the method for quantitative determination of saccharin in a variety of food products such as non-alcoholic beverages, fruit juices, liquid concentrates or food sweetener tablets, jellies, preserves, syrups, chocolate bars, high protein-low caloric liquids and powder foods. The method was as follows : samples containing saccharin were subjected to a clean-up procedure, using lead acetate or alcohol precipitation. Saccharin was extracted with a combination of solvents and the extract was treated with phenol and sulfuric acid, then heated at 175°C for 2 hours to form colored phenolsulfonephthalein derivative. The color was measured spectrophotometrically at 588 nm. Recoveries ranged from about 90 to 120 per cent in spiked samples. Satisfactory recovery was obtained from level as low as 5 mg/100 ml sample, but this method

was not suitable for routine work because of complex formation and vigorous reaction conditions required (Tanaka et al., 1977).

Ratchik and Visawanathen (1975) determined the saccharin in foods and beverages by gas-liquid chromatography. Infra red spectrophotometry was applied by Coppini and Albasini (1969).

An ion-electrode method was used to determine saccharin by Hazemoto et al. (1974). Basile (1966) reported an ultraviolet procedure for the determination of saccharin in wine. The stability of saccharin solution in sodium hydroxide solution was determined by ultraviolet procedure (DeGarmo et al., 1952). Hussein, Jacin, and Rodriguez (1976) also used ultraviolet spectrophotometry for quantitative determination of saccharin in food products.

The method used to determine the saccharin in beverages in this report utilized the solubility of saccharin in sodium carbonate solution and its unique absorption characteristic in this solution. Two distinct absorption maxima are exhibited at 229 nm and 235 nm, in addition to the broad absorption band exhibited in 265 nm to 275 nm range. The double absorption maxima can serve as an identity test for saccharin.

The absorbance at 244 nm was introduced to correct for any interference, however, if there was no apparent interference no correction was needed. Table 12 shows the results obtained from various sample solutions when calculation based on absorbance

at 235 nm with and without correcting for the absorbance at 244 nm. The best results were obtained when calculation were based on the absorbance at 235 nm corrected for that at 244 nm (Hussein, Jacin and Rodriguez, 1976).

Table 12

Results obtained at various wavelengths

(After Hussein, Jacin and Rodriguez, 1976)

Sample	% Saccharin added	% Saccharin found		
		A235-A244	A235	A268
Concentrated liquid sweetener	22.7	22.5	22.15	22.59
Carbonated soft drink (no saccharin and benzoates)	0	0	0.004	0.001
Iced tea mix (low caloric)	3.47	3.40	3.88	5.13
Fruit gum (sugarless)	0.10	0.11	0.14	0.12
Regular iced tea mix (no saccharin)	0	0	0.036	0.036

The interfering materials, that are encountered in the beverages are preservatives and flavorants. The flavorants are eliminated by pre-extraction with chloroform before acidification of sample solution, while preservatives : sorbates and parabens, are removed by pre-extraction with carbon tetrachloride after acidification (Hussein, Jacin, and Rodriguez, 1976).

The solvent diethyl ether was used for extraction of saccharin, because it is easily evaporated to dryness, (chloroform can also be used).

The percentage recovery of this experiment ranged from 94.9 to 102.0, the mean value was 98.5 per cent, standard deviation was 2.2. The results of detection and determination of saccharin in beverages showed that the producers still added saccharin in their products, in order to reduce the cost and ignored about the Food Quality Control Act which do not permit to use any sweetener in place of sucrose.

The UV-spectrophotometric method used in this experiment gave a high sensitivity. The method was simple and accurate, requiring minimal preparation to remove interfering substances. It is suitable for routine determination of saccharin in beverages and can also be applied to another food products.