

Section F. Chemical - Chairman: H. W. Dahlberg

SULPHITES IN SUGAR HOUSE PRODUCTS

Origin and Control

F. S. Ingalls

The study of sulphites in sugar house products has been one of rather wide scope extending into each unit of the Utah-Idaho Sugar Company's group of factories. Organization members from each has contributed toward the general survey.

The initial investigation consisted merely of the determination of the sulphites in the granulated output at each factory using the method of direct titration with standard iodine into 100 gms. sugar dissolved in 150 ml. freshly boiled and cooled distilled water. Inorganic SO_2 only was considered and the total iodine absorbed was calculated to SO_2 . Results from the initial survey showed wide differences in the SO_2 saturation of our granulated sugar not only as between plants but also as between strikes at the same plant, reaching a high of 170 ppm. and a low of 35 ppm. One plant however, produced sugar generally falling in the lower bracket while from another the granulated carried saturations within the higher ranges. Thus, we had two plants operating according to the recognized standards of the day, yet producing sugar strikingly different insofar as the sulphite sulphur content of the product was concerned.

Growing interest in control of the sulphites prompted further study with respect to source and probable quantities of the salt. Survey was commenced covering the battery supply water, kiln gas, 1st and 2nd carbonation juices, thin juices, thick liquors and massecuite. The SO_2 determination of materials up to the thin liquor failed to reveal SO_2 in any quantity. However, the thin liquor in all cases carried a relatively heavy saturation of SO_2 , pointing definitely to the fact that presence of sulphites in the liquors is directly traceable to use of sulphur dioxide in the process of manufacture.

Samples of thin liquor were collected from seven of the operating factories and immediately analyzed. Determinations for SO_2 were made by distillation following the scheme described under "Official Method - Association of Official Agricultural Chemists." Results from analyses of the juice collected showed a relationship between pH and degree of SO_2 saturation. In general, the degree of SO_2 saturation varied inversely with the pH, although a lack of correlation between the two lead us to suspect a satisfactory control of the sulphites would involve regulation over the juices even before the addition of sulphur dioxide.

The next step in our investigation involved study of effect from changes in lime addition per cent on beets, alkalinity of the first carbonation juice, pH of second carbonation juice, pH and SO_2 saturation of the thin liquor. Changes in lime addition where batch carbonation was employed appeared to be a factor; with all other values constant, sulphites in the granulated were found to be in the lower regions when the lime addition exceeded 2.4% CaO on beets. Alkalinity of the first carbonation juice, when held within the

limits necessary for proper filtration and normal colors, is definitely not a sulphite control feature.

During carefully regulated periods in which pH of 2nd carb. was varied and pH of thin juice held within narrow limits, the SO₂ saturation of the thin juice followed much more closely pH of the thin juice than during any previous survey.

This study was followed by one arranged to determine a desirable combination between pH of second carb. and pH of thin juice, with second carb. pH running from 8.6 to 9.0 and thin juice pH running from 7.4 to 8.6. From data assembled during the study, it appeared that SO₂ concentration of the thin juice reached its maximum when a relatively high pH second carb. was sulphured to 7.4 pH, 7.4 pH at this time being designated as the low point in the tolerant limits for thin juice. The minimum SO₂ saturation was obtained as a relatively high pH second carb. was sulphured to 8.6 pH.

Selecting from the lot of tests a group where the pH drop was constant at .4 second carb. pH running from 8.6 to 9.0 and thin juice pH from 7.4 to 8.6, the lowest thin juice saturation followed the sulphitation of 8.8 pH second carb. to 8.4 thin juice. Sulphitation of 9.0 pH second carb. to 8.5 pH thin juice, showed a somewhat higher SO₂ saturation of the thin juice than did the 8.8 - 8.4 combination.

Control of the thin juice pH to measurements falling within the region above 8.4 was at first viewed with some concern, particularly respecting effect on the sugar and simple syrup colors. However, colors have been generally better since higher pH controls were established, although no claim is made that higher pH were instruments in the improvement of colors, since several other process refinements have been put into force during the past few years.

Quoting values from our 1939 General Laboratory Report, sulphites in the granulated sugar from the factory during which the time of initial survey showed sulphite concentration up to 170 ppm. now show an average sulphite of 7.7 and sulphites in the sugar from the factory formerly showing 35 ppm, now show an average to date of 4.9. Other factories of the group are showing generally low sulphites with periods wherein the saturation reaches one and two ppm.

Conclusion: Always assuming sufficient lime addition and first carbonation manipulations of character to provide desired reactions within the first carb. tanks, we have found that further carbonation during the second stage preceded by heat on the high alkalinity side and carbonated to a pH of 8.8-9.0, followed by SO₂ saturation to pH of 8.4-8.6, will provide thin juice from which sugar of relatively low SO₂ content may be expected.

In closing, acknowledgement is made for the many helpful suggestions offered from the Bureau of Agricultural Chemistry and Engineering through Mr. J. C. Keane who was associated with the Division at the time our survey commenced.