

A DEMONSTRATION OF THE NECESSITY FOR CARE IN SAMPLING

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PROPER sampling is often more difficult than the analysis of the sample after it is obtained. This fact is seldom appreciated by students in quantitative analysis, even though they may be given several lectures on the subject. Their failure to appreciate the necessity for care in sampling is due, in part at least, to the fact that the samples usually issued to students for analysis are practically fool-proof. They are very finely ground in order that individual samples may be taken quickly, without special precautions, from a stock sample.

During the past four years, an exercise intended to impress upon students the importance of sampling has been included in a specialized course in analysis given by this department. It has proven so simple, and so effective, that it may be of interest to other teachers in analytical courses.

All of the students in this course have completed at least three hours, and some have completed six hours, of quantitative analysis in the chemistry department. As a preliminary exercise, they are asked to standardize approximately tenth normal solutions of acid, alkali, and silver nitrate. Each student is then given a sample and asked to analyze it in triplicate with great care. He is told that this is a test of his ability to do accurate work. The nature of the sample is such that great care is necessary in sampling, but almost invariably the students treat it as though it were already finely ground and intimately mixed. Later there is consternation and dismay when they discover that duplicate titrations may differ in the ratio of 2:1, or even more.

The results obtained by the class in 1935 may be considered as typical. There were fifteen students in the course and each reported the results of three analyses. The samples were all identical and each contained chloride equivalent to 39.20 per cent. NaCl. The results of individual determinations ranged from 25.40 per cent. to 79.16 per cent. The nearest single determination was 44.79 per cent. The nearest average of three titrations was 42.40 per cent. the individual titrations being 29.66 per cent., 47.58 per cent., and 49.97 per cent.

One student weighed out four portions with the in-

tention of reporting the three in closest agreement. When he obtained values of 55.45 per cent., 45.74 per cent., 55.79 per cent., and 56.20 per cent., he accused the instructor of not mixing the sample. He then weighed out three more portions after shaking the remainder of his sample vigorously. These gave values of 25.40 per cent., 47.10 per cent., and 60.92 per cent.

This particular student then weighed and titrated all of the remainder of his sample, and by adding up the chloride found in each titration, and the weights of the various portions, he calculated the sodium chloride in the entire sample issued to him. This result was 39.00 per cent.

When it was finally explained to the class that the samples could not be adequately mixed without additional grinding, this student asked for another sample. He ground it carefully in a mortar before weighing out portions for titration. The duplicates checked closely, and gave an average value of 39.16 per cent. of NaCl.

We do not often have a student as persistent as this one, but each year the results of the class as a whole are similar to those just described, and we feel that the necessity for care in sampling is clearly demonstrated.

The actual preparation of the samples is not difficult. The materials used in this particular case were KCl which had been screened to pass an eight-mesh but not a twenty-mesh sieve, and ordinary granulated sugar. The samples issued to students contained 5.000 grams of each of these substances. Various other combinations have been used, and many are possible. The only requirement is that the active and inert ingredients be of different particle size, those of one component being relatively large. The samples must be prepared individually, but if five-gram portions of high grade chemicals are weighed out with an accuracy of two or three milligrams, the true composition of the mixture is known with greater accuracy than that of many of the samples used in analytical courses. The actual weighing can often be turned over to an assistant who need not even know the nature of the substances weighed, and consequently could not know the composition when calculated in terms of some other salt.