GENERAL ANALYTICAL CHEMISTRY SECTION

(Monthly Summary)

FOR THE PERIOD: August 30 to September 20, 1976

DATE: September 27, 1976

FACILITY LEADER: Elisabeth T. Oakley

I. ROUTINE DETERMINATIONS (1)

Routine determinations made since the last monthly report were as follows:

Chemical	2597
Oven Volatiles	1736
TOTAL	4333

The projects which contributed the heaviest work load and the major analyses requested were:

Charge No. 0108: phenols and gas phase puff-by-puff.

Charge No. 1307: nitrate nitrogen, soluble ammonia, amino sugars, phosphorus, sorbic acid, alkaloids, ash, total and soluble solids, reducing sugars, hot water solubles, total nitrogen, ammonia in smoke, and gas phase puff-by-puff.

Charge Nos. 1801, 1803, 1804: alkaloids, reducing sugars and soluble ammonia for 1801 only; 55% of all the oven volatiles for the three expanded tobacco projects combined.

Charge No. 2305: amino sugars, soluble ammonia, phosphorus, total solids, reducing sugars, total nitrogen, nitrate nitrogen, chlorides, urea, and ammonia in smoke.

Charge No. 8401: alkaloids, reducing sugars, total nitrogen, ash, and petroleum ether extractables.

Charge No. 8505: alkaloids, reducing sugars, total nitrogen, total volatile bases, petroleum ether extractables, ash, soluble ammonia, and carbon in filter plugs.

A series of small problems has slowed the output of the gas phase puff-by-puff lab to 167 runs.

II. METHODS DEVELOPMENT AND IMPROVEMENT

A. Ammonia Ion-Selective Electrode (2)

A series of smoke samples were analyzed by the routine colorimetric method for ammonia on the Robot Chemist and by the NH_3 ion-selective electrode on the AutoAnalyzer II. The ISE system appeared to work satisfactorily with the smoke solutions. Two things were noted, however, that bear further investigation: (1) the NH_3 was about 60% higher using the ISE, and (2) the absolute increase in apparent NH_3 with aging of the smoke solutions was of the same order of magnitude by both procedures.

B. Oven Volatile Study

At the suggestion of Dr. Tibor Laszlo, a comparison study was begun of oven time and temperature *versus* percent oven volatiles. A series of 25 ground bright leaf tobacco samples were dried at 103°C for 100 minutes and compared with samples dried under the normal conditions (100°C for 180 minutes). The agreement was very good. Additional work will be done on various types of ground and unground bright, burley and oriental leaf, filler and sheet tobacco in a wide range of moisture levels.

C. Starch in Green Tobacco (3&4)

Work is continuing on the analysis of fresh green and freeze-dried green tobacco for starch to determine the reason for an apparent substantial decrease in starch content on freeze-drying. Pure amylose and α-amylopectin were found to have 74% and 51% response, respectively, when measured *versus* the soluble starch standard, indicating that a change in the ratio of the two forms would not produce the drastic reduction noted. A manual procedure (4) using cold perchloric acid to solubilize the starch has been used on monitor tobacco and will be used for green tobacco when new samples arrive.

D. Total Nitrogen in Process Water (5)

Efforts continue to achieve quantitative values for total nitrogen in process waters containing 10 to 100μg/ml of nitrate nitrogen. Modification of an AOAC (6) method for nitrate nitrogen in mineral water using a small amount of dilute NaOH during the evaporation step resulted in an average recovery of 96%.

E. Total Nitrogen in Kjeldahl BD Digests (3)

A new AutoAnalyzer I system for total nitrogen in Kjeldahl BD digests has been built and samples are being analyzed on both systems for comparison. The system is essentially the same as the one presently used except that the new system uses sodium salicylate in a buffered alkaline medium instead of phenol. Possible advantages include reagent stability, complexation of interferences by the tartrate buffer, and dialysis to minimize variation in salt concentration in the digests.

III. MISCELLANEOUS

A. Monthly Oven Check

The results of the monthly oven check for September have not been received yet. However, our results may be inaccurate because we have experienced a severe problem with erroneous weights on the electronic balance-microprocessor system. It has been traced to a possible static charge build-up on the balance. This has been alleviated somewhat by use of an anti-static spray, but a more effective and permanent solution is being sought.

B. Non-routine and Semi-routine Analyses

1. A dried, sieved sample of crystals (coded K6CGO) from the denitration pilot plant have been analyzed repeatedly in order to obtain sufficient precision data to set up specifications and also to obtain a material balance. The analyses requested included nitrate nitrogen, inorganic sulfate, phosphorus, total solids, cold water insolubles, and ash. Attempts to identify any organic materials present showed that only 1 to 2% organics were present. (1,5,7)

2. Two water samples (one treated, one untreated) from HPC were analyzed for $SO_4^{=}$ and HCO_3^{-} reported as µg/ml. (1,5)

3. Nicotine and ammonia were determined on two Westab gas samples and also on a series of process residues from Manufacturing Engineering (Charge No. 8205). (7)

4. The filtrate and residue of a sludge sample from the central plant cooling tower were analyzed for phosphorus content (Charge No. 0007). (7)

IV. REFERENCES

1. Routine Analytical File

2. Notebook 6921

REFERENCES *continued*

3. Notebook 7023

4. Oakley, Elisabeth T., Completion Report, "The Determination of Starch in Tobacco," April 21, 1970.

5. Notebook 6610

6. Official Methods of Analysis, A.O.A.C., 11th Edition, Method No. 33.008, p. 565.

7. Notebook 6864

V. MEMOS ISSUED

1. Handy, Betty to Mr. Wayne Galler, "Samples for Ammonia," September 13, 1976.

2. Handy, Betty to Mr. Wayne Galler, "Samples for Nicotine Alkaloids," September 20, 1976.

Elisabeth T. Oakley

/bpp