I. INTRODUCTION

The tobacco industry is one of the leading businesses in the world today. Consumer tobacco products commonly manufactured throughout the world are cigarettes, pipe tobacco, cigars, snuff and chewing tobacco. Among them, cigarettes are the most popular product. "Quality First" has been a key issue within our company, since the introduction of the first tobacco product by R. J. Reynolds in 1895. Aroma plays an important role in the estimation of the quality of cigarettes. Only cigarettes which have the characteristic and typical aroma of cigarettes are acceptable. Today, consumers have become more sophisticated and more demanding. In 1984, a survey conducted by R. J. Reynolds Tobacco Company showed some 17,000 cases of consumer complaint, approximately 10% of which were off-taste cigarettes (10). To ensure the company products remain among the best-selling in the world, the highest standards of quality must be maintained. Quality assurance is, therefore, vital to the future of Reynolds Tobacco Company. The company's future depends on our ability to increase the share in the many markets we serve around the world.

Recently, the number of consumer complaint off-taste cigarettes received for chemical analysis have been increasing. A complete analysis of the off-taste cigarettes became necessary. The capacity for rapid identification of a wide range of volatile contaminants is often minimal. Moreover, the needed tests were virtually unavailable in the past. Various methods for qualitative determinations of the volatile compounds have been described in literature (2,3,5,11-13). In most cases, they are often less specific and included other components. A feasibility study with a GC/MS system was developed to provide volatile analysis with the purge-and-trap technique.

II. EXPERIMENTAL

A. Instrumentation

1. Purge-and-Trap Sampler

A Hewlett-Packard purge-and-trap sampler Model 7675A was used. The trap was a short column of stainless steel packed with 60/80 mesh Tenax GC.

2. ~~Gas Chromatograph/Mass Spectrometer~~ Smoking Machine

A smoking machine, modified from a Filamatic Vial Filler Model AB-5 (National Instrument Co., Baltimore, Maryland) was used.

3. Gas Chromatograph/Mass Spectrometer

A Hewlett-Packard 5985B GC/MS system equipped with Hewlett-packard 5840 GC was used. This GC/MS system was interfaced to a Hewlett-Packard 1000 computer running the HP Mercury Data System. A 60 m DB-5 (bonded liquid phase - phenyl methyl silicone) fused silica capillary column (0.25 mm i.d., 0.25 µfilm thickness) was purchased from J&W Scientific, Rancho Cordova, California.

permits . . . . . vapor

pressures in the presence of a changing matrix and is therefore not reproducible.

Two typical mass chromatograms of the volatile compounds in the control and the customer complaint cigarettes are shown in Figures 1 and 2, respectively. In Figure 2, a group of the peaks, showed between the retention times of 33 to 46 minutes, was found in the customer complaint cigarette profile. The mass spectra of these compounds were identified and are listed in Table I. A typical mass spectra is shown in Figure 3. A group of alkylated benzenes were identified. These compounds were not found in the control cigarettes by the method used as shown in Figure 1. By comparing the differences in the packages between the customer complaint cigarettes and the control cigarettes, very dark tax stamp inks were always printed on the customer complaint cigarette packs. The residual solvents of the tax stamp inks were suspected as the source of these alkylated benzenes. To verify this the volatile compounds of the stamp inks were analyzed in the same manner as that in the customer complaint and the control cigarettes. A typical mass chromatogram and a mass spectrum are shown in Figures 4 and 5, respectively. The volatile compounds between the retention times of 30 to 52 minutes were identified and are listed in Table II. The same types of alkylated benzenes were identified and match alkylated benzenes found in the customer complaint cigarettes.

2. Comparison of the Cigarette Components

The casing materials contain mainly sugars and humectants. The sugars are added to tobaccos to enhance their quality by balancing the chemical composition and to develop certain desired flavor depending on whether the tobacco is destined for cigarettes. The humectants, usually glycerine or a propylene glycol, serve to keep the tobacco moist and less sensitive to changes in humidity. Flavorings or top dressings (usually highly aromatic, perfume-like substances) are added to the tobaccos. These can be natural products or their extracts. Their purpose is to supply aroma or pleasing flavor to the smoke and the package. Menthol is one of the substances. Blends of various tobaccos are commonly used in smoking products to insure the same flavor and taste in the final product mixture. The blending, casing and flavoring of tobaccos are necessary to compensate for variations in the chemical composition of the tobaccos used, such as the relative presence or deficiency of certain classes of constituents. Using certain chemical analyses and flavor evaluations of grades and crop years, blends for cigarettes are made based on the taste preference of the intended customer as well as cost and availability of the tobaccos.

The components in cigarettes are of two characteristics, volatile and non-volatile. The volatile constituents in cigarettes are often transferred directly from the cigarettes into the mainstream of smoke without structural changes during smoking. Because of their high vapor pressures, cigarette volatiles are primarily responsible for the aroma of cigarettes. Two analytical methods for the isolation of volatile compounds from cigarettes are commonly used. They are solvent extraction and steam-distillation. The solvent extraction technique was used in this study.

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The cigarette volatile compounds of the control and the customer complaint cigarettes were analyzed and compared. Two typical mass chromatograms of the control and the customer complaint cigarettes are shown in Figures 6 and 7, respectively. After careful examination of the mass chromatograms and the mass spectra between the control and the customer complaint cigarette samples, there were no significant differences in compounds found qualitatively and quantitatively between the control and the customer complaint cigarettes.

Non-volatile compounds in cigarettes, such as sugars, cellulose, amino acids, long-chain fatty acids, polyphenols and sugar amino acid complexes (Amadori compounds), can be analyzed by the pyrolytic method. Combustion of the cigarette is a complex chemical process in which hundreds of compounds are formed by pyrolysis. Non-volatile compounds pyrolyze and produce completely different constituents. For example, neutral oxygen-containing compounds, such as aldehydes, ketones, alcohols, and furans arise from the pyrolysis of carbohydrates. Nitrogen heterocycles, amines, and nitriles arise from the amino acids and nitrate content of tobacco. These pyrolytic compounds produce specific tastes that enhance smoke flavor.

The pyrolyzates of the control and the customer complaint cigarette samples were analyzed and compared. Two typical mass chromatograms of the control and the customer complaint cigarette samples are shown in Figures 8 and 9, respectively. After careful examination of the mass chromatograms and the mass spectra between the control and the customer complaint cigarette samples, no significant differences in compounds were found qualitatively and quantitatively between the control and the customer complaint cigarettes.

From the above studies, it clearly indicates that there were no variations in tobacco casing, blending and flavoring between the control and the complaint cigarette samples.

IV. CONCLUSION

The customer complaint cigarettes have been analyzed for the residual solvents, casing, flavoring materials, and blend components. The data were compared with those of their control cigarettes. Residual solvents, i.e. alkylated benzenes, were found in the customer complaint cigarettes but not in the control cigarettes. There were no significant differences in casing and flavoring materials and tobacco blend components between the customer complaint and the control cigarettes. In conclusion, some residual solvents from