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To: Don Leydon Date: September 24, 1993
From: W. R. Raymond *W. R. Raymond*
Subject: Ashland Laboratory/Glycerin Analytical Method

Don, per your request, please find summarized below the current method used for the determination of glycerin in tobacco sheet at the Ashland Laboratory. This method was used for the inter laboratory comparison.

1. Extraction Solution: Internal Standard (5mg/ml) prepared by weighing 5.0 g 1,6-hexanediol (Aldrich 99%), to nearest mg into 1 liter volumetric flask and making to volume with 95 % ethanol.
2. Standards: 0.5, 1.0, 3.0 and 5.0 mg/ml glycerin (Baker Analyzed Reagent) in extraction solution. Prepared by diluting 10 mg/ml glycerin stock solution with extraction solution (25 ml volumetric flasks). Glycerin stock solution prepared by weighing to nearest mg, 1 g of glycerin into 100 ml volumetric flask and making to volume with extraction solution.
3. G.C. Column: 30 meter x 0.53 μ I.D. ChromPack bonded CP-Wax-57B flexible silica capillary column (1 μ m film thickness). Carrier Gas = He @ 5.5 ml/min. Split Ratio = 20:1.
4. G.C. Conditions: See attachment.
5. G.C. Calibration: See attachment.
6. Sample Preparation:
 - a. Mat sample cut into small pieces (≤ 5 mm) and pieces mixed.
 - b. 100 \pm 5 mg weighed to nearest 0.1 mg into Pierce 3 ml ReactiVial.
 - c. 2.0 ml extraction solution added.
 - d. Vial sealed and heated in Pierce ReactiTherm block heater for 1.5 hours @ 50 °C.
 - e. Vial shaken at ca. half hour intervals during extraction.
 - f. After cooling to ambient temperature, extract transferred to autosampler vial.
 - g. Filtering (0.45 μ AcroDisk PTFE filter) optional.
 - h. For normal laboratory analysis, samples are run in duplicate.
7. Other Information:
 - a. Ashland samples are equilibrated at 60% R.H. before analysis.
 - b. Results are uncorrected for moisture.
 - c. Column pre equilibrated with several injections of 0.5 mg/ml standard.
Number of pre injections (7 maximum) depends on length of column idle time.

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