CAPSULES
preliminary notes and applications from Bioanalytical Systems, Inc.

Ethylenethiourea in Foods

Purpose
Determination of ethylenethiourea residues in foods by LCEC.

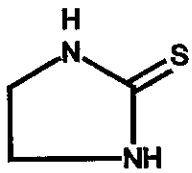

Figure 1. Structure of ETU.

Ethylenethiourea (F1, ETU) is a degradation product of the ethylenebisdithiocarbamate fungicides, which are widely used on agricultural crops. ETU is a suspected carcinogen, so there is a need for accurate and rapid measurements of its concentration in foods.

Existing Methods
Derivatization followed by GLC, but the derivatization reactions do not occur quantitatively in the presence of plant coextractives. LCUV also has been used, but many coextractives are UV absorbing, thus reducing selectivity.

Reference
Krause, R.T. and Y. Wang, Liquid Chromatographic-Electrochemical Technique for Determination of Ethylenethiourea Residues, J. Liq. Chromatogr. 11 (1988): 349-362.

Conditions
Detector: BAS LC-4B Amperometric Detector
Electrode: BAS Au/Hg
Potential: + 0.36 V vs Ag/AgCl
Column: 6 µm spherical, C 8 reverse-phase, 250 x 4.6 mm. The column was maintained at 60 °C.
Mobile Phase: Water. Flow rate was 1.5 mL/min. An electrolyte solution (0.1 M H_3PO_4) was added to the column effluent (upstream of the detector) at a rate of 0.5 mL/min.
Detection Limit: 2 ng injected.
Linear Range: 2-400 ng injected.

Sample Preparation
Crop samples were extracted with an aqueous methanol solution, then filtered and concentrated. ETU was separated from coextractives by elution from a Gas Chrom S/alumina column with ethanol/chloroform. After drying in a rotary evaporator the samples were redissolved in water, filtered through 0.45 µm membranes, and injected in 20 µL aliquots.

Notes
The electrolyte solution was added post-column because C 8 columns are not stable at low pH. The concentration of electrolyte added (0.1 M) provided a concentration of 0.025 M in the thin-layer electrochemical cell.

A wall jet electrochemical cell (ESA) also was evaluated for this determination, but was rejected for excessive noise.

Crop coextractives had no effect on measured recoveries (near 100%) from apples, grapes and potatoes, demonstrating the selectivity of EC detection. Recovery from spinach was only 90%, but it was not known whether this reduced efficiency was caused by coextractives.

The analysis of ETU described above also can be performed on the BAS 400 Liquid Chromatograph and the BAS 200 Problem Solver.

The information in this publication is supplied as a service to our customers. Performance of the methodology has not necessarily been verified by BAS technical staff.

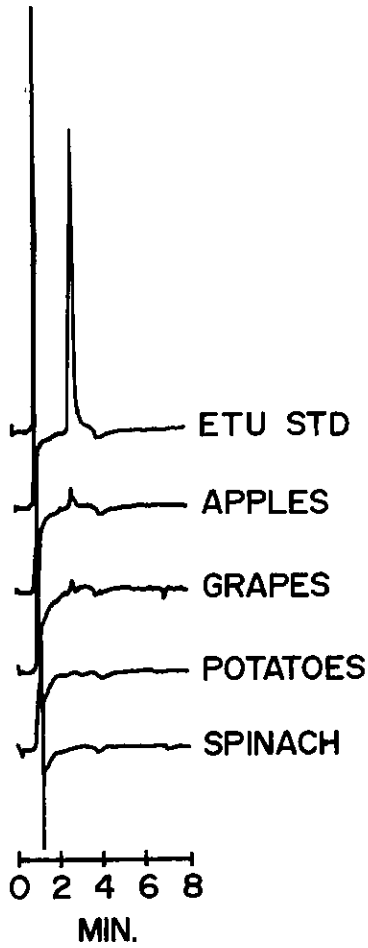

Figure 2. Sample chromatogram of standard ETU (10 ng, top) and crop extracts. Only apples and grapes contained measurable ETU residues.