musty or earthy taint found in some water supplies. It seems possible that the source of the geosmin in the beans then could result from the action of Actinomycetes, either on the beans themselves or in the water supply used during the growing of the beans. In the former case, the beans would have had to be in a moistened condition, i.e., before the beans were dried or by being wet after drying.

A number of musty off-flavored bean samples were studied by the authors. Although geosmin seems to be responsible for the musty off-flavor in the beans used in this study, we do not believe that this is always the case, and feel that other compounds can also be involved in causing musty off-flavor in beans. We have located some of these other compounds by odor evaluation but, up to the present time, have not been able to characterize them.

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A Method for the Determination of Ethylenebis(isothiocyanate) on Food Crops

A method was developed for the determination of ethylenebis(isothiocyanate) on food crops. The method is sensitive to 0.02 ppm and yields mean recoveries of 98% from 0.05 to 10 ppm of added residue. Interferences are not observed with either ethylenebis(dithiocarbamate) or ethylenethiuram monosulfide.

Ethylenebis(isothiocyanate) (EBI) has been postulated as a fungicidal decomposition product of the ethylenebis(dithiocarbamate) fungicides (Thorn and Ludwig, 1962). There is spectroscopic evidence to suggest that EBI can be formed from ethylenethiuram monosulfide, an intermediate in the decomposition of the ethylenebis(dithiocarbamates) (Ludwig et al., 1955). Engst et al. (1968) have presented thin-layer chromatographic evidence for the presence of EBI on tomatoes treated with maneb. The following method was developed to permit the quantitative determination of EBI on food crops treated with ethylenebis(dithiocarbamates).

EXPERIMENTAL SECTION

Materials. Ethylenebis(isothiocyanate) was prepared by thermal decomposition of the bis(ethoxycarbonyl) derivative of disodium ethylenebis(dithiocarbamate) in boiling toluene (Thorn and Huston, 1959). The isothiocyanate was purified by column chromatography on silicic acid (Mallinckrodt, 100 mesh) using hexane–benzene (1:1) as eluent. The infrared spectrum of the purified material in chloroform gave bands characteristic of an isothiocyanate at 2100 and 2125 cm^{-1}, while the 60-MHz NMR spectrum showed a CH_2 singlet at τ 6.13 ppm in deuteriochloroform.

A stock solution of EBI was prepared by dissolving a sufficient amount of the compound in toluene to give a concentration of approximately 200 μg/ml. Standards for gas–liquid chromatography were made by serial dilution of the stock to a concentration of 20 ng/ml. A 5-μl sample of this solution produced a peak with 70% full scale deflection on the gas chromatograph at working attenuation. Solutions of EBI used to fortify crop samples were prepared by dilution of the stock in absolute ethanol. Samples were fortified by the addition of 0.10–0.50 ml immediately before extraction.

Table I. Recovery of EBI from Various Commodities

EBI added, ppm	EBI recovered, %[a]		
	Apple	Tomato	Lettuce
0.047	93.8	85.9	86.8
0.094	108	105	105
0.472	86.5	86.8	90.1
0.944	94.8	99.7	95.9

[a] Values are the means of duplicate determinations.

Procedure. A sample of crop material (5.0 g) was homogenized with absolute ethanol (50 ml) by blending at high speed for 30 sec on a Sorvall Omni-Mixer. The homogenate was filtered through Whatman No. 1 paper and an aliquot (20 ml) of the filtrate placed in a 125-ml separatory funnel. After the addition of 1.0 *M* NaCl (80 ml) the aqueous phase was extracted with toluene (10 ml). The toluene layer was shaken with a fresh portion of 1.0 *M* NaCl (20 ml) and then with 1.0 *M* HCl (10 ml). An aliquot (1–2 ml) of the toluene extract was then passed through a small column of silicic acid prepared by placing a 1-cm bed of silicic acid on a glass wool plug in a pasteur pipet. A sample (5 μl) of effluent was analyzed by gas–liquid chromatography.

Gas–Liquid Chromatography. Samples were analyzed on a Hewlett Packard 5700 A gas chromatograph fitted with a ^{63}Ni electron capture detector and 6 ft × 4 mm i.d. glass column. The column was packed with 5% butanediol succinate on 100–120 mesh Chromosorb W, HP, and was preconditioned at 200°C for 48 hr under a flow of ar-

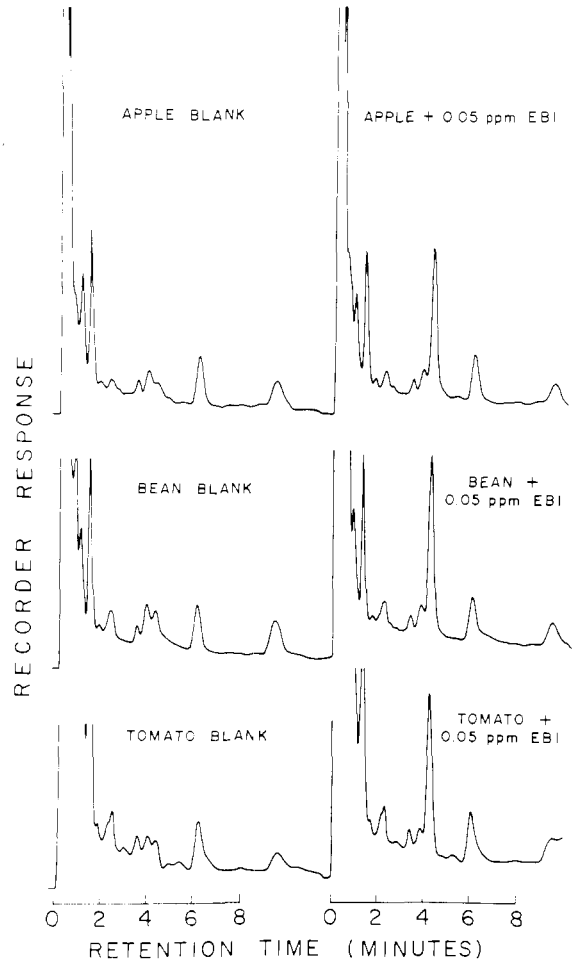

Figure 1. Gas-liquid chromatograms of final extracts of different commodities with and without the addition of 0.05 ppm of EBI. Each injection represents the equivalent of 1 mg of sample.

Table II. Linearity of Recovery of EBI from Beans

EBI added, ppm	EBI found,[a] ppm	Recovery, %
0.050	0.046	92.0
0.100	0.095	95.0
0.500	0.466	93.2
1.00	0.978	97.8
5.00	5.36	107
10.0	10.5	105

[a] Values are the means of duplicate determinations.

Table III. Recovery of EBI from Lettuce in the Presence of Maneb or Zineb

EBI added, ppm	Zineb (5.0 ppm) EBI found, ppm	Zineb (5.0 ppm) Recovery, %	Maneb (5.8 ppm) EBI found, ppm	Maneb (5.8 ppm) Recovery, %
0	0.004		0.008	
0.094	0.088	93.6	0.100	106
0.944	0.808	85.6	0.972	103

gon–methane (95:5) carrier gas. Typical operating parameters were as follows: column oven temperature, 190°C; detector temperature, 300°C; carrier gas flow rate, 30 ml/min. Samples were quantitated by comparison of the peak height to that of the standard.

RESULTS AND DISCUSSION

Typical chromatograms of extracts of various commodities fortified with EBI are shown in Figure 1. The lower limit of detection with a 2:1 signal:background ratio is 0.02 ppm. Since the presence of ethylenethiuram monosulfide in samples would lead to erroneous results due to its decomposition to EBI on GLC (Newsome, 1975), it was necessary to remove it by partitioning with HCl. No interference above normal background was observed when the monosulfide was added to samples at a level of 1 ppm.

The recoveries obtained from various commodities fortified with EBI are shown in Table I. The recovery was found to be linear with the amount of EBI added between 0.05 and 10 ppm (Table II) with an overall yield of 98.3%.

As shown by the data in Table III, the presence of either of the parent compounds, maneb or zineb, does not interfere with the method.

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Extraction of Methabenzthiazuron from the Soil

A fourfold solvent extraction procedure involving the use of acetone–ethyl acetate and acetone–chloroform combinations was developed for quantitative extraction of the herbicide methabenzthiazuron (MBT) from the soil. The recovery of MBT from soils spiked with [^{14}C]MBT 1 day before extraction was consistently over 95%, with each of the first three extractions removing over 60% of the MBT present or remaining in the soil. After a MBT-treated soil was aged for 6 months, only 50–70% of the ^{14}C remaining in the soil could be extracted. However, over 90% of the ^{14}C in the extracts was identified as the parent MBT, and the pattern of MBT removal from the aged samples was similar to that from spiked samples. It appears that the unextracted ^{14}C is either tightly bound by soil components or partially metabolized and could no longer be extracted by this procedure.

The herbicide methabenzthiazuron [*N*-(2-benzothiazolyl)-*N*,*N*'-dimethylurea or MBT] is used both in the fall and in the spring for weed control in cereal crops. It is degraded in the soil only at moderate rates (Cheng et