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Halogen Compounds Identified in the Volatile Constituents of Baked Potatoes

The volatile constituents of baked potatoes were isolated from 540 lb of Idaho Russet Burbank baked potatoes. The components of the baked potato volatiles were separated into relatively pure fractions by repetitive gas chromatography. The fractions were then analyzed by GC-MS and infrared spectrometry. Fourteen halogenated compounds were identified.

Residues of halogenated compounds used as pesticides, herbicides, and fungicides found in food is a topic which might be of vital importance to the health of consumers. Organochlorine pesticides were found to be persistent in the soil (Brown, 1978). The half-life in the soil for heptachlor, chlordane, and lindane was ~1 year, and for dieldrin, endrin, and DDT the half-life was in excess of 2 years (Edwards, 1973). Residues of heptachlor, aldrin, and dieldrin were found in various crops including soybeans (Eden and Arthur, 1965; Bruce and Decker, 1966) and potatoes (Terriere and Ingalsbe, 1953). The residual 2,2-dichloropropionic acid, a herbicide, was found in citrus fruits (Getzendaner et al., 1965). 1,2-Dibromo-3-chloropropane is used as a soil fumigant. Residues of 1,2-dibromo-3-chloropropane were found in beans, in potatoes (Guinn and Potter, 1962), in oranges (Castro and Schmitt, 1962), and on grains (Fishbein, 1976). The use of ethylene dichloride and ethylene dibromide in fumigant mixtures of disinfecting fruits, vegetables, food grains, and tobacco suggests the possibility that their residues or their respective hydrolytic products may be present in fumigated material (Beck, 1974; Fishbein, 1976; Dumas, 1973). The accumulation of organochlorine pesticides in poultry has been extensively studied (Kan, 1978). Organochlorine

pesticides were found to be persistent, and residues can accumulate in fat and eggs (Kan, 1978). In general, it can be said that halogen residues are persistent and undesirable in animal and plant products because of their possible toxic properties.

Although the use of many chlorinated hydrocarbon insecticides has been banned, some halogenated insecticides and fungicides are still being used on crops. For example, thiodan and phosphamidon are recommended in Idaho to be used to control the Colorado potato beetle (Homan et al., 1979) and ethylene dibromide is used as fumigant to control wireworm in potato (Sandvol et al., 1978); also, preplant treatment of pentachloronitrobenzene is used to control the rhizoctonia disease of potato (Davis, 1977). In addition, pentachloronitrobenzene is also used to control the common scab of potato (Davis and Garner, 1978).

In our study of the volatile flavor compounds of baked potatoes, we identified 14 halogenated compounds. The present paper reports their identification.

EXPERIMENTAL SECTION

The details of experimental procedures were described in Coleman et al. (1981).

Table I. Halogen Compounds Identified in the Volatile Constituents of Baked Potatoes

no. of peaks ^a	identified as	mass fragmentation, <i>m/e</i> (%)
1-5-2	1,1,1-trichloroethane	97 (100), 99 (71), 117 (28), 61 (24), 119 (18), 101 (15), 63 (11), 62 (8)
1-5-3	tetrachloroethylene	166 (100), 164 (81), 129 (58), 131 (55), 168 (47), 94 (30), 133 (22), 47 (18)
1-6-5	trichloroacetic acid	44 (100), 85 (75), 83 (52), 36 (38), 35 (22), 47 (20), 38 (16), 87 (11)
2-1-2	2-chloropropane	43 (100), 63 (30), 27 (21), 78 (20), 41 (18), 80 (10), 65 (6), 39 (6)
2-12-4	chloroform	83 (100), 85 (67), 47 (35), 49 (20), 35 (10), 48 (9), 50 (7), 37 (5)
3-8	1-chloroheptane	91 (100), 43 (50), 55 (49), 41 (40), 69 (31), 93 (29), 57 (25), 56 (18)
4-3	1,1-dichloroheptane	70 (100), 43 (60), 41 (47), 55 (42), 27 (38), 67 (22), 81 (20), 29 (19)
4-4	1-chloro-2-methylbutane	70 (100), 43 (95), 41 (90), 55 (71), 42 (65), 27 (45), 29 (40), 39 (35)
10-13	<i>o</i> -chloroaniline	127 (100), 65 (37), 129 (35), 92 (30), 128 (25), 91 (22), 64 (17), 63 (15)
10-25	2-chlorobiphenyl	188 (100), 152 (33), 153 (32), 190 (30), 189 (20), 76 (18), 151 (15), 63 (10)
11-6	2-bromo-5-ethylnonane	57 (100), 55 (60), 43 (59), 69 (44), 41 (32), 71 (28), 97 (18), 85 (16)
11-12	<i>p</i> -chloroaniline	127 (100), 65 (35), 129 (32), 92 (25), 39 (15), 63 (12), 100 (12), 128 (10)
12-6	1-iodooctadecane	57 (100), 71 (72), 43 (52), 85 (50), 41 (42), 55 (32), 69 (27), 99 (18)
13-7	1-chlorohexadecane	57 (100), 43 (90), 71 (56), 41 (49), 91 (39), 55 (22), 85 (18), 69 (16)

^a The first, second, and third numerals indicate the number of gas chromatographic peaks during the original broad gas chromatographic fractionation, the second chromatography, and the third chromatography, respectively.

The volatile compounds were isolated from 540 lb of baked Russet Burbank potatoes. After extensive gas chromatographic fractionation, the relative pure fractions were analyzed by GC-mass and infrared spectrometry.

The eight major mass fragmentation ions of each halogenated compounds identified are listed in Table I.

Since most of the halogenated compounds analyzed did not have major functional group peaks in their IR spectra, the fingerprint matching identification technique was used.

RESULTS AND DISCUSSION

Fourteen halogenated compounds identified in the volatile compounds of baked potatoes are listed in Table I. The eight major mass fragmentation ions of each compound are also listed in Table I. The mass spectra of these compounds are in good agreement with the published data (Eight Peak Index of Mass Spectra, 1974).

There are two possibilities for the formation of these halogen compounds which were identified in baked potato. The first is the persistence of now banned insecticides in established turf. Since 1943, many chlorinated hydrocarbon insecticides have been used including DDT, toxaphene, chlordane, dieldrin, aldrin, and heptachlor. These chlorinated hydrocarbons are not very volatile and are persistent in the soil (Brown, 1978). Second, some halogenated insecticides and fungicides are still in use on potatoes used for baking. The decomposition of pesticide residues or the interaction of pesticide residues with potato during baking may lead to the formation of the halogenated compounds identified.

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Trypsin-Inhibitor and Chymotrypsin-Inhibitor Studies with Soybean Extracts

The effect of dry and wet heat treatments on proteinase inhibitors of soybean flour was studied. The inhibitors were extracted from the flour by physiological saline before or after the heat treatment. The inhibitors showed a higher heat resistance in the dry flour than after extraction by saline. The heat inactivation curve of trypsin- and chymotrypsin-inhibiting capacities differed more in the case of the wet treatment. It was also found that trypsin and α -chymotrypsin could release themselves from the inhibition to some extent on standing for long periods.

Raw soybean flour contains several antinutritive substances, among others proteinase inhibitors. The latter is composed of two major fractions: the inhibitors of Kunitz (1945) and of Bowman and Birk (Bowman, 1946; Birk et al., 1963). The former inhibits trypsin well and α -chymotrypsin slightly, while the latter is effective on both enzymes.

In the present experiments the total trypsin- and chymotrypsin-inhibiting capacities of soybean flour were compared under different modes of heat inactivation. The effect of incubation time on the proteinase-soybean inhibitor system was also studied.

EXPERIMENTAL SECTION

Preparation of Samples. Fat-containing soybean flour with 93% dry matter was used throughout. Samples (0.5 g) of flour were weighed into Erlenmeyer flasks, and 24.5 mL of physiological saline was added. The flasks stood at room temperature for 4 h and were shaken periodically by hand. Then they were kept in a refrigerator at 4 °C overnight. The next day the samples were shaken again periodically at room temperature for 1 h, centrifuged, and frozen until used. In experiment 1, heated soybean flour samples were extracted; in experiment 2 and 3 unheated