Spurious GLC Peaks in Cereal Grains Stored in Cloth Bags

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During the course of routine analysis of Canadian cereal grains for chlorinated hydrocarbon residues, it was observed that samples handled in cloth bags consistently produced multipeaked chromatograms and that these chromatograms had similar peak patterns. The occurrence of multipeaked chromatograms was associated only with the use of cloth bag containers. Samples stored in kraft envelopes never exhibited comparable glc peaks.

Since it was possible that spurious results on cereal grains were being derived from storage in cloth bags, an investigation of these containers became necessary. Details and results of this investigation are given in this paper.

The cloth bags in question were used for handling carlot samples. These samples were required for grading purposes, but occasionally they were also used to determine the pesticide content of grain in suspect carlots. It was observed that most cloth bags in use were soiled to some degree, probably due to continual reuse.

Methods

Pesticide analysis of cereal grain

Chlorinated hydrocarbon analysis of whole grain was performed by an electron capture glc technique. Grain samples were processed according to the method of Levi et al (1). Aliquots of 5 µl or less of the eluant concentrates were injected onto a 3% SE-30 column in an MT-220 gas chromatograph equipped with an Ni-63 detector.

Analysis of cloth bags

Cloth bags were tested for the presence of electron capturing residues which could interfere with glc-ecd analysis for chlorinated hydrocarbons. Soiled used cloth bags and clean unused cloth bags were stripped with a 3% ethyl ether in hexane solvent system and the extracts were cleaned up by the florisil column technique described by Levi et al (1). Aliquots of 1 µl of the unconcentrated eluants were injected into the previously described gas chromatograph.

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Experiment to compare effect of storage in cloth bags to storage in kraft envelopes

Four wheat samples were divided into two portions. One portion weighing approximately two pounds was stored in a cloth bag for one week. The other portion weighing approximately 200 g was stored in a kraft envelope for the same period of time. Ten grams of grain from both portions of each sample were analysed by the procedure (1) for pesticide analysis of cereal grains.

Results

Figure 1 shows glc results for two cereal grain samples analysed for chlorinated hydrocarbon residues. These chromatograms are representative of glc results for most samples handled in cloth bags and kraft envelopes. Samples stored in cloth bags exhibit numerous high chromatogram peaks, however only minor peaking arises from samples stored in kraft envelopes.

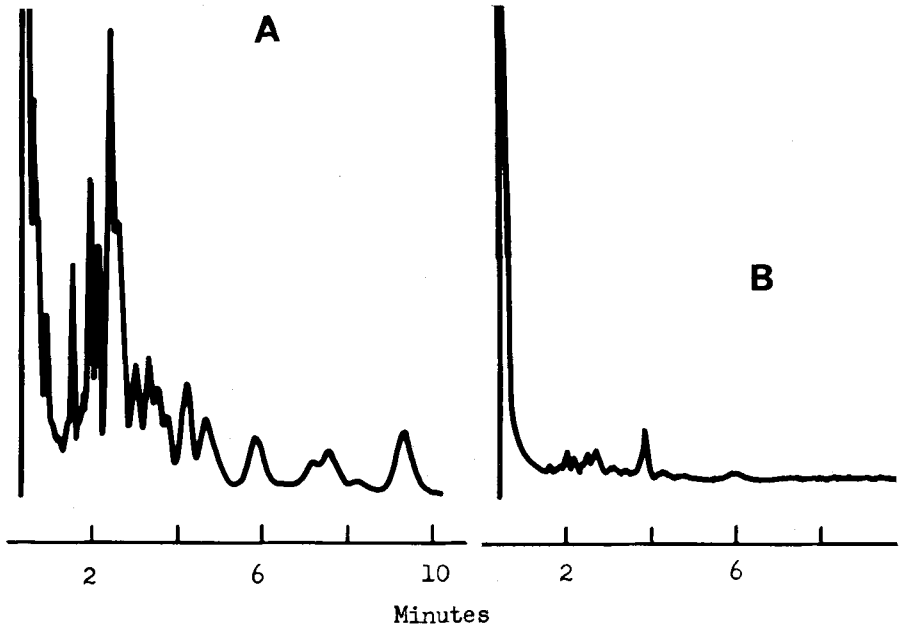

Figure 1. Representative gas chromatograms of cereal grain samples. (A) sample stored in a cloth bag; (B) sample stored in a kraft envelope.

Figure 2 shows chromatograms from the analysis of a soiled cloth bag and an unused cloth bag. All soiled cloth bags produced chromatograms with many high peaks, but only some of the unused cloth bags produced chromatograms displaying peaks. The soiled cloth bags obviously produce more early eluting glc peaks than the unused cloth bags and contain higher levels of other peaks common to both chromatograms.

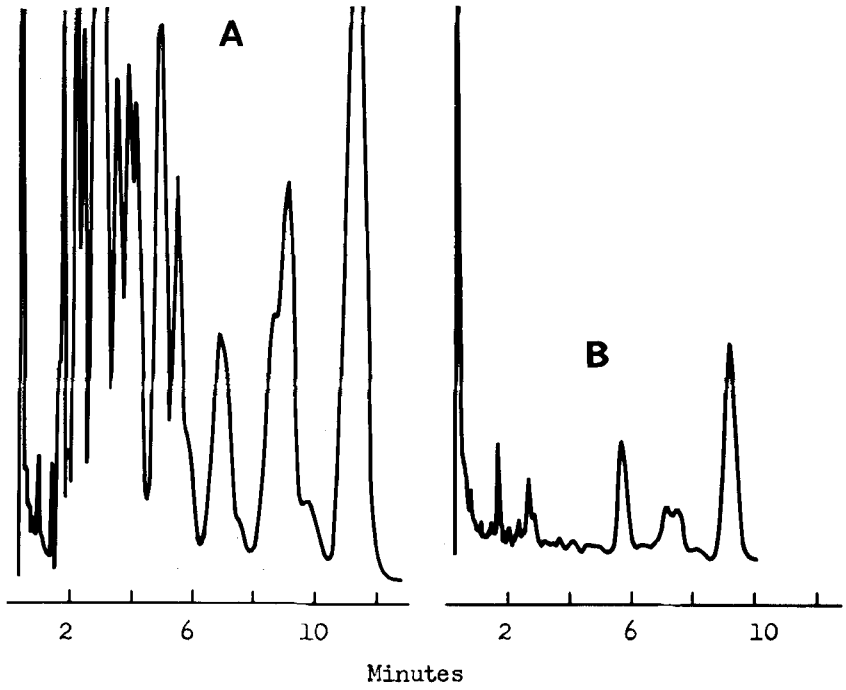

Figure 2. Gas chromatograms of cloth bag material. (A) used soiled cloth bag; (B) clean unused cloth bag.

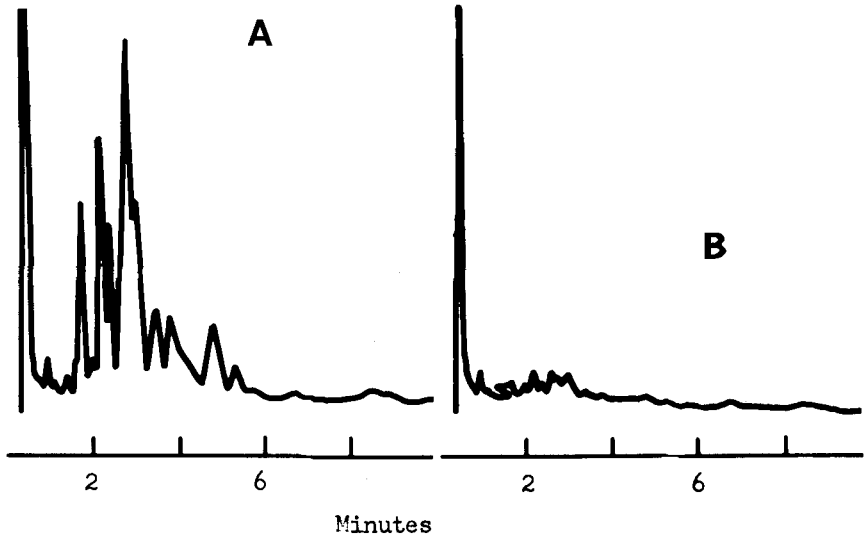

Figure 3. Gas chromatograms of a wheat sample stored in two different containers. (A) storage in a cloth bag; (B) storage in a kraft envelope.

In Fig. 2(A) some of the chromatographic peaks have retention times identical to those of known pesticides. Two of the earlier peaks resemble HCB, and PCNB, and some of the later peaks are identical in retention time to p'p' DDE, P'P' DDD, O'P' DDT, and P'P'DDT.

Chromatographic results of both portions of one of the four divided samples are shown in Fig. 3. All four samples displayed high peaks for the bagged samples and no peaks for the samples kept in kraft envelopes.

Discussion

It is obvious that cloth bags can contain residues which react to an electron capture detector and that cereal grains stored in cloth bags absorb these peak producing contaminants. Accordingly cloth bags either directly or indirectly contaminate whole grain wheat and barley samples.

For our purpose, it was not necessary to chemically identify the contaminants. Eliminating the use of cloth bags as containers for cereal grain samples destined for pesticide residue analysis avoided this contamination problem.

One can only speculate as to how cloth bags become contaminated. The buildup of dust may be an important factor. Dust from cereal grain can easily build up on the inside of the bags and dust from environment can easily accumulate on the outside.

Acknowledgments

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References

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