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Report ORF 66-1

April 22, 1966

Determination of endrin in cigarette smoke condensate

for
Imperial Tobacco Company
of Canada, Limited
Montreal 30, Quebec
Attention: Mr. R.S. Wade

Introduction

Two samples of cigarette smoke condensate received from Imperial Tobacco Company of Canada, Limited, on March 2, 1966, were analysed for residues of endrin.

Because of the very difficult problem of cleaning up the sample prior to the final determinative step, several preliminary experiments were undertaken to determine recoveries when known amounts of endrin were added to control samples. The method used by Mr. Ralf Miles of the Entomological Laboratory in Chatham (essentially that of the Shell Development Company method) for the determination of endrin on tobacco proved unsatisfactory. The cleanup method which was used eventually, consists of three main parts and is described below.

Method

1. Freezing out of fatty materials at -75°C.

The condensate was transferred to a 500 ml flask with n-hexane and the solvent removed with a rotary evaporator at 45°C with vacuum. The residue was dissolved in benzene:acetone (1:19, 130 ml) and cooled

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to -75°C in a methanol-dry ice bath. The cold solution was stirred for 30 minutes and then filtered through a carbon:Solka floc pad (2 gm: 10 gm) using a sintered, jacketed glass funnel through which the cold methanol was circulated. The filtrate was dried with Na_2SO_4 and the solvent removed with a rotary evaporator. The residue was then dissolved in a small volume of n-hexane.

2. Florisil column cleanup

The residue dissolved in hexane was transferred to a chromatographic column containing 30 gm Florisil (reactivated at 1200°F) topped with about 1/2" layer of anhydrous Na_2SO_4 and which had been pre-wetted with about 40 ml of hexane. The container was rinsed with 3 portions of about 5 ml each of hexane and poured into the column, which was then eluted with 200 ml of 6% ether in hexane at a rate of about 5 ml per minute (this results in eluate "a" as shown in the accompanying scheme). The receiver was then changed and the column eluted further with 300 ml of 15% ether in hexane (eluate "b").

Both eluates were concentrated to almost dryness and were then given a further cleanup using a charcoal-Solka floc column.

3. Charcoal-Solka floc column cleanup

The concentrated eluates were transferred to a chromatographic column containing charcoal:Solka floc (5 gm:10 gm and prepared by making a slurry with 10% acetone in hexane) which was supported by a glass wool plug and 1/2" layer of anhydrous Na_2SO_4 . The column was eluted with 250 ml of 10% acetone in hexane. The eluates were concentrated and made to 5 ml volume with hexane.

Final analysis was carried out by gas liquid chromatography (GLC) using an electron capture detector, while thin layer chromatography (TLC) was used to confirm the results.

A recovery of 85.7% was obtained when a known amount (8 µg) of endrin was added to an untreated control sample and subjected to the cleanup procedure.

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Results and Discussion

<u>Sample</u>	<u>Endrin (p.p.m)</u>
RL-183 (Control)	None detected
RL-183 A (Endrin treated)	0.13

The sensitivity of the method for practical purposes is about 0.02 p.p.m. when the condensate from 200 cigarettes (~ 200 gm) is used.

The estimation is based on total area under the two GLC peaks for endrin and on the assumption that 1 cigarette weighs 1 gm approximately.

During the recovery experiments, no endrin was recovered in eluate "a" (see accompanying scheme) hence no attempt was made to qualify or quantitate any of the pesticides in this fraction.

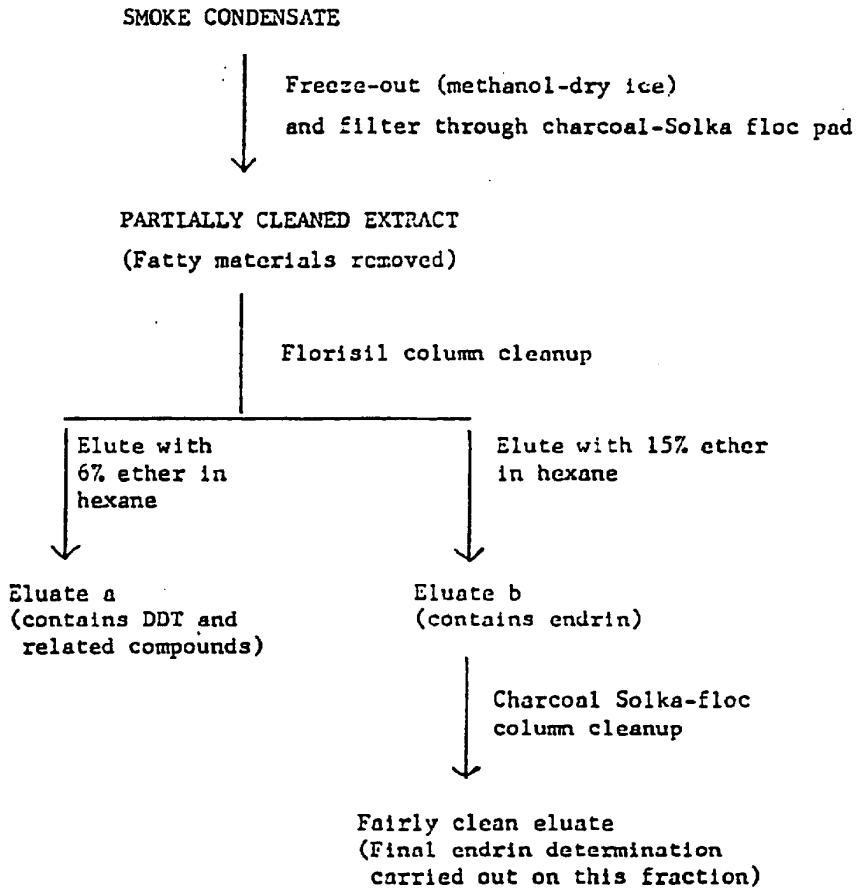
L.M. Reynolds
L.M. Reynolds / *en*
Research Scientist

Attached: 1 scheme
cc: Dr. J.E. deSouza

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SCHEME FOR THE ISOLATION OF ENDRIN



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