

MEAT AND MEAT PRODUCTS-DETERMINATION
BY GAS-LIQUID CHROMATOGRAPHY OF THE
RESIDUES OF THE BHC AND DDT

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INTRODUCTION

BHC and DDT are the two kinds of pesticide widely used the latest dozens of years. Though they had been stopped using since 1983. There are pesticide residues to a different degree in natural, soils, plants and bodies of animals. Because they are difficult to be decomposed. These residues directly harm the health of human. This report describe the method of Gas Chromatograph for determination of BHC and DDT in the meat and meat products. Preparation of meat sample are as follows: Extraction, Purification and Concentrate. Then the sample is passed 0.8% OV-17 + 3% OV-210 /Chromosorb G (60-80 mesh) (AW-DMCS) mixture solid phase (Dia 3mm x L 1.6m Glass column.)

This method can achieve linear separation and quantitation analysis of eight compositions, including the four BHC isomerides (alpha-BHC, beta-BHC, gamma-BHC, delta-BHC.), two DDT isomerides (o.p'-DDT, p.p'-DDT.) and p.p'-DDE and p.p'-DDD.

EXPERIMENTAL METHODS

A. Extration:

weigh 10g meat sample to mince, add 25 ml 1:1 (V/V) Gaolusuan / Acetic acid vibrate 20 minute on the 80°C water bath. Then the acid liquids were washed by water; add 100 ml N-Hexane to the sample mixtur to extract pesticide from acid liquids.

B. Purification:

Add 1/10 (V/V) H₂SO₄ to the extration liquids and purify 2-5 times. Throw away acid layer. again add 100 ml 2% NaSO₄ (absolute) solutions to it for washing extration liquides. Then use NaSO₄ (absolute) for deprivation.

C. Concentration:

Concentrate the purified extration liquids from 100 ml to 1 ml with K-D

concentrator.

D. GC determination:

a. preparation of column:

The column with single solid-liquid can't separate eight compositions. We have packed both columns of 2% OV-17 / Chromosorb G and 5% OV-210 / Chromosorb G to analyse the standard sample of the BHC and DDT. o.p'-DDT and p.p'-DDD can't be separated with 2% OV-17 column. (Fig 1). p.p'-DDD and p.p'-DDT can't be separated with 5% OV-210 column. (Fig 2). Hence we had made tests for the rate of mixture solid liquids with both methods of the diagrams (Fig 3) and formula (Fig 4). Test proving: if 40% solid phase of 2% OV-17 and 60% solid phase of 5% OV-210 were mixed together to pack into Glass column (Dia 3mm x L 1.6 m). the eight compositions can be separated ideally. (Fig 5).

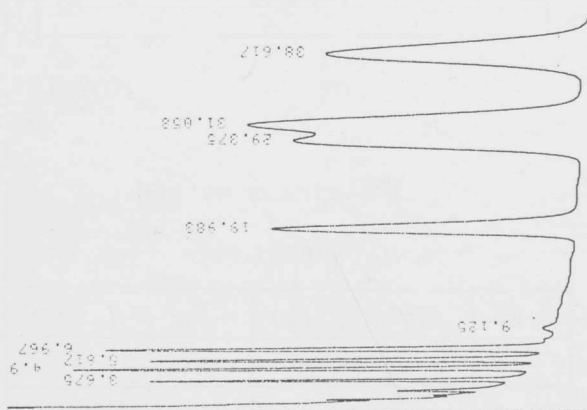


Fig 1 2% OV-17 column

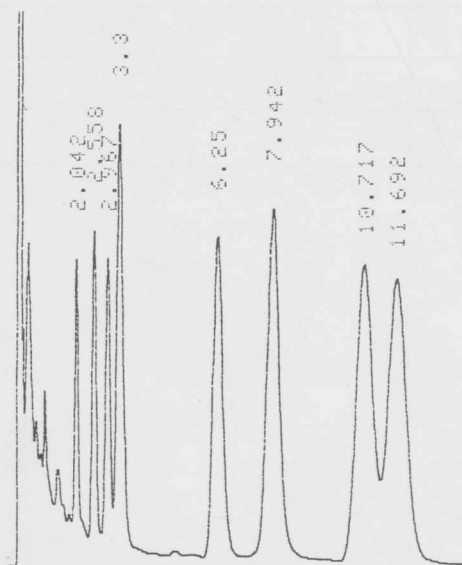


Fig 2 5% OV-210 column

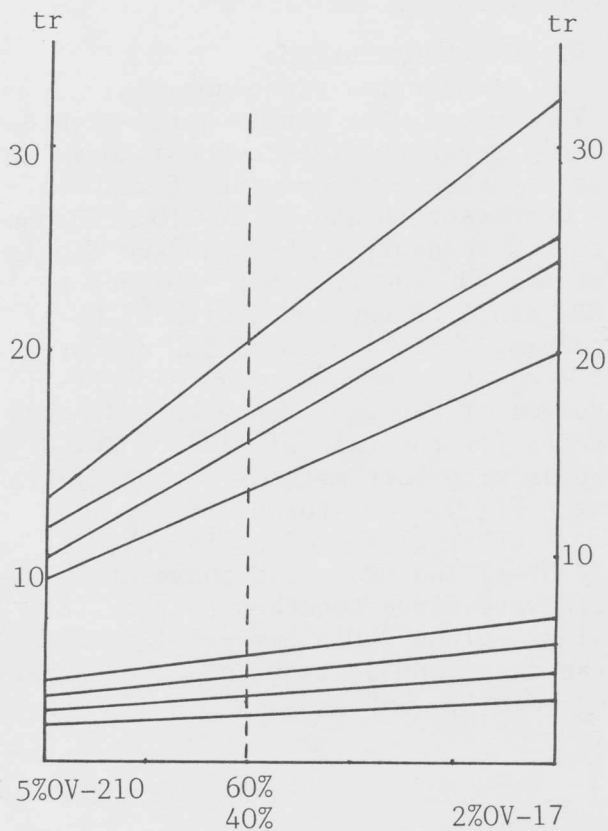


Fig 3 diagrams method

R date table

R	tr 6 / tr7	tr 7 / tr 8
0.0	1.09	1.29
0.2	1.12	1.20
0.4	1.15	1.15
0.6	1.17	1.08
0.8	1.20	1.04
1.0	1.22	1.00

note: tr 6 -- o.p'-DDT
tr 7 -- p.p'-DDD
tr 8 -- p.p'-DDT

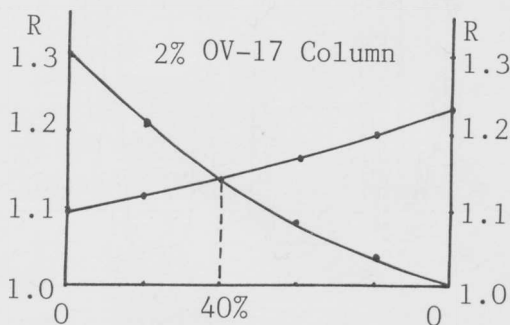
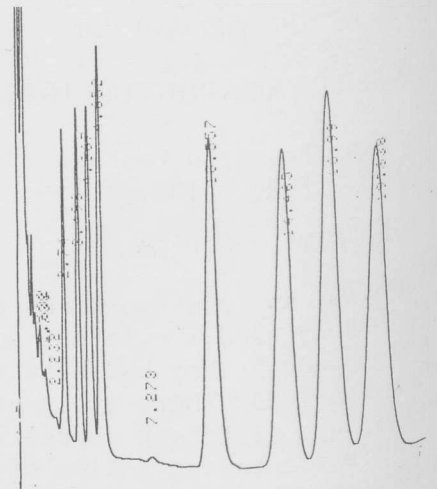


Fig 4 formula method



- 1.alpha-BHC
 - 2.gamma-BHC
 - 3.bata-BHC
 - 4.dalta-BHC
 - 5.p.p'-DDE
 - 6.o.p'-DDT
 - 7.p.p'-DDD
 - 8.p.p'-DDT
- Fig 5 GC-figure of eight composition

b. GC condition:

Instrument: GC - 9AM
(Shiamdzu Japane)
Detector: ECD
Column: 0.8% OV-17 + 3% OV-210
/Chromosorb G (60-80 mesh)
Injection temperature: 250°C
Detector temperature: 230°C
Column temperature: 215°C

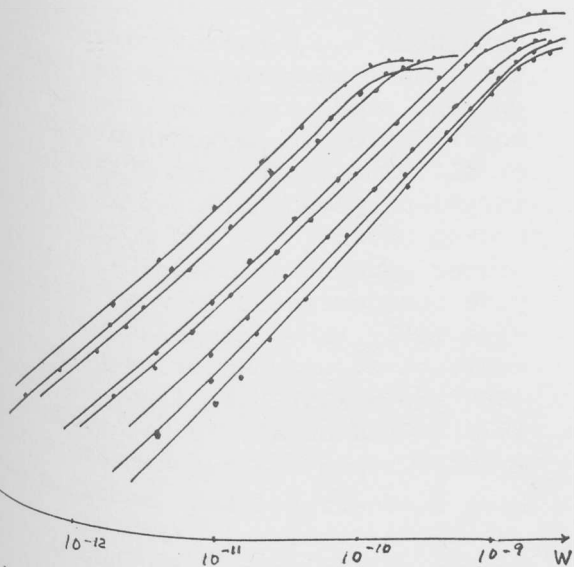
c. Quantition formula:

$$X_i = A_i \times 1000 / M \times V_2 / V_1 \times 1000$$

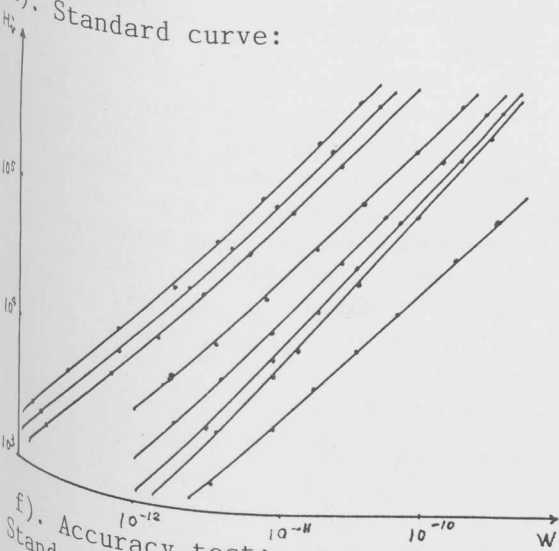
X_i : single contents of sample composition i (mg/kg)
 A_i : single contents of standard composition i (ng)
 V_2 : volumn of sample concentration composition i (ml)
 V_1 : injection weight (ug)
 M : weight of the sample (g).

d. Method verification:

- a). Instrument sensitivity:
 1.2×10^{-13} g/ml
- b). Minimum detectable sample:
 0.12×10^{-4} ppm
- c). Linearity range:
about $10^3 - 10^4$
(Fig 6)



d). Standard curve:



f). Accuracy test:
Standard sample of the difference concentration were added into meat sample to do retrieve rate test. The result as follows:

Table 1. Retrieve rate test date

Ci	Si	meat (fat 10%)	meat (fat 10%)	Jin Hua ham	Beer ham
		(pork)	(pork)	(Pork)	(pork)
alpha -BHC		85%	83%	85%	79%
bata -BHC		80%	81%	82%	81%
gamma -BHC		84%	77%	88%	88%
dalte -BHC		78%	97%	90%	89%
p.p' -DDE		84%	92%	84%	81%
p.p' -DDT		89%	85%	79%	105%
p.p' -DDD		92%	83%	86%	92%
p.p' -DDT		87%	103%	92%	75%

f). Precision test:

To replicate injection sample 10 times and handle determination datumes as follows:

Table 2 Coefficient of variation

Si	meat (fat 10%)	meat (fat 10%)	JinHua ham	Beer ham
alpha -BHC	5.1%	4.6%	6.3%	4.2%
bata -BHC	8.2%	9.9%	8.5%	8.6%
gamma -BHC	/	/	/	/
dalta -BHC	/	/	/	/
p.p' -DDE	10%	9.6%	7.8%	9.6%
o.p' -DDT	/	8.8%	9.2%	/
p.p' -DDD	9,7%	9.4%	8.9%	9,5%
p.p' -DDT	9.1%	9.1%	9.3%	/

RESULTS

We have determinated the BHC and DDT pesticide residues in several kinds of meat and meat produces. All the date below limit weight of hygiene standard of nation Result as follows:

Table 3 Determination date of BHC and DDT of four meat foods (ppm)

Ci	Si	JinHua ham (pork)	Beef	Roast -ed mutton	Chick -en
alpha-BHC		0.040	0.005	0.010	0.064
bata -BHC		0.051	0.004	0.028	0.055
gamma-BHC		/	/	/	/
dalta-BHC		/	/	/	/
p.p' -DDE		0.225	0.007	0.009	0.140
o.p' -DDT		/	/	/	/
p.p' -DDD		0.160	/	0.061	0.056
p.p' -DDT		0.290	/	/	/