Determination of organochlorine pesticide residues in eggs by gel-permeation chromatography (GPC) cleanup and GC-ECD

LabTech, Inc.

1. Experimental Section

1.1 Instruments and reagents

AutoClean automatic gel-permeation chromatography system (LabTech, Inc., Boston, USA)
EV331 rotary evaporator (Beijing LabTech Instrument Co., Ltd., Beijing)
GC-ECD
Vortex mixer
Nitrogen blowing instrument
Homogenizer
Ethyl acetate (redistilled for use)
Cyclohexane (redistilled for use)
Acetone (redistilled for use)
Sodium chloride (baked for 4h at 140 ℃)
Anhydrous sodium sulfate (baked for 4h at 140 ℃)

1.2 Sample Preparation

1.2.1 Sample

Take off egg shell, homogenized.

1.2.2 Extraction

Accurately weigh 20.00g of homogenized egg sample to 200ml Erlenmeyer flask, add 5ml of water, 40ml of acetone, shaking 30min, adding 6g of sodium chloride, shake well, then add 30ml of petroleum ether, shaking 30min, after standing layer, the organic phase all transferred to 100ml, dehydrate with anhydrous sodium sulfate, and take 35ml in a rotary evaporator and concentrated to 1ml, add 2ml of ethyl acetate - cyclohexane (1 + 1), and the solution was then concentrated, so repeated three times, final volume to 10ml with ethyl acetate - cyclohexane (1 + 1), to be cleanup.

1.2.3 Cleanup

GPC cleanup conditions:
Flow rate: 5ml / min
Cleanup column: 20 × 300mm SX-3
Mobile phase: ethyl acetate - cyclohexane (1 + 1)
Collection time: 12min-16min
The cleaned samples was collected in the nitrogen blowing instrument, evaporated to near dryness under nitrogen blowing at 50 ℃, to volume 1ml with n-hexane, for further GC analysis.

1.3 Instrument analysis conditions

1.3.1 Chromatographic conditions

Gas: GC-ECD
Column: TM-Pesticide 1 30m × 0.53mm × 1.0um
Column temperature program: heating at 170 ℃ for 1 minute, and then heat to 210 ℃ with the rate of 5 ℃ / min, and then heat to 230 ℃ with the rate of 2 ℃ / min, keeping on 10min.

Sample loading Inlet temperature: 280 ℃
Detector temperature: 300 ℃
Split ratio: splitless
Injection volume: 1 uL

1.3.2 GC-ECD spectra

Chromatogram result under the chromatographic conditions are as follows:

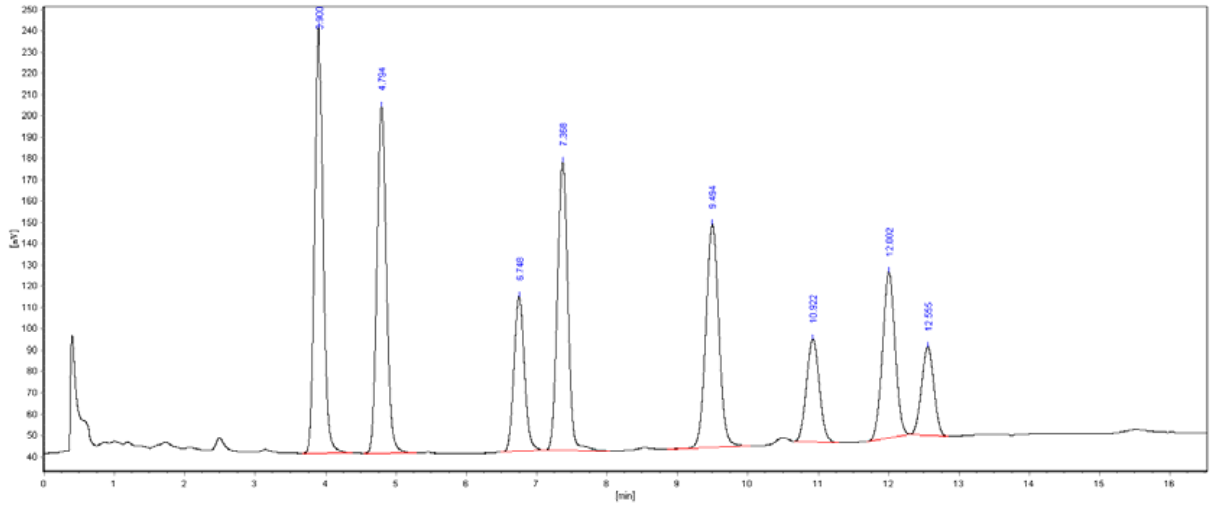

Figure 1, 8 GC-ECD chromatogram of organochlorine pesticides
(From left to right: α-666; β-666; γ-666; δ-666; P, P-DDT; O, P-DDT; P, P-DDE; P, P-DDD)

2. Results and discussion

2.1 Standard curve

2.2.1 Optimization of GPC cleanup conditions

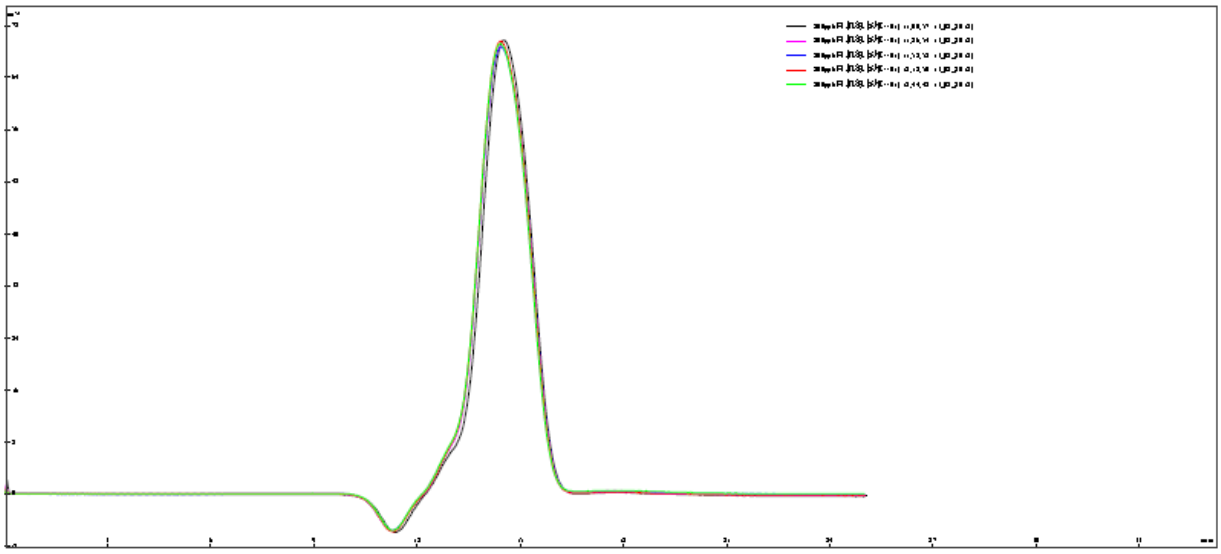

Figure 2. GPC cleanup reproducibility

Take standard mixture of 8 organochlorine pesticides, dilute to 200ppb with ethyl acetate: cyclohexane (1 + 1), as a GPC cleanup loading sample, using a UV detector at 254nm conditions to detect and get chromatogram shown in Figure 2; Loading sample 5 consecutive times, good reproducibility. Identify collection time of 12min-16min.

2.1.2 Optimization of the chromatographic conditions

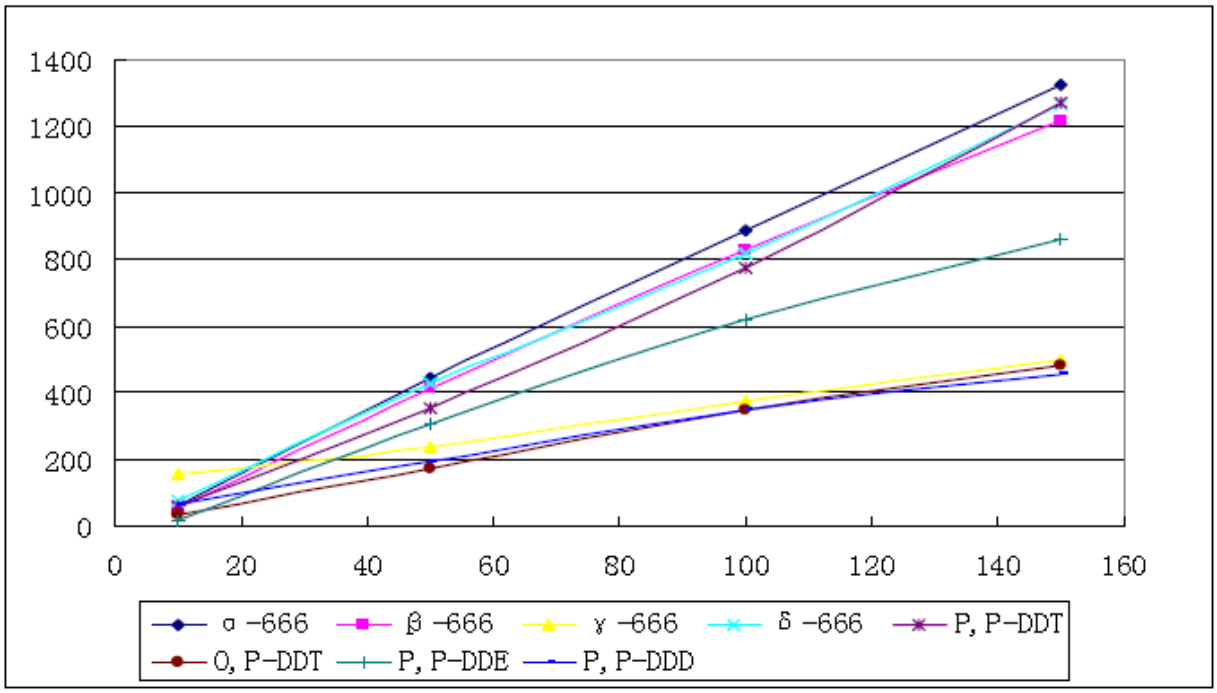

Figure 3. Regression curves of 8 organochlorine pesticides

To calculate the standard curve regression equation and linear range by setting sample concentration as abscissa (X) and the peak area of the vertical axis (Y). In the range of 10ppb-150ppb, linearity is good. The linear equation are:

Pesticide	Regression equation	Regression coefficient
α-666	y = 9.0287x - 21.465	R^2 = 0.9996
β-666	y = 8.3155x - 15.119	R^2 = 0.9993
γ-666	y = 2.4953x + 120.71	R^2 = 0.9965
δ-666	y = 8.4382x - 8.4691	R^2 = 0.9994
P,P-DDT	y = 8.6550x - 7.7220	R^2 = 0.9958
O,P-DDT	y = 3.2334x + 7.0878	R^2 = 0.9960
P,P-DDE	y = 6.0085x - 14.612	R^2 = 0.9922
P,P-DDD	y = 2.8035x + 46.326	R^2 = 0.9903

2.2 Recovery precision

In the optimization conditions, standards are added in a sample, and cleanup by LabTech AutoClean GPC system, GC-ECD analysis and the resulting spectrum as shown in Figure 4.

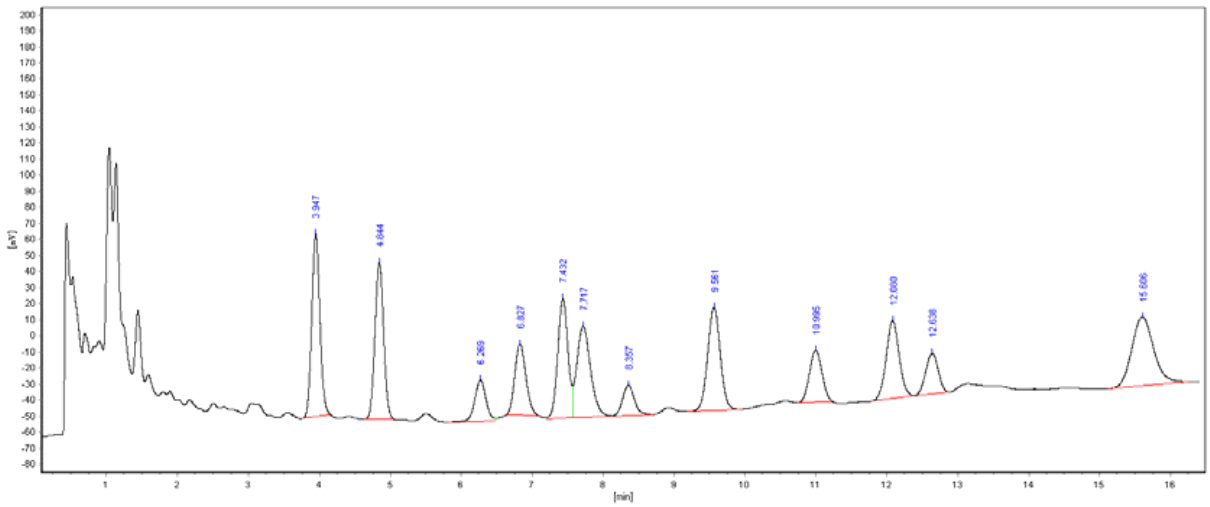

Figure 4. GC-ECD spectra of 8 organochlorine standards in egg by GPC cleanup

2.2.1 Recovery rate of standards

Recovery experiment is tested for the accuracy of determination of organochlorine pesticides in foods by spiking and recovery experiments, and results are shown in the below.

Number	Pesticide	Addition amount (mg/kg)	Measured amount (mg/kg)	Recovery (%)
1	α-666	0.1	0.108	108.3
2	β-666	0.1	0.108	107.6

3	γ-666	0.1	0.120	119.5
4	δ-666	0.1	0.102	101.9
5	P,P-DDT	0.1	0.086	86.1
6	O,P-DDT	0.1	0.111	111.0
7	P,P-DDE	0.1	0.104	103.9
8	P,P-DDD	0.1	0.088	87.8

2.3.1 Results of real sample

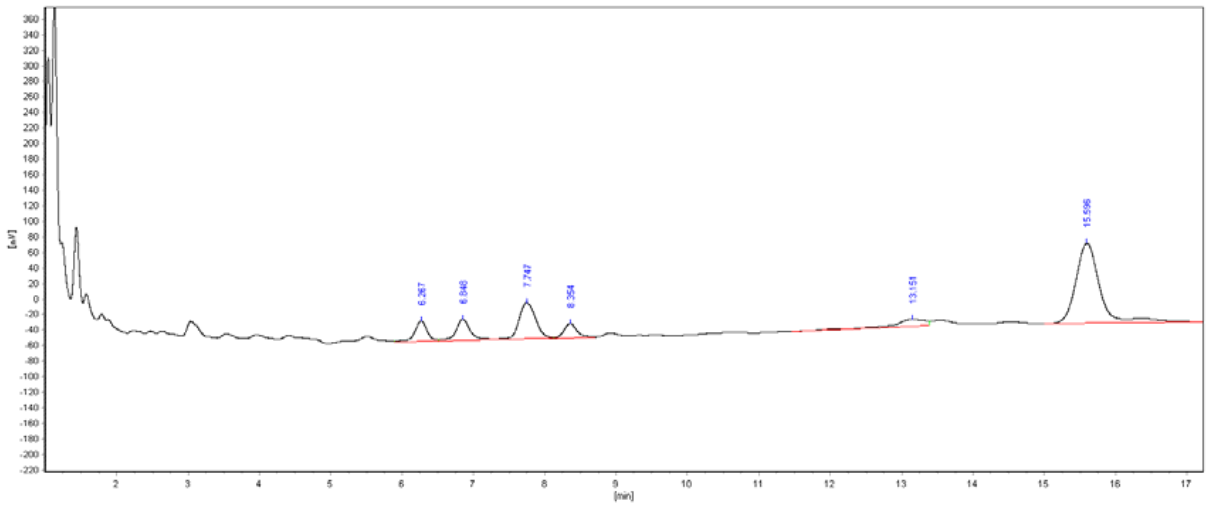

Figure 5. GC-ECD chromagram of egg sample after cleanup

3. Conclusion

In this application note, a method is created to cleanup and analyze of 8 organochlorine pesticide residues in eggs. The operation is convenient and the recovery rate is ideal.

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