

Survey on 3-Monochloro-1,2-propandiol (3-MCPD) Contents of Soy Sauce Products during Fiscal Year 2002 in Taiwan

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(Received: February 5, 2004; Accepted: July 2, 2004)

ABSTRACT

A survey of 3-monochloro-1,2-propandiol (3-MCPD) content in soy sauce products is reported for determining the distributions of 3-MCPD concentrations. Two hundred and fourteen samples of soy sauce products, including 188 domestic and 26 imported soy sauce products were analyzed using a GC/MS method (detection limit 0.01 ppm) that was validated and promulgated by the Department of Health to check for compliance with the government's recommended limit of 1 ppm for 3-MCPD in 2002. Results indicated that 3-MCPD was undetectable in 87 samples of domestic soy sauce products and 23 samples of imported soy sauce products 3-MCPD. Ninety-one samples of domestic soy sauce products contained 3-MCPD at concentrations in the range of 0.01 ppm to 1 ppm while 10 samples of domestic soy sauce products were found to contain 3-MCPD at concentrations greater than 1 ppm. Only 3 samples of imported soy sauce products contained 3-MCPD at concentrations in the range of 0.01 ppm to 0.03 ppm. 3-MCPD contents of domestic soy sauce products in 2002 decreased significantly in comparison with the surveyed contents in 2000. Ten domestic soy sauce products containing 3-MCPD over the regulation limits set by the government were sampled and reanalyzed again after improvements made by the manufacturers. 3-MCPD concentrations ranged from 0.02 ppm to 0.11 ppm in 5 samples and were undetectable in 4 samples, while 1 sample was no longer produced after the survey.

Key words: soy sauce, 3-monochloro-1,2-propandiol, 3-MCPD, GC/MS

INTRODUCTION

3-monochloro-1,2-propandiol (3-MCPD) is a member of a group of chemical contaminants known as chloropropanols. It is formed as a product of acid hydrolysis of vegetable proteins during the manufacturing of food seasonings when ingredients hydrolyze vegetable proteins⁽¹⁾. Several studies reported that 3-MCPD could be detected as contaminants in several food and food ingredients, including soy sauce products, and hydrolyzed vegetable protein (HVP)^(2,3). 3-MCPD could also be formed in other food products during storage and processing⁽⁴⁾. Soy sauces or acid HVPs exported from some Asian countries found to contain high levels of 3-MCPD⁽⁵⁾ have been rejected from European countries.

Toxicological studies had shown that 3-MCPD is carcinogenic in rat and has genotoxic activity *in-vitro*. 3-MCPD may be metabolized to genotoxic intermediates, particularly glycidol. The available evidence indicates that 3-MCPD is not a genotoxic carcinogen, but rather a chemical that causes tumors in rats through nongenotoxic mechanisms⁽⁶⁾. 3-MCPD has been identified by the European Union Scientific Committee for Food as a potential genotoxic carcinogen in 1994⁽⁴⁾. The EC committee set a Tolerable Daily Intake (TDI) for 2 $\mu\text{g}/\text{kg}$ bw based on the threshold-based approach in 2001⁽⁷⁾. The

UK Food Advisory Committee recommended that the industry take all steps to ensure that 3-MCPD is undetectable in food⁽⁸⁾. Recently, the UK Committee on Mutagenicity and Carcinogenicity concluded that 3-MCPD was an *in-vitro* mutagen but had no significant genotoxic potential *in-vivo* after reviewing all the available mutagenicity and carcinogenicity data^(9,10).

Several analytical methods for 3-MCPD determination have been demonstrated in many studies^(11,12,13). Gas chromatography with Mass was frequently used as the qualitative and quantitative analysis method. We also set up the method for the 3-MCPD determination in soy sauce products by ethyl acetate extraction and direct derivatization with heptafluorobutyl imidazole (HFBI). The method was announced by the Department of Health in 2001⁽¹⁴⁾.

Soy sauce is one of the most important seasonings in Chinese society. As a result, monitoring the 3-MCPD contents is an important job for improving people's health. For the determination of 3-MCPD contents in soy sauce products, the Department of Health set a safety regulation on 3-MCPD soy sauce products at 1 ppm⁽¹⁵⁾. The aim of this study was to understand the distributions of 3-MCPD contents in soy sauce products sold in Taiwan. The study also compared results from different cities, years, countries and 3 different kinds of soy sauce products. The result could provide information for government regulations and apply pressure on the manufacturers to improve the quality of soy sauce products by decreasing the 3-MCPD content.

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MATERIALS AND METHODS

I. Sample Collection

Two hundred and fourteen samples of soy sauce products, including 188 domestic and 26 imported soy sauce products were sampled from manufacturers or purchased from retail outlets by the local hygiene bureaus in 18 cities and prefectures.

II. Reagents and Chemicals

Ethyl acetate and *n*-hexane were both pesticide residue grade; anhydrous sodium sulfate and sodium chloride were AR grade, and Extrelut[®] NT20 column were purchased from Merck; 3-chloro-1,2-propanediol was from Aldrich. Heptafluorobutyl imidazole (HFBI) was from Sigma and *p*-dichlorobenzene (PDCB) was from Chem Service.

III. Instrument and Apparatus

- (I) Gas chromatograph / electron impact mass spectrometer
- (II) Rotary evaporator
- (III) Gas-tight syringe
- (IV) Shaker bath
- (V) Ultrasonic vibrator
- (VI) Micro reaction vessels (screw cap septum vials, 5 mL)

IV. Methods

(I) Internal standard solution preparation

One hundred milligram of PDCB was weighed to a 100-mL volumetric flask and made up to volume with *n*-hexane. The stock solution was diluted with *n*-hexane to 0.05 $\mu\text{g/mL}$ to be served as the internal standard solution.

(II) Standard solution preparation

One hundred milligram of 3-MCPD was weighed to a 100-mL volumetric flask and made up to volume with ethyl acetate. The stock 3-MCPD solution was diluted with *n*-hexane to 0.025, 0.05, 0.1, 0.2, 0.5 and 1.0 $\mu\text{g/mL}$, each with an internal standard of 0.05 $\mu\text{g/mL}$.

(III) Sample preparation

Five gram of soy sauce was weighed into a glass beaker; 15 mL of 5 M sodium chloride solution was added, mixed well and sonicated in an ultrasonic bath for 15 min. Prepared samples were then applied to an Extrelut[®] NT20 column and allowed the samples to spread over the inert matrix as a thin layer for 15 min. The column was then eluted with 250 mL of ethyl acetate. The liquid elute was shaken well with 30 g of anhydrous sodium sulfate, allowed to stand for 15 min, then filtered through a glass funnel containing 50 g of anhydrous sodium sulfate. The sodium sulfate was rinsed with 50

mL of ethyl acetate and the volume of the combined extract was reduced to 3 mL with a rotary evaporator at 40°C. The extract was quantitatively transferred to a graduated tube and made up to 5 mL with ethyl acetate.

(IV) Derivatization

For derivatization, 1 mL of the sample extract was transferred to a micro reaction vessel. Sample extract was blown to dryness under a gentle stream of nitrogen before 1 mL of internal standard solution was added into the micro reaction vessel. Fifty microliter of HFBI was added and the vessel was capped, shaken and incubated at 70°C for 20 min. The reaction mixture was cooled to room temperature, 2 mL of deionized water was added and the vessel was shaken for 30 sec. The organic phase was collected and dried with 300 mg of anhydrous sodium sulfate and was then applied to GC/MS analysis.

(V) Gas chromatography-mass spectrometry analysis

A Hewlett-Packard 5890 gas chromatograph/5970B mass selective detector (GC/MS) equipped with an HP-G1034C Chemstation software was used for this study. The gas chromatograph was equipped with a 30 m \times 0.25 mm i.d. HP-5MS column with a 0.25 μm film thickness. The oven temperature was programmed at 50°C, 5°C/min to 100°C, 30°C/min to 280°C, hold for 5 min; injector 250°C. The following parameters were used for injecting samples into the GC-MS system⁽¹⁴⁾: sample size, 1 μL ; injection mode, split; injector purge-off duration, 0.75 min. The carrier gas was helium and the head pressure was set at 10 psi. The ionization energy was set at 70eV.

(VI) Quantitative analysis

Quantitative analysis was carried out by monitoring characteristic ions at *m/z* 453, 277 and 275 for 3-MCPD; 146, 148 and 113 for PDCB. 3-MCPD was calculated by the internal standard method. The ratios of the peak area response for 3-MCPD (*m/z* 453, 277 and 275) to the peak area response for PDCB (*m/z* 146, 148 and 113) was determined. 3-MCPD were determined from standard curve constructed by plotting the peak area ratios against the concentrations of 3-MCPD.

(VII) Quality assurance and quality control

Quality assurance: The standard curve should be liner with $R^2 \geq 0.995$. The recovery of spiked sample should range between 90% and 110%.

Quality control: The sample peak area ratios should fall within the range of those of the calibration standards for that batch. If not, reduce the suitable volume to less than 1 mL for derivatization. The 3-MCPD-HFBI spectrum of sample should match the 3-MCPD-HFBI spectrum of standard reference.

RESULTS AND DISCUSSION

I. Survey Results

Two hundred and fourteen samples of soy sauce products, including 188 domestic and 26 imported soy sauce products were analyzed by the GC/MS method for 3-MCPD contents. Among the imported soy sauce products, 24 samples were made in Japan and 2 samples were made in Hong Kong.

3-MCPD was undetectable in 87 samples of domestic soy sauce products, but 101 samples of domestic soy sauce products were detected with 3-MCPD. Ten samples (5%) contained 3-MCPD in the concentration range of 0.01 to 0.02 ppm. Fifty-three (28%) samples contained 3-MCPD in the range of 0.03 to 0.10 ppm. Twenty-eight (15%) samples contained 3-MCPD in the range 0.11 to 1 ppm. Ten (5%) samples contained 3-MCPD in the range of 1.01 to 10 ppm. The investigation results of Table 1 show that the 3-MCPD contents in 10 domestic soy sauce products violated the 3-MCPD limitation (1 ppm) set by the government. The highest level of 3-MCPD in the survey samples is 6.63 ppm (Table 2).

3-MCPD was undetectable in 23 samples of imported soy sauce products. Only 3 samples of imported soy sauce products were detected with 3-MCPD. Two (8%) samples contained 3-MCPD at the concentration in range from 0.01 to 0.03 ppm. One (4%) sample contained 3-MCPD in the range of 0.03 to 1.0 ppm (Table 1).

Table 3 shows the comparisons of the 3-MCPD contents of three kinds of soy sauce products in the fiscal year of 2002. In 118 samples of soy sauce products, 65 (55%) samples were not found to contain 3-MCPD, 47 (40%) sample were found to contain 3-MCPD not over the limitation set by the government, and 6 samples were found to contain 3-MCPD exceeding the limitation. In 90 samples of soy sauce paste products, 41 (46%) samples were not found to contain 3-MCPD, 45 (50%) sample were found to contain 3-MCPD which the amounts did not exceed the limitation set by the government, and 4 samples were found to contain 3-MCPD with amounts over the limitation. In 6 samples of reduced salt soy sauce products, 4 (67%) samples were not found to contain 3-MCPD, and 2 sample (33%) were found to contain 3-MCPD but the amounts were not over the limitation set by the government.

Table 4 shows the results of 3-MCPD contents in soy

sauce products sampled in different cities and counties. Soy sauce products from the soy sauce factories or supermarkets were sampled from 18 cities or counties. Pingtung county, Tainan county and Taipei city hygiene bureaus sampled contributed 47 (22%), 34 (16%) and 31 (14%) samples, respectively. All imported soy sauce products were sampled from Taipei city. The 3-MCPD contents did not violate the limitation set by the government. Products containing 3-MCPD were not confined to any single city or county. All the soy sauce products sampled from Taoyuan county were not detected with the 3-MCPD.

II. 3-MCPD Distributions of Soy Sauce Products

Many countries have set the maximum limit for 3-MCPD content in soy sauce products, including USA, Canada, Finland, and Austria (1 ppm), Australia and New Zealand (0.2 ppm), European Union, the Netherlands, Greece, Portugal, Malaysia, and Sweden (0.02 ppm). The

Table 1. Distributions of 3-MCPD contents in soy sauce products of the fiscal year 2002

| 3-MCPD (ppm) | Domestic soy sauce products (%) | Imported soy sauce products (%) | Total |
|-----------------|---------------------------------|---------------------------------|-------|
| ND ^a | 87 (46) | 23 (88) | 110 |
| 0.01-0.02 | 10 (5) | 2 (8) | 12 |
| 0.03-0.10 | 53 (28) | 1 (4) | 54 |
| 0.11-1.00 | 28 (15) | 0 (0) | 28 |
| 1.01-10.00 | 10 (5) | 0 (0) | 10 |
| Total | 188 | 26 | 214 |

^aND: not detected.

Table 2. The survey results of domestic soy sauce products with 3-MCPD contents not in compliance with the sanitary regulation (not > 1 ppm) set by the Department of Health

| Sample no. | 3-MCPD ^a (ppm) | Sample type | Sampling city |
|------------|---------------------------|----------------------|-----------------|
| 1 | 6.63 | Black bean sauce | Hsinchu county |
| 2 | 4.48 | Soy bean sauce | Nantou county |
| 3 | 4.64 | Soy bean sauce | Pingtung county |
| 4 | 3.68 | Soy bean sauce | Pingtung county |
| 5 | 2.82 | Soy bean sauce paste | Hsinchu city |
| 6 | 2.27 | Soy bean sauce | Taipei county |
| 7 | 1.73 | Black bean sauce | Hsinchu city |
| 8 | 1.45 | Soy bean sauce | Yunlin county |
| 9 | 1.37 | Soy bean sauce | Pingtung county |
| 10 | 1.25 | Black bean sauce | Chia-I county |

^aAccording to sanitary regulation set by Dept. of Health (2002), the 3-MCPD contents in soy sauce products shall not exceed 1 ppm.

Table 3. Comparisons of 3-MCPD contents within three kinds of soy sauce products during the fiscal year 2002

| Sample type | Total surveyed samples | No. of samples (%) | | |
|-----------------------------------|------------------------|----------------------|---------|--------------|
| | | > 1 ppm ^b | ≤ 1 ppm | undetectable |
| Soy bean sauce | 118 | 6 (5) | 47 (40) | 65 (55) |
| Soy bean sauce paste ^a | 90 | 4 (4) | 45 (50) | 41 (46) |
| Reduced salt sauce | 6 | 0 (0) | 2 (33) | 4 (67) |
| Total | 214 | 10 | 94 | 110 |

^aSoy bean sauce paste products including soy bean sauce paste, black bean sauce, and black bean sauce paste.

^bAccording to a sanitary regulation set by Dept. of Health (2002), the 3-MCPD contents in soy sauce products shall not exceed 1 ppm.

Table 4. The results of 3-MCPD contents in soy sauce products sampled by local hygiene bureaus in the fiscal year 2002

| Sampling district | Total surveyed samples | No. of samples (%) | | |
|-------------------|------------------------|----------------------|---------|--------------|
| | | > 1 ppm ^a | ≤ 1 ppm | undetectable |
| Keelung city | 2 | 0 (0) ^b | 2 (100) | 0 (0) |
| Taipei city | 31 | 0 (0) | 7 (23) | 24 (77) |
| Taipei county | 10 | 1 (10) | 3 (30) | 6 (60) |
| Taoyuan county | 2 | 0 (0) | 0 (0) | 2 (100) |
| Hsinchu city | 4 | 2 (50) | 2 (50) | 0 (0) |
| Hsinchu county | 3 | 1 (33) | 2 (67) | 0 (0) |
| Nantou county | 4 | 1 (25) | 1 (25) | 2 (50) |
| Taichung city | 4 | 0 (0) | 2 (50) | 2 (50) |
| Taichung county | 16 | 0 (0) | 14 (88) | 2 (12) |
| Changhua county | 20 | 0 (0) | 8 (40) | 12 (60) |
| Yunlin county | 16 | 1 (6) | 9 (56) | 6 (38) |
| Chia-I county | 10 | 1 (10) | 1 (10) | 8 (80) |
| Tainan city | 2 | 0 (0) | 2 (100) | 0 (0) |
| Tainan county | 34 | 0 (0) | 20 (59) | 14 (41) |
| Kaohsiung city | 4 | 0 (0) | 1 (25) | 3 (75) |
| Kaohsiung county | 4 | 0 (0) | 2 (50) | 2 (50) |
| Pingtung county | 47 | 3 (6) | 17 (36) | 27 (57) |
| Hualien county | 1 | 0 (0) | 1 (100) | 0 (0) |
| Total | 214 | 10 | 94 | 110 |

^aAccording to a sanitary regulation set by Dept. of Health (2002), the 3-MCPD contents in soy sauce products shall not exceed 1 ppm.

^bValues in the parenthesis are the percentage relative the number of samples.

Table 5. The 3-MCPD contents in soy sauce products surveyed in the fiscal year 2002 in comparison with the 3-MCPD maximum limits set by different countries

| Sample type | Total samples | No. of samples (%) ^a | | |
|-------------|---------------|---------------------------------|------------------------|-------------------------|
| | | > 0.2 ppm ^b | > 0.1 ppm ^c | > 0.02 ppm ^d |
| Domestic | 188 | 33 (18) | 38 (20) | 91 (48) |
| Imported | 26 | 0 (0) | 0 (0) | 1 (4) |
| Total | 214 | 33 (15) | 38 (18) | 92 (43) |

^a(%) = (no. of detected samples / no. of samples)100%.

^bThe allowable maximums of 3-MCPD in soy sauce in Australia and New Zealand is 0.2 ppm.

^cA sanitary regulation set by Dept. of Health (2002), the 3-MCPD contents in soy sauce products shall not exceed 1 ppm. By 2003, the upper limits will be further reduced to 0.1 ppm⁽¹⁶⁾.

^dThe allowable maximums of 3-MCPD in soy sauce in European Union, the Netherlands, Greece, Portugal, Malaysia, and Sweden is 0.02 ppm.

comparison with the 3-MCPD maximum limits set by different countries was shown in Table 5. The domestic soy sauce products had a higher proportion likely to be detected with 3-MCPD compared to imported soy sauce products in exceeding the maximum limit set by different countries. The proportion of 3-MCPD contents in domestic soy sauce products exceeding the government regulation⁽¹⁶⁾ and European Union maximum limits were 20% and 48% separately. Domestic soy sauce producers still have to closely monitor the 3-MCPD content during production process.

III. Comparison of the Survey Results in the Fiscal Year of 2000 and 2002

The comparison of 3-MCPD contents of domestic soy sauce products in the fiscal year 2000 and 2002 is shown in Figure 1. Fourteen samples of soy sauce products were detected by the GC/MS method in the fiscal year 2000.

Twenty nine percent of the soy sauce products were detected to contain 3-MCPD in the range of 1.01 to 10 ppm and 29% products were found to contain over 10 ppm. Fifty eight percent of soy sauce products surveyed in 2000 were over the 1 ppm maximum limit set by the government in fiscal year 2002. But only 5% of soy sauce products were detected to contain 3-MCPD contents over the maximum limit. Most of the soy sauce products, i.e. 95%, surveyed in 2002 contained 3-MCPD lower than 1 ppm. The 3-MCPD contents of domestic soy sauce products in 2002 are decreasing significantly in comparison with the surveyed contents in 2000.

IV. Comparison of the Survey in Taiwan and UK

We analyzed the data obtained in fiscal year 2002 according to the distributions of 3-MCPD content surveyed in UK⁽²⁾ (Figure 2). The results showed that the proportion of the undetectable sample for the 3-MCPD analysis were

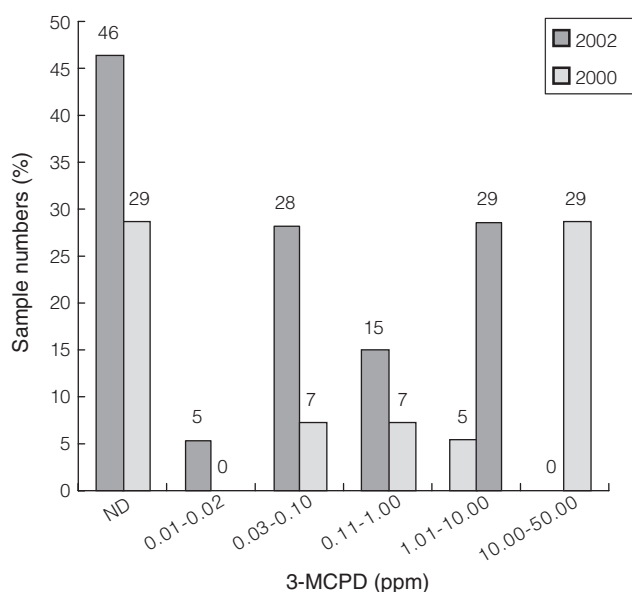


Figure 1. Comparisons of 3-MCPD contents of domestic soy sauce products in the fiscal year 2000 and 2002.

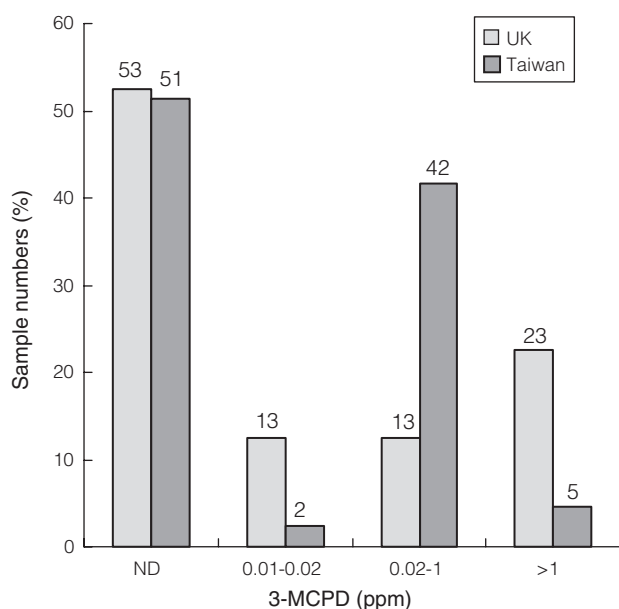


Figure 2. Comparisons of 3-MCPD contents of soy sauce products surveyed in Taiwan (2002) and UK.

53% and 51% in UK and Taiwan, respectively. In Taiwan only 5% of soy sauce products contained more than 1 ppm 3-MCPD, while in the UK there were 23% of soy sauce products that contained more than 1 ppm 3-MCPD. The majority (42%) of the soy sauce products in Taiwan were detected to contain 3-MCPD in the range of 0.02 to 1 ppm.

V. Improvement by Manufactures

Ten domestic soy sauce products where 3-MCPD content violated the sanitary regulation set by government

Table 6. The secondary survey results of domestic soy sauce products with 3-MCPD contents not in compliance with the sanitary regulation (not > 1 ppm) set by the Department of Health

| Sample no. | 3-MCPD (ppm) |
|------------|--------------------|
| 1 | ND. |
| 2 | 0.03 |
| 3 | N.D. |
| 4 | 0.02 |
| 5 | 0.05 |
| 6 | 0.06 |
| 7 | 0.11 |
| 8 | N.D. |
| 9 | N.D. |
| 10 | No longer produced |

were sampled and reanalyzed again after improvement by the manufactures. 3-MCPD concentrations ranged from 0.02 ppm to 0.11 ppm in 5 samples and were undetectable in 4 samples, while 1 sample was no longer produced after the survey (Table 6).

CONCLUSIONS

The results of the survey showed that 3-MCPD was detected in several kinds, brands, and grades of soy sauce products. 3-MCPD contents in imported soy sauce products were not found to be in violation of the sanitary regulation set by the government in fiscal year 2002 but 10 samples of domestic soy sauce products contained amounts greater than 1 ppm. Although the 3-MCPD contents of domestic soy sauce products were lower than before, there is still room for the producer to improve the quality of soy sauce products.

ACKNOWLEDGMENTS

This research was supported by Department of Health, Taiwan, R.O.C. (project no. DOH 91-FD-2046)

REFERENCES

1. Van Bergen, C. A., Collier, P. D., Cromie, D. D. O., Lucas, R. A., Preston, H. D. and Sissons, D. J. 1992. Determination of chloropropanols in protein hydrolysates. *J. Chromatogr.* 589: 109-119.
2. Macarthur, R., Crews, C., Davies, A., Brereton, P., Hough, P. and Harvey, D. 2000. 3-Monochloropropane-1,2-diol (3-MCPD) in soy sauces and similar products available from retail outlets in the UK. *Food Addit. Contam.* 17: 903-906.
3. Crews, C., Hough, P., Brereton, P., Harvey, D., Macarthur, R. and Matthews, W. 2002. Survey of 3-monochloropropane-1,2-diol (3-MCPD) in selected food groups, 1999-2000. *Food Addit. Contam.* 19: 22-27.

4. Crews, C., Brereton, P. and Davies, A. 2001. The effects of domestic cooking on the levels of 3-monochloropropanediol in foods. *Food Addit. Contam.* 18: 271-280.
5. Food Standards Agency. 2001. Some soy sauce products to be removed. <http://www.foodstandards.gov.uk/news/pressreleases/soysaucerecall>
6. Lynch, B. S., Bryant, D. W., Hook, G. J., Nestmann, E. R. and Munro, I. C. 1998. Carcinogenicity of monochloro-1,2-propanediol (α -chlorohydrin, 3-MCPD). *Int. J. Toxicol.* 17: 47-76.
7. European Commission. 2001. Opinion of the scientific committee on food on 3-monochloro-propane-1,2-diol (3-MCPD) updating the SCF opinion of 1994 (adopted on 30 May 2001), SCF/CS/CNTM/OTH/17 Final.
8. Food Standards Agency. 2001. Survey of 3-monochloropropane-1,2-diol (3-MCPD) in soy sauce and related products. Food Surveillance Information Sheet, No. 14/01. <http://www.foodstandards.gov.uk/science/surveillance/fsis-2001/3-mcpdsoy>
9. Committee on Carcinogenicity of Chemicals in Food, Consumer Products and the Environment. 2000. Carcinogenicity of 3-monochloro propane 1,2-diol (3-MCPD). <http://www.doh.gov.uk/mcpd2.htm>.
10. Committee on Mutagenicity of Chemicals in Food, Consumer Products and the Environment. 2000. Mutagenicity of 3-monochloro propane 1,2-diol (3-MCPD). <http://www.doh.gov.uk/mcpd1.htm>.
11. Pesselman, R. L. and Feit, M. J. 1988. Determination of residual epichlorohydrin and 3-chloropropanediol in water by gas chromatography with electron-capture detection. *J. Chromatogr.* 439: 448-452.
12. Meierhans, D. C. Bruehlmann, S., Meili, J. and Taeshler, C. 1998. Sensitive method for the determination of 3-chloropropane-1,2-diol and 2-chloropropane-1,3-diol by capillary gas chromatography with mass spectrometric detection. *J. Chromatogr. A* 802: 325-333.
13. Hamlet, C. G. 1998. Analytical methods for the determination of 3-chloro-1,2-propanediol and 2-chloro-1,3-propanediol in hydrolyzed vegetable protein, seasonings and food products using gas chromatography ion trap tandem mass spectrometry. *Food Addit. Contam.* 15: 451-465.
14. DOH Food No. 0900091913 Announced, 11/12/2001. Method of test for 3-monochloro-1,2-propanediol in Food (I).
15. DOH Food No. 0900056973 Announced, 9/7/2001. Limit of 3-monochloro-1,2-propanediol in soy sauce: 1 ppm.
16. DOH Food No. 0900046807 Announced, 7/12/2001. Draft of the limit of 3-monochloro-1,2-propanediol in soy sauce.

五種轉殖品項基因改造玉米 (Bt11、Event176、GA21、MON810及T25) 即時定量PCR檢驗方法之建立

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摘 要

為配合基因改造食品標示制度之施行，本研究著重於即時定量聚合酶鏈反應 (real-time quantitative polymerase chain reaction, real-time QPCR) 方法之開發，以定量檢測基因改造玉米。針對五種轉殖品項Event176及Bt11 (Syngenta公司)、MON810及GA21 (Monsanto公司)及T25 (Aventis公司)之植入基因設計產品特異性引子及探針，並進行real-time QPCR測試。同時，針對每種轉殖品項基因改造玉米構築一含轉殖基因及內部對照基因標之質體，以作為定量參考分子，並進一步使用構築質體及測試檢體測試，以評估本定量系統。結果顯示，以構築之質體經由系列稀釋所製作之標準曲線，其獲得之slope值介於-3.31至-3.45，correlation介於0.99至1.0。以本研究發展之方法定量測試含1%、2%或5%基因改造玉米之測試檢體，1%測試檢體之平均值 (mean) 介於1.01%至1.23%，2%測試檢體介於2.00%至2.31%，5%測試檢體介於4.46%至5.55%，標準偏差 (standard deviation, S.D.) 介於0.05至0.46，變異係數 (coefficient of variance, C.V.) 介於5%至15%。本方法之定量極限 (limits of quantitation, LOQ) 為0.1% (w/w)。除此，以本研究發展之方法測試精準度試驗檢體，可獲得優異結果。

關鍵詞：聚合酶鏈反應，基因改造玉米

五種台灣非基因改造及進口基因改造大豆中異黃酮 含量之探討

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摘 要

本研究主要比較高雄10號大豆、台南1號大豆、台南4號大豆、台南5號黑豆及市售黑豆等5種台灣非基因改造大豆及進口基因改造大豆之異黃酮總含量及其異構物分佈之差異，並進一步了解大豆分別經浸泡0、4、8、12及16小時水溶液，及於pH 3、7及9溶液中浸泡8小時後，其異黃酮的變化情形。異黃酮的測定乃利用高效液相層析法分析含葡萄糖苷之daidzin、glycitin、genistin、acetyldaizin、acetylglycitin、acetylgenistin及不含葡萄糖苷之daidzein、glycitein及genistein等共9種異黃酮分布情形。分析條件以RP-18逆相管柱為固定相，移動相(A)為氬甲烷及(B)為0.1%三氟醋酸混合溶液。由測定結果顯示台灣非基因改造大豆中以高雄10號黃豆之異黃酮總含量最高約346 mg/100 g，台南5號黑豆異黃酮總含量最低。所分析的大豆均以含葡萄糖苷較不含葡萄糖苷之異黃酮含量高。高雄10號、台南黑豆5號及基因改造大豆於浸泡4小時後不含葡萄糖苷之daidzein及genistein異黃酮含量有顯著增加 ($P < 0.05$)。另外，高雄10號大豆、台南5號黑豆及基因改造大豆浸泡於pH 9溶液下之異黃酮總含量明顯低於pH 3及pH 7的趨勢；以高雄10號大豆浸泡於pH 3溶液daidzein及genistein含量最高，台南5號黑豆其daidzein及genistein於pH 9溶液下含量最低。

關鍵詞：非基因改造大豆，基因改造大豆，異黃酮

以線上微萃取毛細管電泳法分析醬油中之 苯甲酸及其衍生物

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摘 要

本研究運用毛細管電泳配合管內微萃取技術作市售醬油之苯甲酸類防腐劑之檢測。樣品先經酸處理，再加入乙酸乙酯萃取使其分層後，取其上層液進行毛細管電泳。電泳條件為：進樣量30 mbar × 1 sec、75 μm i.d. × 34 cm之未修飾熔融石英毛細管、20 mM 硼酸緩衝液 (pH 9.2)、工作電壓10 kV、檢測波長A₂₂₅。在10分鐘內可完成防腐劑之分離檢測，且以馬尿酸為內標準時可有效地改善再現性 (CV = 4.5%, n = 12)。

關鍵詞：對羧苯甲酸，乙酸乙酯，毛細管區段電泳，馬尿酸，防腐劑

醬油中3-單氯丙二醇含量之調查

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摘 要

為了解市售醬油中3-monochloro-1,2-propanediol (3-MCPD) 含量情形，2002年由衛生署委託基隆市等18個縣市政府衛生局協助至該轄區之醬油工廠或零售店進行採樣，共計採得檢體214件，包括國產醬油188件，進口醬油26件。採得之檢體送至本局檢驗，以衛生署公告方法進行檢測，該方法以GC/MS為分析儀器，方法最低檢出限量為0.01 ppm。檢驗結果87件國產醬油未檢出，23件進口醬油未檢出；101件國產醬油檢出3-MCPD，91件國產醬油含量範圍為0.01 ppm至1 ppm；另外有10件國產醬油3-MCPD含量範圍為1.25 ppm至6.63 ppm，超過現行衛生標準1 ppm。進口醬油中僅有3件檢出3-MCPD，含量範圍0.01 ppm至0.03 ppm。分析比較2000年與2002年之結果顯示後者醬油中3-MCPD的含量有顯著的下降。不符規定檢體衛生署已通知改善並送本局複驗，結果均與規定相符。

關鍵詞：醬油，單氯丙二醇，氣相層析質譜儀