

## Chapter 4

### RESULTS

**4.1 Physico-chemical study of methamphetamine pills** All methamphetamine pills studied were discoid in shape. From physico-chemical investigation during October 2002-March 2003, the average dimension of pills examined was  $6.05 \pm 0.06$  mm in diameter,  $3.02 \pm 0.20$  mm in center thickness,  $2.27 \pm 0.18$  mm in rim thickness. The average weight was  $0.093 \pm 0.005$  gm; the hardness was found to be  $9.2 \pm 3.8$  kg. The various methamphetamine logos in samples collected are illustrated in Figure 3. Table 1 presents physical properties of methamphetamine pills arranged by logo. Regarding pill colour, various colours and shades were observed. Seven colours were found--orange, yellow, green, brown, pink, red and purple --in 8, 4, 3, 3, 3, 1 and 1 shades, respectively. Orange (86.6 %) predominated followed by green (7.4%), brown (2.1%), red (2.0%), yellow (1.0%), pink (0.8%) and purple (0.1%). Three shades of orange found in all provinces, unlike all other colours found. Of logos, wY (38.7%) predominated followed by WY (37.4%), wy (20.6%), R (1.2 %), Wy (0.7%), OK (0.6%), 888 (0.4%), and w/99 (0.4%). Table 2 illustrates chemical properties of methamphetamine pills arranged by logo. Average methamphetamine and caffeine content were found to be  $22.79 \pm 8.12$  mg % and  $61.87 \pm 13.81$  mg %, respectively. Two samples were found to also contain amphetamine 1-3 mg %, 22 samples contained ephedrine 1-4 mg %, 4 samples contained theophylline 0.014 mg % and 1 sample contained phenacetin (undetermined amount).

From physico-chemical study during October 2003-May 2004. Two hundred and ninety-eight samples were comprised of pills displaying 7 kinds of logos: wY ( 70.1%), wy (15.8%), WY (5.1%), R (4.5%), Wy (4.0%), OK (0.3%) and 888 (0.2%). The samples with OK and 888 logo were rejected from the study because of a small number of samples. Regarding pill colour, various colours and shades were observed. Five colours were found--orange, yellow, purple, red and green--in 8, 3, 2, 3 and 1 shades, respectively. Orange (92.0 %) predominated followed by green (3.7%), purple (1.7%), yellow (1.3%) and red (1.3%). Three shades of orange also found in all provinces studied. The average physical properties are shown in Table 3. Methamphetamine and caffeine were found to be the main ingredients in



Figure 3 The various methamphetamine logos in samples collected.

methamphetamine pills. The average content of methamphetamine and caffeine is displayed in Table 4. From data analysis using ANOVA, it was found that diameters of methamphetamine pills were significantly different ( $p < 0.05$ ) across logos (Table 5, 6), and wY logo and wy logo showed significant differences in diameter using Tukey's honestly significant test ( $p < 0.05$ ).

**4.2 The study of disintegration and dissolution profiles** All methamphetamine pill samples included in this study displayed the WY logo. Three variants of the WY logos were found: wY, WY and wy. Most of these samples are orange, with one green sample being the sole exception. The appearance of methamphetamine pills was observed to be as follows. Most pills had smooth surfaces and a non-uniform colouring to the pill surface; only one sample had a shiny surface. The disintegration profile of the methamphetamine pill was observed to a slow dissolution, without fragmentation. Graphic showing disintegration patterns of methamphetamine pills are presented in figures 4 and 5. The disintegration times are shown in Table 7. The average disintegration time was 9 minutes 55 seconds. Disintegration times for different logos varied significantly ( $p < 0.05$ ). Dissolution profiling showed that after 10 minutes methamphetamine from samples with the wY logo had been completely released after 10 minutes whereas only after 30 and 45 minutes respectively for wy

Table 1 Physical properties of methamphetamine pills seized during October 2002-February 2003 classified by logos

Logos (n)	Diameter (mm)		Center thickness (mm)		Rim thickness (mm)		Weight of pill (g)			Hardness (kg)	
	X ± SD	%CV	X ± SD	%CV	X ± SD	%CV	X ± SD	%CV	X ± SD	%CV	
WY (256)	6.05 ± 0.06	0.10	3.08 ± 0.20	6.49	2.29 ± 0.20	8.73	0.0938 ± 0.0056	6.38	9 ± 4	44.44	
Wy (5)	6.03 ± 0.10	1.66	2.99 ± 0.07	2.34	2.22 ± 0.03	1.35	0.0900 ± 0.0014	1.11	12. ± 5	41.67	
wY (265)	6.04 ± 0.06	0.99	2.97 ± 0.02	0.61	2.26 ± 0.17	7.52	0.0914 ± 0.0053	5.50	10 ± 3	30.00	
wy (141)	6.06 ± 0.07	1.16	3.03 ± 0.19	6.27	2.27 ± 0.19	8.37	0.0926 ± 0.0048	5.38	9 ± 4	44.44	
OK (4)	6.16 ± 0.05	0.81	2.98 ± 0.12	4.03	2.28 ± 0.06	2.63	0.0973 ± 0.0023	2.06	7 ± 3	42.86	
w/99 (3)	6.05 ± 0.01	0.17	2.98 ± 0.13	0.44	2.31 ± 0.05	2.16	0.0870 ± 0.0011	1.15	12 ± 1	8.33	
888 (3)	6.09 ± 0.04	0.66	3.10 ± 0.15	0.87	2.42 ± 0.09	3.72	0.0940 ± 0.0040	4.04	12 ± 2	16.66	
R (8)	6.05 ± 0.05	0.83	3.17 ± 0.57	17.98	2.40 ± 0.16	6.66	0.0945 ± 0.0069	7.37	6 ± 5	83.33	
Total (685)	6.05 ± 0.06	0.99	3.02 ± 0.20	6.62	2.27 ± 0.18	7.93	0.0926 ± 0.0054	5.38	9 ± 3	41.30	

Table 2 Chemical properties of methamphetamine pills seized during October 2002-February 2003 classified by logos

Logos (n)	Methamphetamine (mg %)		caffeine (mg%)	
	X ± SD	%CV	X ± SD	%CV
WY (256)	23.06 ± 8.27	35.86	61.42 ± 13.06	21.26
Wy (5)	23.92 ± 4.30	17.98	69.86 ± 17.76	25.42
wY (265)	22.96 ± 8.09	35.24	63.37 ± 10.59	16.71
wy (141)	21.09 ± 2.83	13.42	59.89 ± 12.91	21.56
OK (4)	16.10 ± 0.53	3.29	66.42 ± 1.37	2.06
w/99 (3)	11.49 ± 0.50	4.352	66.66 ± 0.75	1.12
888 (3)	31.87 ± 0.65	2.039	60.63 ± 0.92	1.51
R (8)	20.39 ± 11.20	54.92	57.92 ± 15.36	26.52
Total (685)	22.79 ± 8.12	35.63	61.87 ± 13.81	22.32

Table 3 Physical properties of methamphetamine pills seized during October 2003-May 2004 classified by logos

Logos (n)	Diameter (mm)		Center thickness (mm)		Rim thickness (mm)		Weight of pill (gm)		Hardness (kg)	
	X ± SD	%CV	X ± SD	%CV	X ± SD	%CV	X ± SD	%CV	X ± SD	%CV
WY (15)	6.03 ± 0.01	0.12	3.03 ± 0.13	4.42	2.17 ± 0.14	6.45	0.0923 ± 0.0046	4.98	6 ± 2	33.33
Wy (12)	6.05 ± 0.01	0.12	2.99 ± 0.01	0.23	2.15 ± 0.08	3.72	0.0889 ± 0.0024	2.70	5 ± 1	20.00
wY (209)	6.01 ± 0.00	0.07	3.03 ± 0.14	4.46	2.21 ± 0.10	4.52	0.0910 ± 0.0071	7.80	7 ± 3	42.86
wy (47)	6.05 ± 0.01	0.08	3.02 ± 0.15	4.91	2.21 ± 0.15	6.79	0.0931 ± 0.0057	6.12	7 ± 2	28.57
R (13)	6.01 ± 0.00	0.02	3.07 ± 0.10	3.38	2.27 ± 0.33	14.5	0.0887 ± 0.0024	2.71	5 ± 1	20.00
Total (296)	6.02 ± 0.00	0.07	3.03 ± 0.14	0.45	2.21 ± 0.11	4.98	0.0914 ± 0.0007	7.66	6.8 ± 3.2	47.06

Note: The samples with OK and 888 logo were rejected from the study because of a small number of samples.

Table 4 Chemical properties of methamphetamine pills seized during October 2003-May 2004 classified by logos

Logos (n)	Methamphetamine (mg %)		caffeine (mg%)	
	X ± SD	%CV	X ± SD	%CV
WY (15)	20.62 ± 7.61	36.91	62.43 ± 11.11	17.80
Wy (12)	17.70 ± 1.35	7.63	70.87 ± 4.22	5.95
wY (209)	22.00 ± 6.36	28.91	62.15 ± 9.36	15.06
wy (47)	21.16 ± 6.43	30.39	62.98 ± 7.33	11.39
R (13)	19.54 ± 3.33	17.04	69.91 ± 1.57	2.25
Total (286)	21.76 ± 6.39	29.37	62.43 ± 9.15	14.66

Table 5 ANOVA analysis of the physical and chemical properties of the methamphetamine pills studied (\*  $p < 0.05$ )

Source of variation	F	P-value
Diameter	7.998 *	0.000
Thickness	0.219	0.928
Weight	1.075	0.369
%Methamphetamine content	0.590	0.670
% Caffeine content	1.018	0.399

Table 6 ANOVA analysis of the study on diameter of the methamphetamine pills studied (\*  $p < 0.05$ )

Source of variation		Sum of Squares	df	Mean Squares	F	P-value
Diameter	Between groups	6.869E-02	4	1.717E-02	7.998	.000
	Within groups	.616	287	2.147E-03		
	Total	.685	291			






Figure 4 The picture of methamphetamine pill in a basket rack assembly supporting cylinder glass tube of disintegration apparatus before operation.



Figure 5 The picture of methamphetamine pill after 5 minute of operation

Table 7 The disintegration time of methamphetamine pills classified by logos. The disintegration time of each logo showed to be significantly different ( $p < 0.05$ ).  $n = 14, 15$  and  $9$  for wY logo, WY logo and wy logo, respectively.

Logo (n)	Disintegration time $\pm$ SD (second)	
	average	%CV
wY  (14)	401.49 $\pm$ 69.91	17.41
wy  (15)	514.72 $\pm$ 59.74	11.61
WY  (9)	824.77 $\pm$ 128.08	15.52
Total (38)	595.39 $\pm$ 213.87	35.93

logo and WY logo samples was methamphetamine observed to be completely released (Figure 6). Dissolution profiling data is shown in Table 8. The dissolution profiles of methamphetamine pills with wY, wy and WY were significantly difference ( $p < 0.05$ ).

#### **4.3 For determining food dyes in methamphetamine pills.**

**4.3.1 Chromatographic system suitability** The peaks of sunset yellow FCF, tartrazine and ponceau 4R were identified with the retention times at 5.5, 7.3 and 9.7 minutes, respectively. The tailing factors were 1.0, 1.2 and 1.4, respectively. The theoretical plates were 7872, 5595 and 5955, respectively.

**4.3.2 Linearity of the system** The linearity of the peak area vs. concentration ( $\mu\text{g/ml}$ ) curve for each of standard dyes was investigated in the range 100-500  $\mu\text{g/ml}$ . The regression lines were shown in figure 7. Results of the regression analysis and the correlation coefficients ( $r$ ) were as follows:  $y = 21.372x + 102.8$  ( $r = 0.9999$ );  $y = 54.705x + 189.21$  ( $r = 0.9999$ ) and  $y = 30.84x + 52.191$  ( $r = 0.9999$ ) for sunset yellow FCF, tartrazine and ponceau 4R, respectively.

**4.3.3 Precision** The reproducibility (relative standard deviation) of the proposed method, on the basis of peak-area ratios, were 0.14, 1.33 and 0.23 for sunset yellow FCF, tartrazine and ponceau 4R, respectively

**4.3.4 Accuracy** The results of a standard addition recovery study for standard dyes are summarized in Table 9. The recoveries were 96.2 – 106.9%.

#### **4.3.5 Determination of dyes in methamphetamine pills.**

For the methamphetamine pills studied, dyed in three popular orange shades, the coloured surface of these methamphetamine pills was found to have a mottled appearance. When dyes in methamphetamine pills of orange colour were analyzed by HPLC under the stated conditions, it was found that all samples contained food soluble dyes. The HPLC spectrum of dyes in orange pills is shown in figure 8. Sunset yellow FCF, tartrazine and ponceau 4R were found in all samples. The types and amounts of dyes are shown in Table 10.

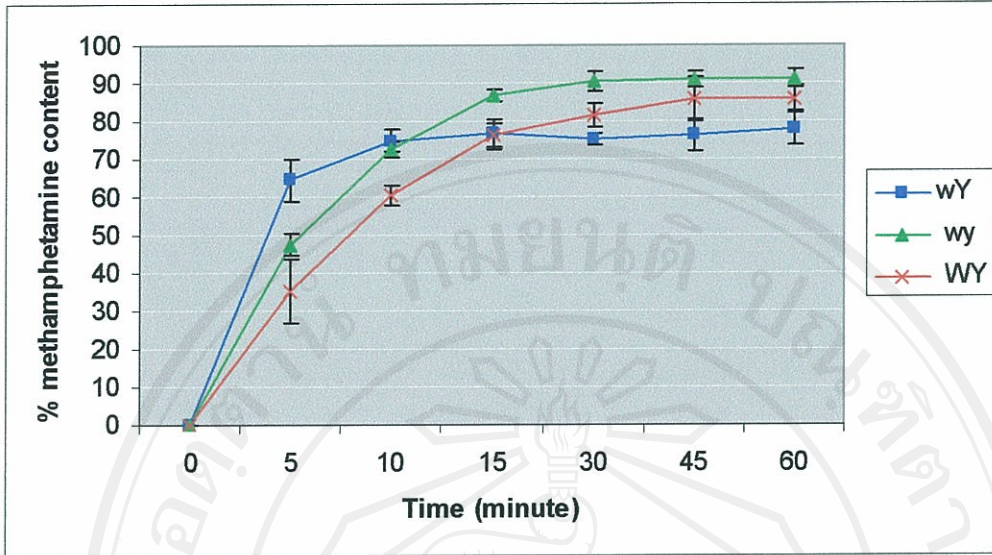


Figure 6 % methamphetamine content which was dissolved from methamphetamine pills with wY, wy and WY logo after the time interval. Data are presented as mean  $\pm$  SD

Table 8 The dissolution time of methamphetamine pills at the time interval (n = 5)

Logo (n)	methamphetamine content (%)						
	0 min	5 min	10 min	15 min	30 min	45 min	60 min
wY (5)	0	64.5 $\pm$ 5.3	74.9 $\pm$ 2.8	76.6 $\pm$ 3.8	75.5 $\pm$ 1.6	76.4 $\pm$ 4.1	75.9 $\pm$ 4.2
Wy (5)	0	47.6 $\pm$ 2.9	72.8 $\pm$ 2.2	86.9 $\pm$ 1.7	90.7 $\pm$ 2.7	91.2 $\pm$ 2.0	91.05 $\pm$ 2.4
WY (5)	0	35.3 $\pm$ 8.4	60.6 $\pm$ 2.8	76.1 $\pm$ 3.3	81.8 $\pm$ 3.2	84.5 $\pm$ 5.8	84.5 $\pm$ 3.4

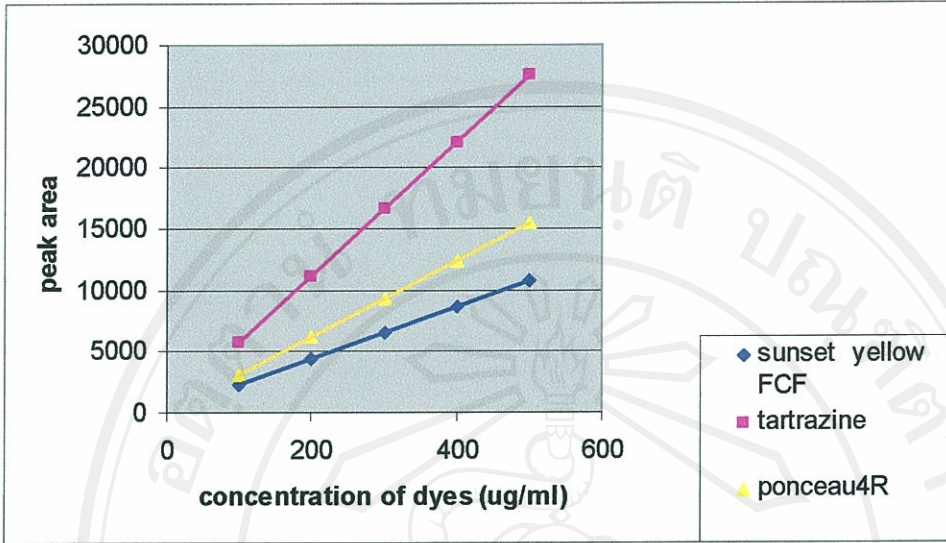


Figure 7 Regression lines illustrate the relationship between the concentration of standard sunset yellow FCF, tartrazine and ponceau 4R and peak areas

Sunset yellow;  $y = 21.372x + 102.8$  ( $r = 0.9999$ )

Tartrazine;  $y = 54.705x + 189.21$  ( $r = 0.9999$ )

Ponceau 4R;  $y = 30.84 x + 52.191$  ( $r = 0.9999$ )

Table 9 Data for recoveries of dyes in methamphetamine pills (n = 5)

Added dyes (ug/ml)	Recovery (%)		
	Sunset yellow FCF	Tartrazine	Ponceau 4R
100	105.5 ± 2.4	106.3 ± 6.2	98.3 ± 5.7
200	105.7 ± 3.2	105.2 ± 3.7	96.2 ± 4.1
300	104.6 ± 4.6	105.2 ± 2.8	101.3 ± 3.3
400	106.9 ± 5.3	97.2 ± 5.5	103.0 ± 2.0
500	106.2 ± 3.2	97.0 ± 4.7	101.9 ± 3.3

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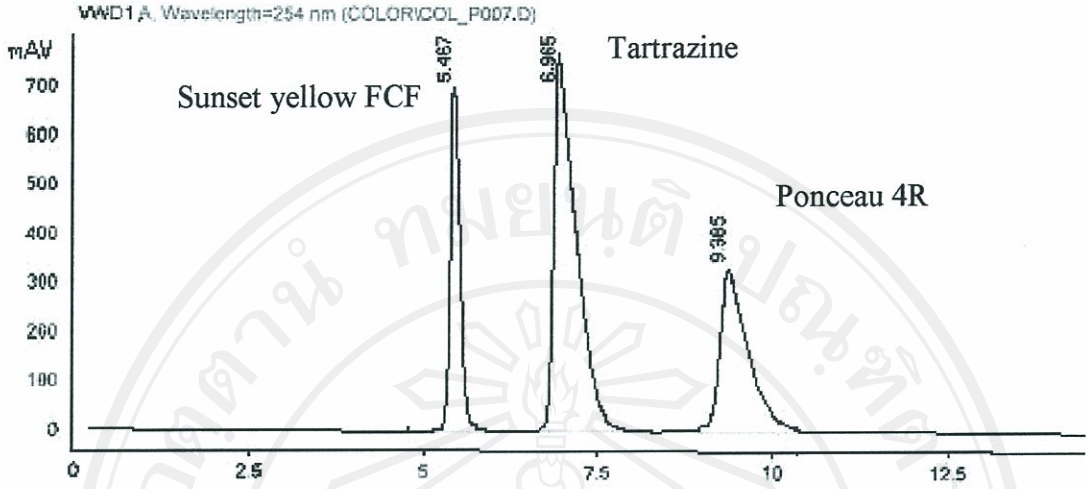





Figure 8 Chromatogram of dyes

Table 10 Contents (ppm) of dyes in methamphetamine pills for orange colour (n = 5)

Orange coloured methamphetamine pills	Sunset yellow (ppm)	Ponceau 4R (ppm)	Tartrazine (ppm)
1 	1961.9 ± 9.5	1752.5 ± 41.1	472.5 ± 9.7
2 	4303.1 ± 25.2	1239.2 ± 36.7	35.4 ± 5.24
3 	4709.0 ± 41.6	890.4 ± 44.7	292.6 ± 10.8

**4.4 Trafficking routes and distribution patterns of methamphetamine pills** The pictures of trafficking routes and distribution patterns of methamphetamine were drawn on the basis of identified physical and chemical properties of the samples. Among key findings are these: 29 samples having identical physical properties were seized in two different districts. In addition, 1 set of samples having identical physical and chemical properties was found in two different districts, Chiang Dao district in Chiang Mai province and Maesai district in Chiang Rai province (Figure 9). It was also found that the dominant logo in Chiang Mai province and Tak province was wY whereas WY predominated in other provinces including Chiang Rai. The relationship of physico-chemical properties of methamphetamine pill samples and trafficking routes from the main smuggling areas (Chiangmai province, Chiangrai province and Tak province) was studied using predictive Discriminant analysis. The predictive equations were obtained in a process in which 6 variables were included: % caffeine, % methamphetamine, diameter, weight of pill, logo wY and logo WY. It was assumed that there were three main smuggling areas: Chiangmai, Chiangrai, Tak. The predicted equations were obtained in relation to 6 variables: logo WY, logo wY, diameter, weight, % methamphetamine, % caffeine. The variables in the equations will be substituted for with the data obtained from studied pills. For the equations which give the highest Y value, it can be predicted that pill comes from that province. The reliability of the test was found to be 76.5%. For Chiangmai, Chiangrai and Tak provinces, the result were reliable at 72.6, 66.7 and 93.8%, respectively.

Predicted equation from Discrimination analysis:

$$1. \text{ Chiangmai } Y = -5078.494 + 45.2 (WY) + 69.364 (wY) + 1647.156 (\text{diameter}) + 458.464 (\text{weight of pill}) + 2.484 (\%Meth) + 1.063 (\%Caff).$$

$$2. \text{ Chiangrai } Y = -5095.072 + 46.459 (WY) + 68.140 (wY) + 1648.269 (\text{diameter}) + 552.833 (\text{weight of pill}) + 2.428 (\%Meth) + 1.093 (\%Caff).$$

$$3. \text{ Tak } Y = -5210.774 + 46.471 (WY) + 71.436 (wY) + 1665.788 (\text{diameter}) + 488.693 (\text{weight of pill}) + 2.678 (\%Meth) + 1.216 (\%Caff).$$

GIS is an information system which is designed to work with data referenced by geographic coordinate. The physico-chemical characteristic of samples will be linked with the place where samples were seized so the distribution picture of samples can be drawn as shown in figure 10.

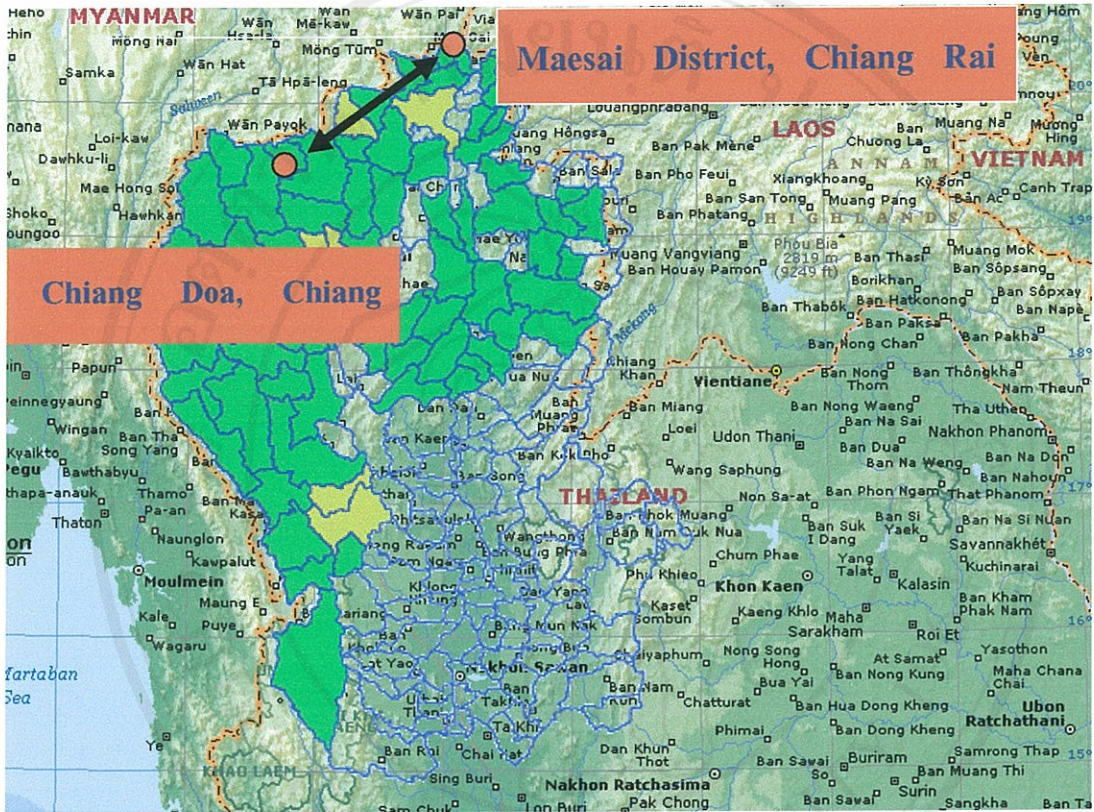


Figure 9 A picture showed one set of samples having identical physical and chemical properties was found in two different districts.

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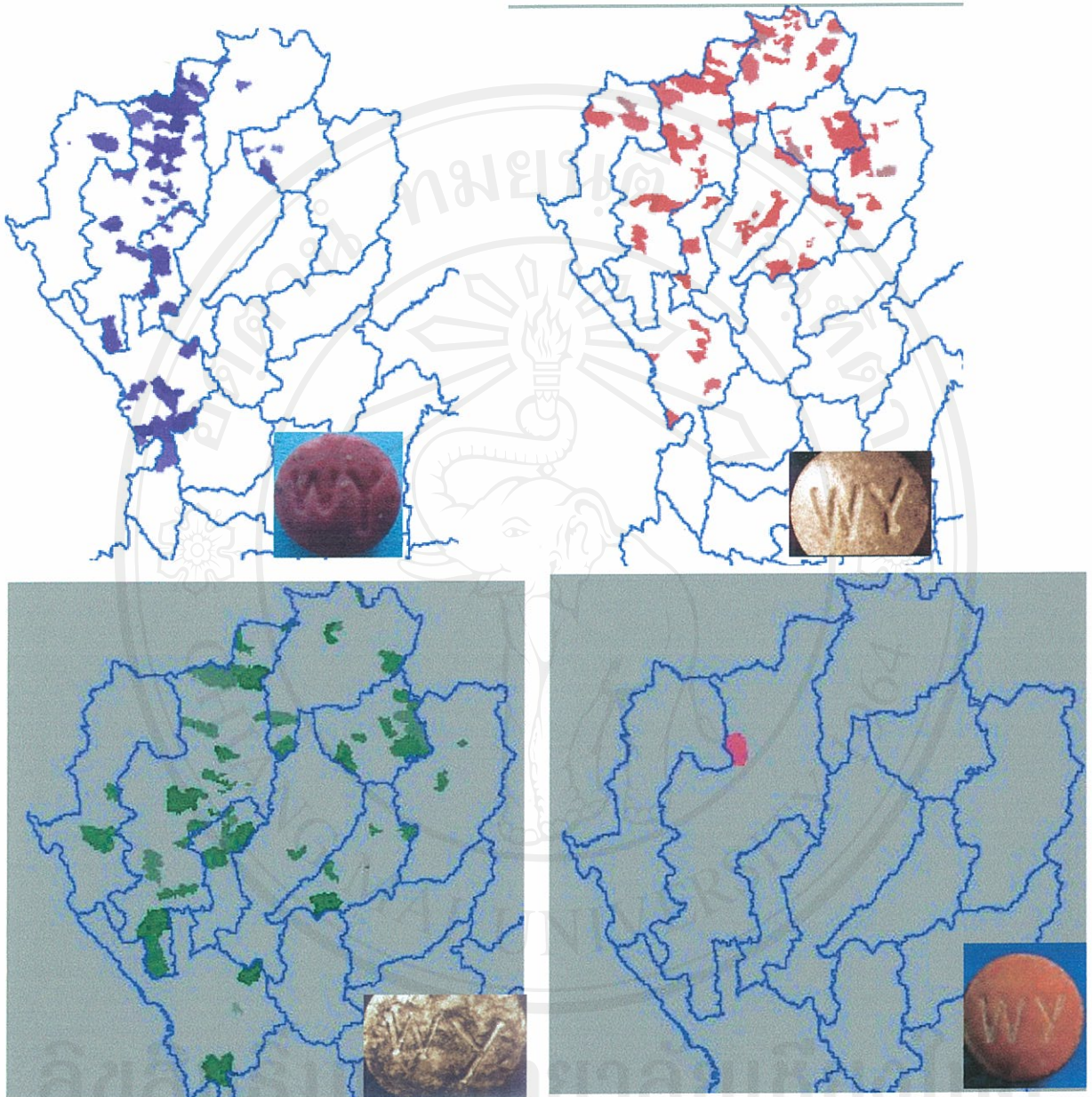


Figure 10 Distribution pictures of methamphetamine pills using GIS to draw the picture classified by logos.