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Constituents of *Cannabis sativa* L. XXIV: The Potency of Confiscated Marijuana, Hashish, and Hash Oil Over a Ten-Year Period

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ABSTRACT: The chemical analysis of 2169 *Cannabis* preparations confiscated in the United States over a ten-year period is discussed. Samples are categorized according to physical appearance and potency trends are noted. The appearance of sinsemilla and buds, more potent forms of marijuana, and their effects on overall potency are emphasized along with discussion on domestically grown marijuana.

KEYWORDS: toxicology, marijuana, illegal drug sales

Cannabis and its preparations (marijuana, sinsemilla, buds, hashish, hash oil, and so forth) are the most widely used group of illicit drugs in the world. Efforts have been concentrated in the last decade towards evaluating the health problems associated with *Cannabis* use, with conflicting results [*1,2*]. Varying biological effects of *Cannabis* [*3,4*] are attributed to the complex chemical composition of the plant material [*5*]. Because *Cannabis* is an illicit drug, it is only available to the general public through illegal channels. Consequently, the chemical analysis of confiscated material becomes important in understanding the health problems to the public associated with the use of any form of the drug.

Review of the literature dealing with confiscated marijuana and other *Cannabis* preparations revealed that the analyses were essentially carried out for the identification of the drug for legal or forensic science purposes [*6-9*]. In some instances, the analysis was conducted along with physical description in attempts to identify the country of origin [*10-13*].

In mid 1975, the National Institute on Drug Abuse and the Department of Justice's Drug Enforcement Administration (DEA) established a collaborative agreement whereby our laboratory would receive for analysis samples of all DEA seizures of hashish and hash oil and of marijuana seizures over 90 kg (200 lb). This program was called the Potency Monitoring Pro-

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gram. Efforts were also made to acquire samples from other sources such as physicians, local and state law enforcement agencies, and the U.S. Customs Service. The program was designed to determine the current potency of illicit marijuana samples and the total cannabinoid profile of such samples. The analytical data generated were to be used to show trends in increasing or decreasing potency, to help identify the country of origin whenever possible, and to provide information for policymaking decisions at the national and possibly international levels. In addition, the analytical data were to provide information to the scientific community in studying health problems associated with *Cannabis* use.

This manuscript summarizes the analytical data generated by this program.

Experimental Procedure

Samples

All samples analyzed in this investigation were confiscated during the years 1972 through 1981 by law enforcement agencies in the United States including the Drug Enforcement Administration (DEA) and, previously, the Bureau of Narcotics and Dangerous Drugs (BNDD), state and local police, as well as the United States Customs Service.

Samples were classified as PM, PS, PD, ST, or FG depending on the source from which they were obtained according to the following key:

PM—Potency Monitoring: designates those samples received through the DEA or its predecessor BNDD.
PS —Psychiatric: received through a psychiatrist or other medical doctor from a patient having psychiatric or medical problems related to marijuana use.
PD —Police Department: designates those samples received from police departments or sheriffs' offices.
ST —State Crime Laboratories: designates those samples received from state crime labs or other state agencies.
FG —Fugitive: designates samples received when no arrests were made.

In addition, samples were classified according to their physical characteristics into the following categories:

Marijuana—
1. MH (Marijuana): when samples are received in the form of loose *Cannabis* plant material with leaves, stems and seeds.
2. KB (Kilobricks): *Cannabis* compressed in the form of a kilobrick (classical Mexican packaging) with leaves, stems, and seeds.
3. BD (Buds): *Cannabis* in the form of buds or flowering tops of the plant with seeds.
4. SM (Sinsemilla): *Cannabis* in the form of flowering tops of the female plant with no seeds.
5. TS (Thai sticks): *Cannabis* in the form of leafy material tied around a small stem.

Hashish and Hash Oil—Samples were received in sealed plastic bags and were stored at room temperature. Analyses were performed within one month of receipt. Most samples were not received in this laboratory until the disposition of court cases. Thus the age of the samples varied from a few weeks to almost two years. No attempt was made to compensate for the loss of Δ^9-tetrahydrocannabinol (THC) during storage.

Domestically Cultivated Cannabis—*Cannabis* preparations (marijuana, buds, and sinsemilla) known to have been produced from plant material grown in the United States, while classified like other confiscated samples, are further identified as being domestically produced.

Analysis

Marijuana

All samples that were primarily classified as *Cannabis* plant material (as opposed to hashish or hash oil) were extracted according to the basic procedure of Lerner [*14*] and modified by Fetterman and Turner [*15*] and Turner and Hadley [*16*]. Briefly, the samples were manicured by passing through a 850 μm (No. 20) sieve to remove seeds and stems. Duplicate 1-g samples were each extracted simultaneously with 40 mL of spectrograde chloroform for 1 h at ambient termperature. Plant material was removed by filtration and the resulting liquor was concentrated by vacuum to a solvent-free greenish residue. The residue was then dissolved in 1.5 mL of absolute ethanol containing 15 mg of androst-4-ene-3,17-dione as the internal standard. The solution was subjected to an ultrasonic vibrator until all resin was in solution. A 0.1-μL aliquot of this solution was injected into the gas chromatograph.

Hashish

Samples were first prepared by grinding to a fine powder using a mortar and pestle or an electric blender. Duplicate 0.5-g samples were then extracted as described under the marijuana section.

Hashish Oil

Samples of hashish oil (0.5 g) were dissolved in 10 mL of absolute ethanol containing 100 mg of the internal standard and a 0.1-μL aliquot of this solution was injected. Some hashish oils have exhibited the unusual property of being insoluble in organic solvents, but soluble in water. These samples were prepared by partitioning a 0.5-g aliquot between 40 mL each of chloroform and water in a separatory funnel. After three extractions with chloroform, the extracts were combined, dried over anhydrous sodium sulfate, evaporated to dryness, and the residue dissolved in 1 mL of ethanolic solution containing the internal standard.

Gas Chromatographic Analysis

Gas chromatographic (GC) analyses were performed using Beckman GC72-5 or Hewlett Packard 5750A gas chromatographs equipped with flame ionization detectors. Two columns were used: (1) 2% OV-17 on Gas Chrom Q, 100–120 mesh (2.4-m [8-ft] glass, 6.35-mm [0.25-in.] outer diameter, 2-mm inner diameter); column oven, 210°C isothermal: inlet, 240°C; detector, 260°C; nitrogen carrier gas at a flow rate of 20 to 30 mL/min; and a head pressure of 207 to 276 kPa (30 to 40 psi) and (2) 6% OV-1 on Gas Chrom Q, 100–120 mesh 1.8-m [6-ft] glass, 6.35-mm [0.25-in.] outer diameter, 2-mm inner diameter); column oven, 180°C isothermal; inlet, 240°C; detector, 260°C; nitrogen carrier gas flow rate at 10 to 30 mL/min, and a head pressure of 138 to 176 kPa (20 to 40 psi).

The Beckman GC72-5 instruments were also equipped with Hewlett Packard automatic sampler systems model 7670A. Model HP7671A Automatic Sampler was installed on the HP5750A gas chromatograph. Each GC/Automatic Sampler unit was interfaced to a time-sharing data acquisition system.[2] Because the control unit of the automatic sampler allowed only a fixed duration of time between injection cycles and the computer data analysis time was variable, a circuit was designed that allowed the computer to control the automatic injection cycle.

The instruments were calibrated each time columns were changed and routinely checked for compliance with the calibration. Duplicate analyses were run on all samples.

[2]Digital Equipment Corp. PDP-8 Computer with a chromatographic data processor.

Peak Area Calculation

The chromatographic data processor monitored the analog signal of each gas chromatograph to establish a baseline value. The peak area was measured in millivolts and, based on established relative retention times, compared with the peak area of the internal standard. The results were reported as percent by dry weight.

Data Reporting

For the purpose of this manuscript, averages of cannabinoid concentration in different samples are reported in two ways.

Normalized Average—(also known as weighted average). The mean is adjusted to proportionally reflect the weight of each seizure in relation to the total weight of all seizures. This is calculated as follows:

$$\text{Normalized average} = \frac{\Sigma C_1 \times W_1, C_2 \times W_2, C_3 \times W_3 \ldots C_n \times W_n}{\Sigma W_1, W_2, W_3 \ldots W_n}$$

where $C_1, C_2, C_3, \ldots, C_n$ are cannabinoid concentrations in Seizures 1, 2, 3, . . ., and n. W_1, W_2, W_3, . . ., W_n are the weights of Seizures 1, 2, 3 . . ., and n.

Nonnormalized Average—This is the arithmetic average (simple mean) of all seizures regardless of the weights seized. It is obtained by dividing the sum of the percentage of cannabinoid of each seizure by the number of seizures.

Results and Discussions

As of December 1981, a total of 2169 samples representing 734 618.1 kg of confiscated *Cannabis* preparations have been analyzed for this report. Table 1 summarizes the average Δ^9-THC concentration and the number of samples confiscated under each drug group.

This overall picture shows that the potency increases according to plant parts used and manufacturing procedures, with hash oil being the most potent *Cannabis* preparation. Buds and sinsemilla, which are forms of *Cannabis* plant material, are shown to be much more potent than the average hash sample. This is in contrast to the common belief that hashish is more potent than preparations comprising *Cannabis* plant material.

TABLE 1—*Percent by dry weight of* Δ^9*-THC in different* Cannabis *preparations analyzed.*

	Number of Samples Analyzed[a]	Nonnormalized Average[b]
Kilobricks	579	0.56
Marijuana	876	1.45
Hashish	412	2.23
Buds	96	3.00
Sinsemilla	86	5.47
Hash oil	120	17.84

[a]Number of samples include some samples confiscated before 1972.
[b]See Experimental Procedure section for detailed explanation.

Marijuana

Table 2 shows a breakdown of the number of marijuana samples analyzed, by year seized and source from which received. The data show that most samples (68%) were received from DEA (PM samples) sources and that the number of large seizures (over 90 kg [200 lb]) confiscated by the DEA was on continuous annual increase for the years 1973 to 1977 when it reached its maximum (241 seizures). There was a significant drop in the number of seizures in 1978 (107 seizures) and an even more drastic decrease in 1979 (47 seizures). This apparent reduction in the illicit flow of marijuana into the United States could have been, at least in part, the result of the *Cannabis* eradication program initiated in 1976 by the Mexican government and intensified in 1977 [*17*].

Cannabinoids analyses were performed on all confiscated samples regardless of their source, and the average yearly concentration of each cannabinoid is reported in two ways. The normalized average takes in consideration both the number and weight of seizures while the nonnormalized is the simple average or the arithmetic average. Table 3 shows the normalized and nonnormalized average concentration of the major cannabinoids (Δ^9-THC, cannabidiol [CBD], cannabichromene [CBC], and cannabinol [CBN]) of illicit *Cannabis* by year seized. Figure 1 shows graphic presentation of the data for Δ^9-THC which reflects on the trends of overall potency of all illicit *Cannabis* seizures. While the nonnormalized average shows continuous and significant increase in Δ^9-THC concentration since 1975, the normalized average on the other hand showed an increase up to 1977 with slight decline in 1978 and a significant decline in 1979. This is attributed to a few large (in terms of weight) seizures of low potency during these two years. In 1978, for example, 37% of the total weight of the 130 *Cannabis* seizures came from only three seizures having Δ^9-THC concentrations of 0.36, 0.16, and 0.26%. In 1979, the figures were more pronounced, where 49% of the total weight of the 220 *Cannabis* seizures came from 3 seizures having Δ^9-THC concentrations of 0.34% (2 seizures) and 0.17% (1 seizure). This significantly affected the normalized but not the nonnormalized averages.

Regarding the other cannabinoids reported in Table 3, examination of CBD and CBC concentrations shows that most samples confiscated belong to the drug type *Cannabis* (low CBD, high CBC) [*18*]. However, the data for 1980 and 1981 indicate that more of the fiber or intermediate type *Cannabis* is beginning to contribute a significant portion of confiscated material. Six of the 151 samples confiscated in 1980 had CBD concentrations ranging from 1.12 to 3.06%. Thirty three of the 1981 confiscations (249) had CBD concentrations ranging from 1.02 to 6.67%. Additionally, of these 33 confiscations, 32 were from domestically grown material.

TABLE 2—*Number of* Cannabis *seizures analyzed by source from which received.*

Year Seized	FG	PD	PM	PS	ST	Total
Prior to 1972	33	23	6	1	2	65
1972	9	...	20	...	5	34
1973	11	7	15	...	...	33
1974	18	3	74	...	19	114
1975	11	...	123	...	15	149
1976	5	...	204	...	...	209
1977	4	1	241	2	3	251
1978	5	1	107	17	...	130
1979	162[a]	3	47	6	2	220
1980	31	21	75	13	11	151
1981	1	6	176	16	50	249
Total	290	65	1088	55	107	1605

[a]Includes 150 samples received from Mexican government in conjunction with the U.S. Departments of State and Agriculture.

TABLE 3—*Normalized*[a] *versus nonnormalized*[a] *cannabinoid averages of illicit* Cannabis *samples by year seized.*

Year	Number of Seizures	Δ^9-THC, %		CBD, %		CBC, %		CBN, %		Total Weight of Seizures in Kg[b]
		Normalized	Nonnormalized	Normalized	Nonnormalized	Normalized	Nonnormalized	Normalized	Nonnormalized	
1972	34	0.18	1.17	0.04	0.28	0.09	0.01	1.78	0.29	15.3
1973	33	0.22	0.72	0.00	0.06	0.15	0.05	0.98	0.61	3 367.3
1974	114	0.36	0.92	0.00	0.03	0.08	0.08	0.44	0.49	18 013.3
1975	149	0.48	0.71	0.00	0.03	0.08	0.11	1.18	0.55	67 136.8
1976	209	0.98	0.73	0.00	0.00	0.12	0.09	0.62	0.37	101 168.3
1977	251	1.76	0.91	0.00	0.08	0.10	0.10	0.74	0.43	173 611.1
1978	130	1.73	1.39	0.01	0.01	0.12	0.12	1.28	0.66	153 296.2
1979	220	1.53	1.67	0.02	0.02	0.12	0.12	1.40	0.23	71 859.1
1980	151	2.15	2.09	0.01	0.11	0.16	0.14	0.68	0.47	39 860.0
1981	249	2.11	2.37	0.02	0.36	0.18	0.16	0.98	0.38	147 033.6

[a]See Experimental Procedure section for detailed explanation.
[b]Weights are rounded to one decimal place.

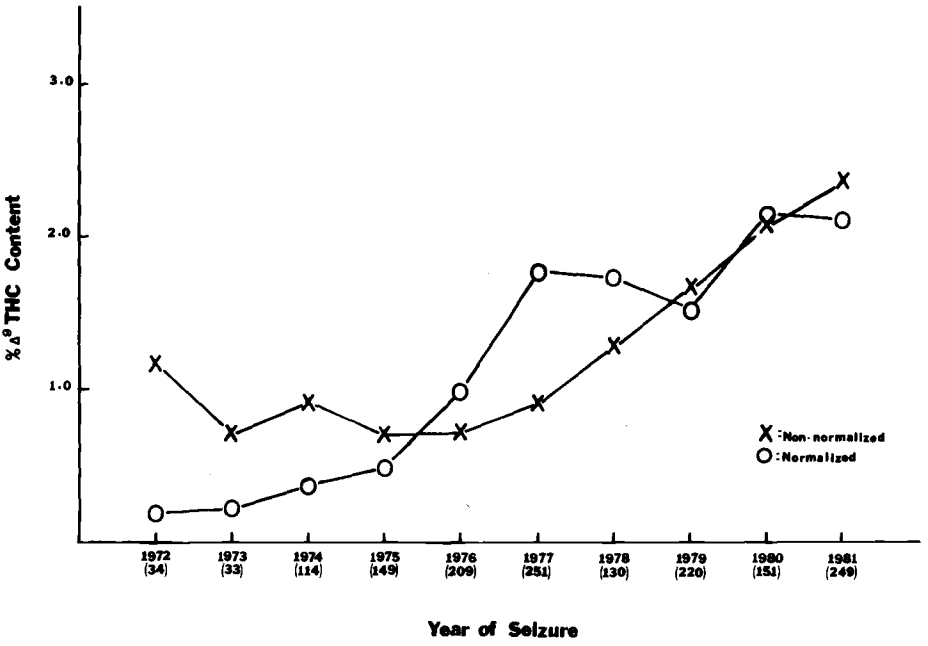

FIG. 1—*Normalized and nonnormalized percentage of Δ^9-THC versus year of confiscation (number of seizures in parentheses).*

The concentration of CBN is a good indication of the age of samples [*19,20*] as well as the storage condition [*21*]. Table 3 shows significantly high averages of CBN indicating either old plant material or poor storage conditions [*21*].

As indicated earlier (see Experimental Procedure section), *Cannabis* plant material appears in the illicit market in different forms (buds, kilobricks, marijuana, sinsemilla, and Thai sticks, see Figs. 2 through 5) that reflects to some extent on its country of origin. Table 4 shows the different forms of illicit *Cannabis*, the number of seizures, and the concentration of Δ^9-THC in each case by year confiscated. In addition, Fig. 6 shows a barographic comparison of the average potency of the different forms of *Cannabis* plant material over the period of the study. Sinsemilla is by far the most potent preparation followed by buds and marijuana (loose plant material) while kilobricks are the least potent.

Kilobricks are the classical form of Mexican produced marijuana. It is evident from Table 4 that kilobricks were the most abundant *Cannabis* preparation up to 1978 and peaked in 1976 (182 seizures) and 1977 (165 seizures). The number of kilobrick seizures was drastically reduced in the last four years of this study (1978 to 1981) to five seizures in 1980 and three seizures in 1981. This could be attributed to the *Cannabis* eradication program in Mexico.

Note that confiscated sinsemilla appeared first in 1977 and has become the second most abundant form of confiscated *Cannabis* since 1980. It is also important to indicate that almost all the sinsemilla seizures were known to be domestically produced in the United States (compare Tables 4 and 5). Sinsemilla, by definition, is produced from the unfertilized flowering tops of female *Cannabis* plants. This is attained by removal of all male plants from the growing field at the first sign of their appearance. It follows that the production of sinsemilla requires a great deal of attention and care and is therefore cultivated on small plots. It is estimated that one full-time person can handle 40 plants because of the method of production.

FIG. 2—*Kilobricks of illicit* Cannabis *plant material.*

Sinsemilla is the most potent preparation of *Cannabis* when drug type seeds are used in planting. Table 4 compares the average (normalized and nonnormalized) Δ^9-THC concentration of the different forms of *Cannabis* and shows that the potency follows the order sinsemilla > buds > marijuana > kilobricks.

Cannabis Domestically Cultivated in the United States

Table 5 shows the different drug forms of the confiscated material known to have been domestically produced, the number of seizures, and the cannabinoid analysis by year seized. The data presented in Table 5 show that the number of seizures known to be of domestic material has significantly increased in the last two years of this study (1980 and 1981). The percent of all *Cannabis* seizures known to be of domestic origin has risen from an average of about 6% in 1979 (and the previous years) to 23% in 1980 and over 50% in 1981.

The potency of domestic *Cannabis* has increased gradually from 1978 to 1980 where the average Δ^9-THC reached its maximum of 4.64%. However, the average Δ^9-THC concentration fell in 1981 down to 2.92%. This is attributed to the fact that in 1980 most of the domestically produced drug was in the form of sinsemilla which represented about 70% the seizures (24/35). In 1981, however, sinsemilla represented only about 23% of the seizures (30/129). Figure 7 shows the yearly averages of Δ^9-THC in domestically produced *Cannabis*.

Domestically produced *Cannabis* plant material was in the form of buds, marijuana (loose material), or sinsemilla. Sinsemilla was the most abundant form of domestically produced drug up to 1980. Since 1980, seizures of loose marijuana known to be domestically produced have exceeded those of sinsemilla (80 versus 30). This might indicate that domestic marijuana production in the United States is increasingly getting in the hands of less experienced growers of

FIG. 3—*Loose marijuana of illicit* Cannabis *plant material.*

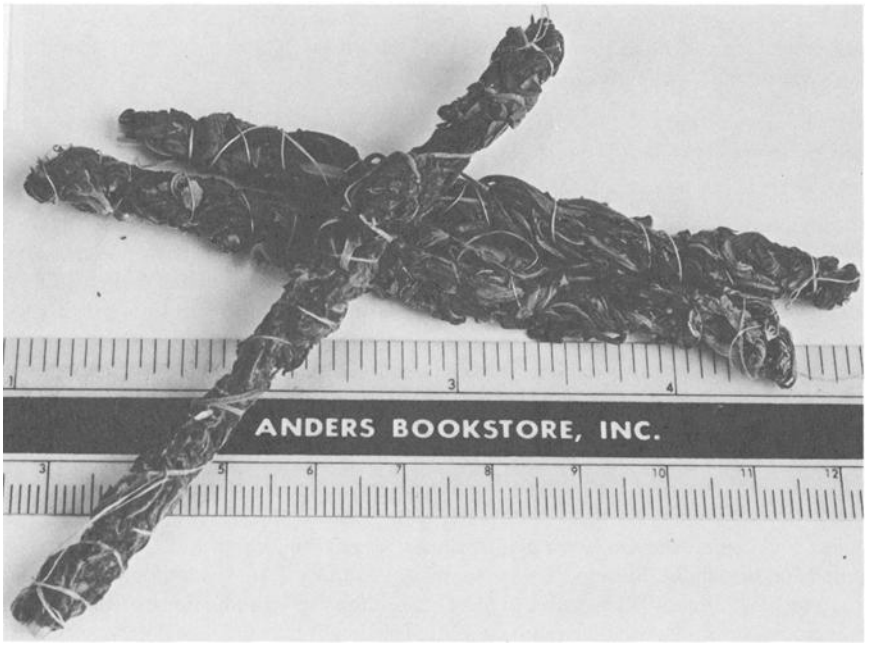

FIG. 4—*Thai sticks of illicit* Cannabis *plant material.*

TABLE 4—*Normalized[a] versus nonnormalized[a] Δ^9-THC content of illicit* Cannabis *samples in different forms by year confiscated.*

	Buds			Kilobricks			Marijuana			Sinsemilla			Thai Sticks		
Year	Norm.	Nonnorm.	No. of Seizures	Norm.	Nonnorm.	No. of Seizures	Norm.	Nonnorm.	No. of Seizures	Norm.	Nonnorm.	No. of Seizures	Norm.	Nonnorm.	No. of Seizures
1972	0.19	0.21	2	...	...	0	0.11	1.23	32	...	...	0	...	...	0
1973	...	...	0	0.13	0.21	6	0.27	0.83	27	...	...	0	...	...	0
1974	...	...	0	0.21	0.40	50	0.53	1.34	63	...	...	0	0.54	0.54	1
1975	0.96	1.34	3	0.53	0.47	88	0.44	1.05	58	...	...	0	...	...	0
1976	3.03	3.03	1	0.85	0.54	182	1.60	1.94	26	...	...	0	...	...	0
1977	0.53	1.38	7	0.47	0.53	165	2.28	1.27	63	4.25	3.20	15	4.91	4.91	1
1978	2.44	2.11	25	1.54	0.96	60	1.52	1.47	43	6.28	6.28	1	0.82	0.82	1
1979	3.35	3.03	11	1.26	0.79	18	0.55	1.58	180	3.52	3.66	10	0.13	0.13	1
1980	4.26	3.81	6	0.91	0.63	5	0.74	1.04	112	3.60	6.40	27	0.05	0.05	1
1981	4.46	3.52	33	0.81	0.78	3	1.64	1.48	181	4.10	6.38	32	...	...	0

[a]See Experimental Procedure section for detailed explanation.

FIG. 5—*Sinsemilla, no seeds* (left) *and buds, with seeds* (right) *of illicit* Cannabis *plant material.*

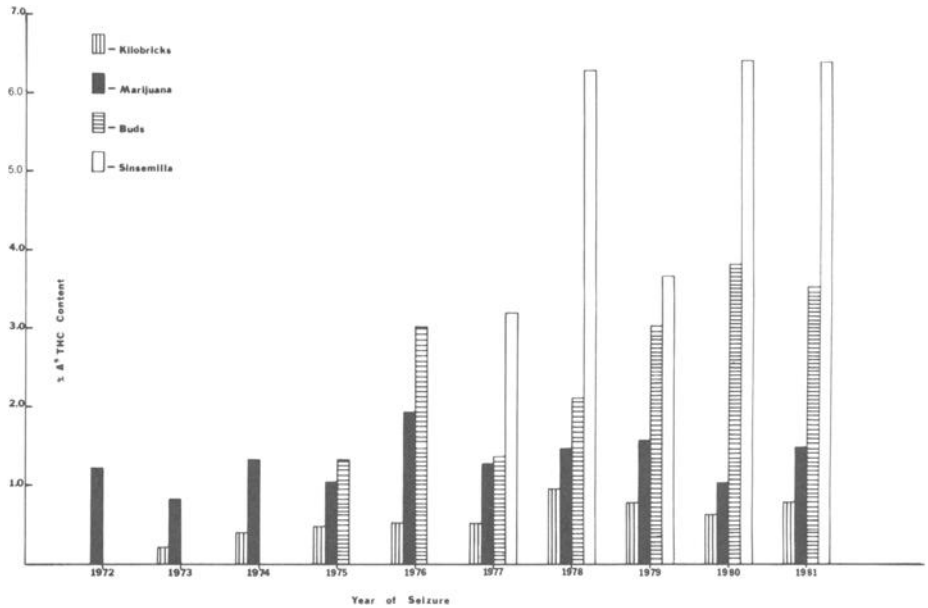

FIG. 6—*Nonnormalized percentage of* Δ^9*-THC content of illicit* Cannabis *samples in different forms versus year of confiscation.*

TABLE 5—*Nonnormalized*[a] *cannabinoid averages of domestically cultivated* Cannabis *samples by physical description and year seized.*

Year	% of All *Cannabis* Seizures Known to be Domestic	Buds					Marijuana					Sinsemilla				
		% Cannabinoids				No. of Seizures	% Cannabinoids				No. of Seizures	% Cannabinoids				No. of Seizures
		Δ^9-THC	CBD	CBC	CBN		Δ^9-THC	CBD	CBC	CBN		Δ^9-THC	CBD	CBC	CBN	
1972	5.9	...	...	...	...	0	1.43	0.10	0.00	0.07	2	...	...	...	...	0
1975	6.0	...	...	...	...	0	1.24	0.00	0.19	0.02	9	...	...	...	...	0
1977	6.4	...	...	...	...	0	0.31	0.00	0.01	0.42	1	3.20	1.26	0.26	0.18	15
1978	9.2	1.68	0.02	0.19	0.24	1	1.42	0.01	0.12	0.15	10	6.28	0.02	0.20	0.22	1
1979	6.4	...	...	...	...	0	3.95	0.06	0.15	0.12	4	3.66	0.28	0.22	0.22	10
1980	23.2	...	...	...	...	0	0.72	0.46	0.08	0.07	11	6.44	0.35	0.22	0.11	24
1981	51.8	2.87	0.51	0.14	0.07	19	1.62	0.62	0.17	0.05	80	6.42	0.70	0.22	0.10	30

[a]See Experimental Procedure section for detailed explanation.

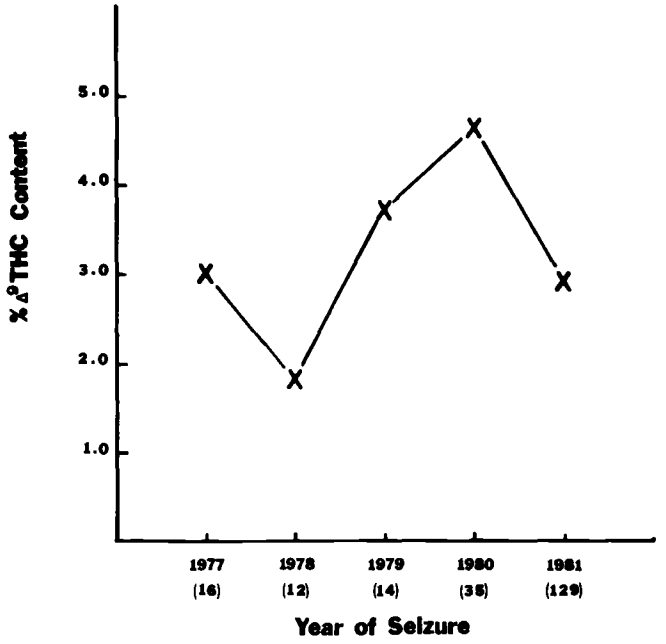

FIG. 7—*Nonnormalized percentage of* Δ^9*-THC content of domestically cultivated* Cannabis *versus year of seizure (number of seizures in parentheses).*

Cannabis. Alternatively, the increase in domestic production might be due to increased acreage under cultivation by professional growers. In a forthcoming publication, we will discuss, in more detail, the characteristics of domestically produced *Cannabis*, locations, methods of production, and their potencies.

Hashish

Table 6 shows the cannabinoid analysis of hashish samples confiscated between 1972 and 1981 by year seized. The average Δ^9-THC concentration (nonnormalized) ranged from 0.6 to 3.37%. Although there is a slight indication that the potency of hashish is increasing yearly, particularly during the last five years, examining the individual hashish samples showed no unusual reason for the increase.

Note that the number of hashish seizures (Table 6) has shown a decline over the years with almost a record low in 1981. This could be attributed to the wars in Lebanon and Afghanistan which are two of the major hashish producing countries. In addition, the demand for hashish has probably declined with the availability of more potent marihuana, particularly sinsemilla and buds, in the last few years.

Hash Oil

Table 7 shows the cannabinoid analysis of confiscated hash oil, the number of seizures, and the total weight seized by year confiscated. The average potency of hash oil has been reasonably consistent over the years. The number of seizures, however, has been declining since 1975 and reached its lowest level in 1981 (four seizures of small amounts). This could be at-

TABLE 6—*Nonnormalized*[a] *average concentrations of four cannabinoids found in hashish samples.*

Year	No. of Seizures	Δ^9-THC, %	CBD, %	CBC, %	CBN, %	Total Weight of Seizures in Kg
1972	6	0.60	2.25	0.30	2.24	2.0
1973	11	1.00	3.19	0.43	1.94	531.7
1974	53	0.86	1.99	0.28	2.28	2195.6
1975	87	2.34	2.62	0.39	1.68	2040.6
1976	52	3.28	3.23	0.37	2.54	3093.0
1977	44	1.81	2.94	0.22	1.72	687.1
1978	47	2.20	3.91	0.24	2.13	2226.7
1979	43	2.32	5.45	0.16	1.76	383.3
1980	36	2.60	7.79	0.12	1.86	967.3
1981	10	3.37	7.10	0.30	1.57	12.8

[a]See Experimental Procedure section for detailed explanation.

TABLE 7—*Nonnormalized*[a] *average concentrations of four cannabinoids found in hash oil samples.*

Year	No. of Seizures	Δ^9-THC, %	CBD, %	CBC, %	CBN, %	Total Weight of Seizures in Kg
1972	0	...	...	...	...	...
1973	6	22.00	10.77	1.51	6.11	28.7
1974	18	15.34	11.42	1.43	3.93	20.4
1975	29	13.09	6.71	0.86	4.21	16.9
1976	18	18.82	10.25	1.16	5.07	31.6
1977	17	18.89	6.83	0.57	4.98	42.6
1978	9	21.31	6.06	1.39	5.07	26.1
1979	9	20.91	0.57	1.54	6.00	8.2
1980	8	16.56	8.67	1.02	5.30	23.8
1981	4	19.79	4.70	1.69	3.59	0.5

[a]See Experimental Procedure section for detailed explanation.

tributed to the decreased demand because of more potent marijuana being made available in the illicit market.

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