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Cannabinoid Content of Fifty Seized Hashish Samples

During the last few years, police seizures of hashish have increased rapidly in Sweden. A large part of the seized samples have been sent to this laboratory for analysis, and the case load has risen accordingly (Table 1).

TABLE 1—*Increase of case load for the analysis of Cannabis sativa and products obtained from this plant.*

Year	1965	1966	1967	1968	1969	1970
Number of cases	25	51	90	169	230	244

In view of the rapid spread of the abuse of cannabis, the prosecuting authorities decided to establish certain rules pertaining to the severity of this offense. One of the criteria for judging this is the amount of material seized. Another is its content of pharmacologically active constituents.

Thus, a method for quantitative analysis of cannabis as well as a knowledge of the content of active constituents in illicit hashish was needed.

Gas chromatographic analysis of cannabis constituents was first introduced by Farmilo and Davis [1]. This is the technique of choice for the quantitative determination of cannabinoids. Various stationary phases have been recommended for this purpose such as Apiezon M [2], Carbowax [3], and miscellaneous silicones [4-15].

The precision of the method is improved by the use of an internal standard. Among various internal standards such as *n*-eicosane [5], dibenzylphthalate [3,16], tetraphenyl ethylene [6], methadone [14], methyl stearate [10] and caffeine [15], triphenylcarbinol was found to be the best choice for the JXR methyl silicone stationary phase as suggested by Agurell and Leander [17].

The cannabinoid content of nine samples of hashish was reported in a paper by Machata [10] who also cited the earlier literature in this respect. In a total of five published reports, 27 samples of marijuana and hashish were analyzed. The content of Δ^1 -tetrahydrocannabinol (Δ^1 -THC) varied from 0 to 8.0 percent. Only 12 samples contained more than 1.0 percent of this cannabinoid.

Since the literature data were considered to be insufficient for obtaining a clear picture of cannabinoid content found in illicit hashish samples, a systematic gas chromatographic

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study of this problem was undertaken at the beginning of 1970. In this paper the cannabinoid content of 50 hashish samples seized by Swedish police or customs authorities during the years 1970 to 1972 is reported.

Experimental

All seizures covered in this report had the appearance of pressed resinous material of the hashish type. Each sample had undergone botanical identification, based on positive findings of cystolith hairs and glandular hairs as found in *Cannabis sativa*. Extracts were then analyzed by chromatography on silica gel thin-layer plates, using hexane:ether 4:1 as the developing solvent, and Echtblausalz B (Diazo blue B) in the detecting spray.

A Perkin-Elmer F11 gas chromatograph with flame ionization detector and all glass system was used in connection with a Perkin-Elmer 159 recorder.

Gas chromatography was carried out using JXR methyl silicone as the stationary phase as suggested by Ohlsson et al [8]. One gram of the powdered hashish material was extracted with methylene chloride in a semi-micro Soxhlet extractor until the extract appeared colorless (2 to 4 h). The extract was then evaporated to a small volume and transferred to a 2-ml volumetric flask containing 10 mg of triphenyl carbinol. One μ l of this solution was injected into the gas chromatograph under the following conditions.

Column: outside diameter—6 mm, inside diameter—3 mm glass tube 2.7 m long, 130-mm coil diameter, packed with Gas-Chrom Q (100/120 mesh), coated with 5 percent JXR methyl silicone.

Inlet pressures: carrier gas (N_2)—1.7 atm
hydrogen—1.4 atm
air—1.8 atm

Temperatures: column—230 C
injector—230 C

Results and Discussion

Calibration was carried out by chromatographing 6 solutions, each containing known amounts of cannabidiol (CBD) and triphenylcarbinol. A good linear relationship was obtained between concentration ratios and peak area ratios.

Since Δ 1-tetrahydrocannabinol (Δ 1-THC) and cannabinol (CBN) standards were not available, relative response factors for these compounds from the literature [4] were employed.

The cannabinoid content of the 50 samples are shown in Table 2. Since cannabinoid acids readily decarboxylate in the injection system of the gas chromatograph, the figures actually represent the sums of the content of the respective acids and their corresponding free phenols. The mean values and standard deviations obtained for the content of the three cannabinoids are listed in Table 3. Figure 1 shows a histogram of the Δ 1-THC content found in the 50 samples.

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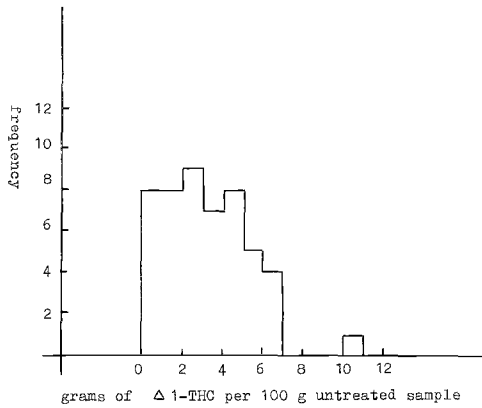


FIG. 1—Histogram of the $\Delta 1$ -THC content of hashish samples listed in Table 2.

TABLE 2—Content of three cannabinoids in 50 consecutive samples of hashish.

Sample	Cannabinoid content, % wt./wt.			Sample	Cannabinoid content, % wt./wt.		
	CBD	$\Delta 1$ -THC	CBN		CBD	$\Delta 1$ -THC	CBN
1714	7.8	2.1	2.3	662.2	4.0	4.3	1.8
2261.1	1.3	0.6	0.4	670.1	7.5	4.5	0.6
2261.2	0.4	0.3	0.2	670.2	7.1	4.3	0.7
2261.3	0.4	0.3	0.2	670.3	8.0	5.0	0.7
2262.1	0.9	2.0	0.2	670.1A	10.4	6.1	1.2
2262.2	1.1	6.1	0.3	670.2A	7.4	4.3	0.5
2304	2.0	2.3	0.4	670.3A	8.0	5.0	0.5
448.2A	7.3	3.7	0.9	670.2B	7.8	4.7	0.5
448.3A	1.5	1.9	0.1	720	4.8	6.1	1.5
448.4A	7.5	3.5	0.8	733	6.5	0.3	2.3
448.9A	1.6	2.1	0.2	852	1.2	1.7	0.3
448.15A	7.7	3.3	0.8	935	0.9	5.0	0.3
448.9D	6.7	3.2	0.7	967	4.7	6.7	1.4
527.1	1.8	2.2	0.2	981	2.6	10.5	0.7
527.2	0.8	1.0	0.1	1088.1	0.9	3.9	0.4
533	0.5	1.6	0.2	1088.2	2.4	4.1	0.3
X1	1.7	3.5	0.7	1101.1	0.3	1.0	0.1
X2	1.0	2.0	0.4	1101.2	0.1	0.5	0.1
X3	1.6	2.8	0.9	1101.3	0.2	0.6	0.1
X4	1.1	2.7	0.5	1101.4	0.1	0.6	0.1
549	1.4	1.2	0.3	1101.5	0.2	0.6	0.1
627.1	4.9	4.8	2.1	1101.6	2.1	2.6	0.6
627.2	5.1	5.2	2.2	1123.1	9.1	5.8	0.6
652	1.6	1.4	0.2	1123.2	2.3	3.5	0.4
662.1	3.9	4.3	1.7	1146	0.1	1.7	0.3

TABLE 3—Average content of cannabinoids in 50 samples analyzed.

	CBD	$\Delta 1$ -THC	CBN
Mean value	3.4	3.2	0.7
Standard deviation	3.1	2.1	0.6

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