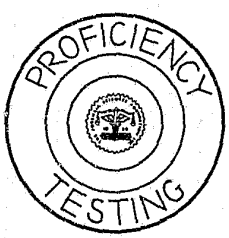
LABORATORY PROFICIENCY TESTING PROGRAM

REPORT NO. 1

CONTROLLED SUBSTANCE



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LABORATORY PROFICIENCY TESTING PROGRAM

CONTROLLED SUBSTANCE

Prepared under Grant 74 NI-99-0048 from the National Institute of Law Enforcement and Criminal Justice, Law Enforcement Assistance Administration, Department of Justice.

Points of view or opinions stated in this document are those of the authors and do not necessarily represent the official position or policies of the Department of Justice.

FOREWORD

The analysis summarized in this report is the first of ten such analyses that will be made in conjunction with this proficiency testing research project.

In the course of this testing program participating laboratories will have analyzed and identified ten different samples of physical evidence similar in nature to the types of evidence normally submitted to them for analysis.

"Test Sample #1" involved the identification of a federally regulated controlled substance (a narcotic or dangerous drug). Specifically, each laboratory was asked to:

1) Examine the substance according to its normal laboratory procedures.

2) Identify the substance in a manner which complied with its reporting policy.

3) Indicate the analytical method(s) used.

4. Include any additional data (quantitative or qualitative) that it routinely developed in such cases.

The results of Test Number One are reflected in the charts and graphs which follow.

At a later date, individual test reports will be grouped and studied as a basis for the design of laboratory educational programs.

Comments or suggestions relating to any portion of this report or of the program in general will be appreciated.

March 1975

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BACKGROUND

This laboratory proficiency testing research project, one phase of which is summarized in this report, was initiated in the fall of 1974 with the following objectives.

> Through the use of voluntary, anonymous proficiency testing, assess the analytical accuracy of criminalistic laboratories in the processing of selected physical evidence.
>
> Make statistical studies of laboratory proficiency in the processing of openly submitted test samples and of the accuracy and precision of the various analytical methods used.
>
> Establish the basis for the design of educational programs in the areas of analytic methods, to the end that they will assist the criminalistics profession in the attainment of higher levels of proficiency.

A total of ten samples will be prepared and submitted to the participating laboratories in two cycles during the course of this eighteen month project.

In Cycle I five samples of the following types will be distributed: a controlled substance; firearms evidence; blood; paint; glass. In Cycle II the same categories of samples will be repeated (with different properties and property levels).

As noted earlier, participation in the program is voluntary. Accordingly, invitations have been extended to 236 laboratories to share in the research. It is recognized that all laboratories do not perform analyses of all possible types of physical evidence. Thus, in the data summaries included in this report, space opposite some Code Numbers (representing specific laboratories) may be blank, or marked "No Data Returned."

The procedures used in the processing of each test sample are indicated below.

Sample Manufacture - Once the Project Advisory Committee has selected a sample and has determined its precise specifications, a manufacturer is selected. The manufacturer may be a commercial

enterprise, an agency of government, or it may be one of the participating laboratories. The criteria used in selecting the manufacturer are: quality and cost.

Referee Laboratories - Simultaneously, Referee Laboratories are selected to provide reliable evidence of the characteristics of the sample. Again, agencies or laboratories are selected whose quality of work in this area is known.

Sample Mailing - The delivered samples are packaged, necessary forms and letters are prepared and on a specified date the samples are mailed as Certified, First Class material. It should be noted that the laboratories are asked to process, analyze and report their findings in the manner normally used by them when dealing with that type of physical evidence.

Quick Report - Data Sheets received from the participating laboratories are acknowledged through the use of a Quick Report which identifies the particular sample involved in the test, and summarizes the Referee Laboratory results. This report allows the participants to determine if their responses were correct.

Test Analysis Report - After all results are in, a Test Analysis Report, such as this, is prepared and mailed to all laboratories regardless of whether or not they participated in any given test.

At some subsequent date, the results from groups of tests will be studied to the end that educational programs can be designed.

And, of course, a final report will be prepared at the conclusion of Cycle II.

The Project is under the direct control of the Project Advisory Committee whose members' names are listed on the Title Page. Each is a nationally known criminalistic laboratory authority.

Supporting the Project Advisory Committee in their efforts is the Forensic Sciences Foundation with additional support from the National Bureau of Standards in the areas of sample evaluation and data analysis and interpretation.

CONCLUSIONS

Test Sample #1, a controlled substance, was mailed to 236 laboratories throughout the United States and its possessions. (Unfortunately, problems related to secured delivery precluded sending the samples to the Canadian laboratories that are participating in the overall project.)

On the accompanying information summaries, 200 laboratories are shown as having submitted Data Sheets in response to the test. Actually 203 laboratories responded but three Data Sheets were received too late to be included in the summaries. This represents a raw participation rate of 86%. However, when the eight laboratories are removed who formally indicated that they do not perform such tests, the participation rate was approximately 90%.

No effort was made in this report to highlight areas wherein laboratory improvement programs might be instigated. Insufficient data and information are available at this time to support such decisions.

ANNEX A

FIGURE 1

Lab Code A-_____

PROFICIENCY TESTING PROGRAM

TEST NO. 1

Examine according to your normal laboratory procedures and complete portion(s) below which complies with your laboratory policy.

1. (a) What is the controlled (narcotic or dangerous drug) substance_____

(b) Indicate method(s) used.

2. (a) Please add any other data (quantitative -qualitative) that you routinely develop.

(b) Indicate method(s) used.

IMPORTANT

DO NOT SIGN THIS DATA SHEET OR IN ANY OTHER WAY IDENTIFY YOUR LABORATORY.

RETURN COPY TO: KENNETH S. FIELD, FORENSIC SCIENCES FOUNDATION, SUITE 515, 11400 ROCKVILLE PIKE, ROCKVILLE, MARYLAND 20852.

ANNEX B

National Bureau of Standards Analysis
LABORATORY PROFICIENCY TESTING PROGRAM
Test No. 1 Controlled Substance

A one-half gram sample marked only as a Controlled Substance, was sent to each of 230 laboratories throughout the United States for identification. The substance, as described by the manufacturer, was 74 ±5% sodium pentobarbital, the remainder being starch. This was verified by two referee laboratories.

Table 1 summarizes the findings of the laboratories regarding their identification of the substance, and Table 2 shows schematically the relationships among the drug names used by the laboratories to describe the drug found. The tests that were used are shown in Table 3 and Table 4. Quantitative results were submitted by twenty four laboratories as shown in Table 5. A detailed list of laboratory responses to the four questions on the questionnaire is given in Table 6.

This annex was prepared by the Law Enforcement Standards Laboratory of NBS in conjunction with the NBS Laboratory Evaluation Technology Section (LETS). The anonymous test results reported by the participating forensic laboratories were analyzed and tabulated by Jeffrey Horlick and Charles G. Leete of LETS. This work was supported by National Institute of Law Enforcement and Criminal Justice, Department of Justice.

TABLE 1

CONTROLLED (NARCOTIC OR DANGEROUS DRUG) SUBSTANCE FOUND

Part I of this table names the drug found as the laboratory would normally report it. If more than one name was used in answer to question 1a, the more descriptive name was counted in Part I. Drug reporting may involve state law, laboratory procedure, or reporter's discretion. Part II names the drug as actually identified. An example of this is laboratory A758 which reported "a barbituric acid derivative", according to state law, but added that further analysis indicated sodium pentobarbital.

Reported name of substance	Part I As normally reported		Part II As actually identified	
	number of labs reporting	percentage of total labs reporting	number of labs reporting	percentage of total labs reporting
1. barbiturate	8	4%	5	2.5%
2. barbituric acid derivative	15	7.5	8	4
3. pentobarbital 5-ethyl-5(1-methylbutyl) barbituric acid	136	68	138	69
4. soluble pentobarbital salt of pentobarbital	4	2	4	2
5. sodium pentobarbital pentobarbital sodium	24	12	30	15
6. amobarbital	2	1	3	1.5
7. butabarbital	4	2	4	2
8. secobarbital	2	1	2	1
9. phenobarbital	1	.5	1	.5
10. sodium butabarbital	1	.5	1	.5
11. sodium secobarbital	1	.5	2	1
*12. barbituric acid	--	--	--	--
13. librium	1	.5	1	.5
14. no drug found	1	.5	1	.5

* Reported as a product of an intermediate analysis.

TABLE 2

RELATIONSHIP AMONG THE DRUG NAMES USED

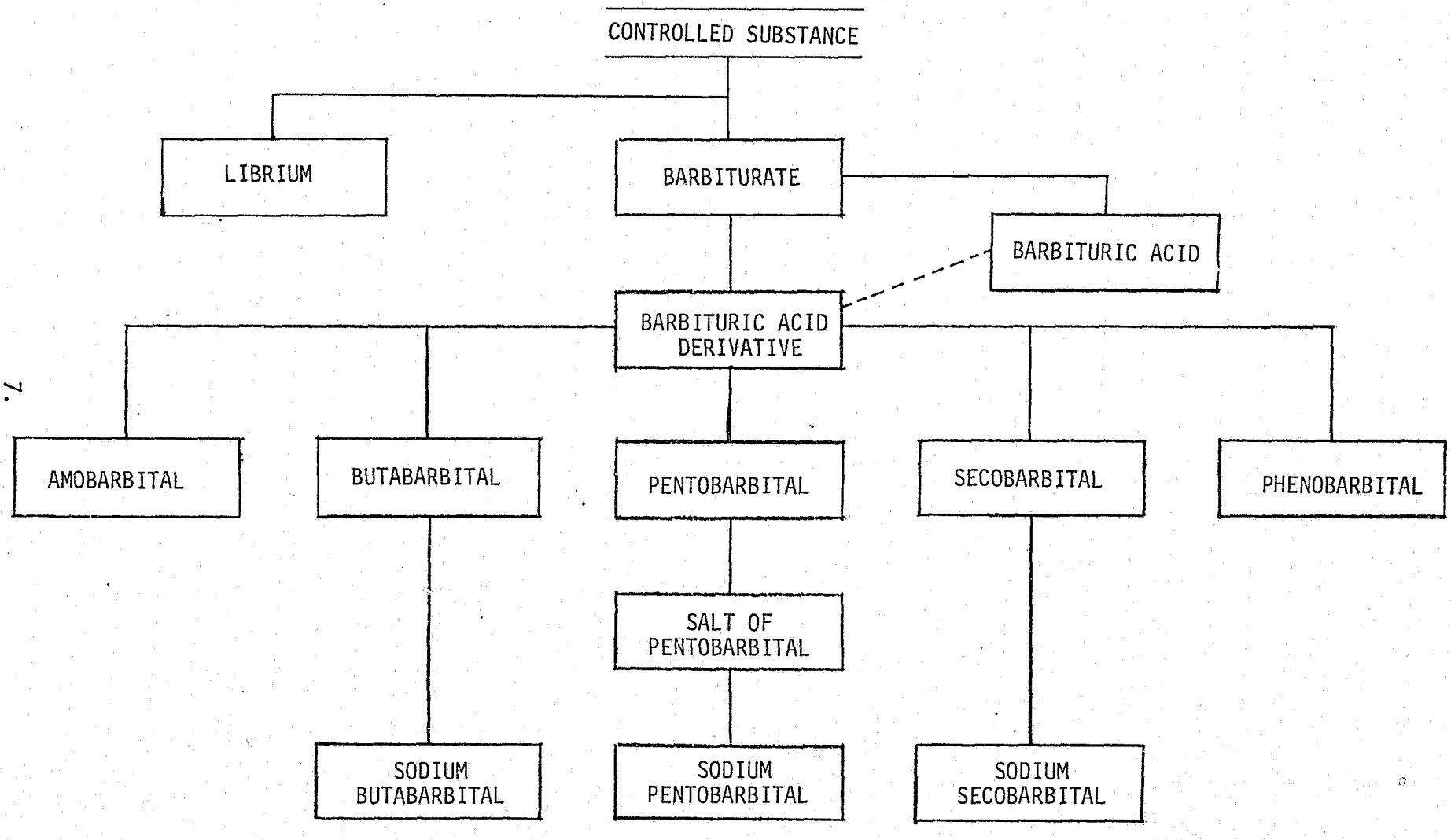

TABLE 3

METHODS USED IN DETERMINING SUBSTANCE

This table gives the number of laboratories which used each type of test. Since most laboratories used more than one test, the total number of tests performed is more than the total number of laboratories.

	Test or method	Number of laboratories	% of total labs (total=200)
A	Color Tests	166	83%
B	$KMnO_4$	2	1%
C	Crystalline Tests	97	49%
D	Commercial Kit	1	.5%
E	Flame Test	2	1%
F	Melting Point	13	7%
G	TLC	50	25%
H	UV	121	61%
I	IR	99	50%
J	NMR	3	2%
K	GC	79	40%
L	GC/MS	7	4%
M	MS	3	2%

TABLE 4

INSTRUMENTS AND METHODS
(IN APPROXIMATE ORDER FROM PRESUMPTIVE TO DEFINITIVE)

A SCREENING COLOR TESTS [PRESUMPTIVE]

1. Koppanyi Reagent
2. Dille-Koppanyi Spot Color Test [cobalt acetate-isopropyl amine, test for barbiturates]
3. Zwicker's [copper sulfate-pyridine, test for barbiturates]
4. Mayer's [screening test], positive for alkaloids
5. Marquis' [screening test, positive for alkaloids and amphetamine]
6. Mecke [screening test, positive for alkaloids and amphetamine]
7. fluorescence in tartaric acid
8. PDMB [p-dimethylaminobenzaldehyde, screening test, positive for LSD]
9. cobalt(II) thiocyanate [$Co(CNS)_2$, screening test, positive for cocaine type materials]
10. Furfural/HCl
11. Froehde's [screening test]
12. Liebermann's [screening test]
13. Parri [Dille-Koppanyi]
14. VanUrk
15. cobalt nitrate [$Co(NO_3)_2$, screening test]
16. Sanchez

B POTASSIUM PERMANGANATE FOR SECOBARBITAL ($KMnO_4$)

C CRYSTALLINE TESTS

1. Wagenaar's Reagent [copper sulphate-ethylenediamine, positive for barbiturates]
2. Davis Silver Reagent
3. sulphuric acid and water (H_2SO_4-H_2O)
4. potassium hydroxide and phosphoric acid (KOH-H_3PO_4)
5. Wagner's reagent (I_2-KI)
6. potassium iodide and phosphoric acid (KI-H_3PO_4)
7. pptd free acid, microscopic recognition
8. perchloric acid ($HClO_4$)
9. gold chloride

D COMMERCIALLY AVAILABLE ANALYSIS KIT

E FLAME TEST

F MELTING POINT

1. melting point
2. mixed melting point

TABLE 4
CONTINUED

G [THIN LAYER CHROMATOGRAPHY] TLC

H UV [ULTRAVIOLET SPECTROPHOTOMETRY] FOR IDENTIFICATION

I IR [INFRARED SPECTROPHOTOMETRY] FOR IDENTIFICATION

J NMR [NUCLEAR MAGNETIC RESONANCE]

K GAS CHROMATOGRAPHY (including: 1) gas chromatography-GC, 2) gas-liquid chromatography-GLC, 3) vapor phase chromatography-VPC)

L GAS CHROMATOGRAPHY/MASS SPECTROMETRY [GC/MS]

M MASS SPECTROMETRY [MS]

TABLE 5

% CONCENTRATION OF SUBSTANCE [1]

Two samples were found by two referee laboratories to contain sodium pentobarbital and starch. The sodium pentobarbital assayed at 70.4 and 71.4 percent, respectively.

LAB CODE	SODIUM PENTOBARBITAL	PENTOBARBITAL OR FREE ACID	PENTOBARBITAL BY WEIGHT	NOT SPECIFIED
A726	76.4	65		
A747	(71.3)	53.7		
A748	(58.9)			
A763				76.3
A765	78			
A785	78.1			
A789				45.9
A813				67
A824			67	
A837	(106)*	97 *		
A842	75			
A844			56.4	
A872	(68.6)	62.5		
A907				11 *
A917	80			
A925	82.2	74.9		
A927	78			
A937				80+ *
A961				58
A970	(75.7)	69		
A974	35.7*			
A975	(81.2)	74		
A983			75	
A989	(60.3)	55		
AVERAGE	78.2 [2] 74.1 [4]	64.9 [3]	66.1	61.8 [3]

NOTE 1: Determination and reporting of the percent concentration was done by the laboratories on a totally voluntary basis.

NOTE 2: Average of seven values not including * and ( ). Values in ( ) are free acid values converted to sodium salt values correcting for molecular weight.

NOTE 3: Average not including *

NOTE 4: Average of thirteen values not including *

TABLE 6

TABULATION OF LABORATORY RESPONSES

LAB CODE	1a CONTROLLED SUBSTANCE FOUND (see table 1) I	II	1b INSTRUMENTS AND METHODS (see table 4)	2a ADDITIONAL INFORMATION REPORTED	2b ADDITIONAL INSTRUMENTS AND METHODS
A703	3		A2,3;I;K1		
A705	5		A;C;H;I;K2		
A706	3		A;G;H;K2;L		
A708	3		A2;G;H;K1		
A709	3		A;H;K2		
A710	NO DATA RETURNED				
A711	5		A2;I		
A712	3		A;C;H;I		
A713	3		F1;H;I		
A717	3		A2;C;H;I		
A718	3		A2,3;C1,2,4;H		
A719	2	6	A;C;H;I	starch found	
A720	3		H;L		
A721	3		A;H;K1		
A722	3		A2;C;K2		occasionally G,I
A723	3		G;I;K2		F2
A724	4		A;C;K2		
A726	5		A1;I	76.4% sodium pentobarbital; starch found	gravimetric, microscope
A727	3		A3;C1,3,4;I;K2		
A728	NO DATA RETURNED				
A729	3		A2,3;C1;G;K1		
A730	1	5	A;G; ;I		
A731	5		A2;H;K1	free base or salt	
A732	3		A;C;H;I		
A733	13		G		
A734	3		A2;C1;G;H		
A735	3		A;C;I		
A736	NO DATA RETURNED				
A737	4		A2,3;B;C2;H		
A738	5		A2;C1,2,3		
A739	2	3	H		I,K1
A740	3		A2;C1,2,3;K1	no opium, cocaine, amphet.; hallucgn.	A5,A6,A8,A9
A741	NO DATA RETURNED				
A742	3		A;H;I;K1		
A743	3	5	A;E;G;H;I;K1		
A744	3		A2;C1,2;H;K1		
A745	3		A2;5,9;C1,2,4		
A746	NO DATA RETURNED				
A747	5		H;I;K1	65% ± 3% free acid	A,H, polarizing microscope
A748	3		C;H;K1	53.7% pentobarbital; starch found	H, microscope
A749	7		A2,3;C1,3;G;I		
A750	3		A2;C1,2,4		
A752	3		A2,5;C1;H;K1		
A753	3		A2,4,5,8,9;G;H;K2		
A754	3		A2;C1,3,4;G;H;K1	starch found	G, microscope
A755	3		A2,3;C1,4		
A756	3		A;C;H;I;K1		
A757	3		C1;K1		
A758	2	5	A2,4,5,6,7,8,9;F2;H;I;K1		
A759	3		G;H;K1		
A760	3		A5,6,9,11,14,15,16;C1,2;H;I		
A761	DOES NOT DO THESE TESTS				
A762	5		A3;K1		
A763	3		A2,5,6,9,11,16;C1;I	76.3% drug concentration	H, NH_4OH in water
A764	3		A2,5;C1		
A765	3	5	A2,5,6,9,11,14;C1,2,3;H;K1	78% sodium pentobarb.; no other drug present	H,K1
A766	5		A2;H;I;K1		
A767	3		A2;C1,6		
A768	3		A2;C1,2,3,4		
A769	3		A2;G;H;I		

TABLE 6
CONTINUED

LAB CODE	1a CONTROLLED SUBSTANCE FOUND (see table 1) I	II	1b INSTRUMENTS AND METHODS (see table 4)	2a ADDITIONAL INFORMATION REPORTED	2b ADDITIONAL INSTRUMENTS AND METHODS
A770	3		A2;C6;H;I		A5
A772	3		A2;C1,4		
A773	NO DATA RETURNED				
A774	NO DATA RETURNED				
A775	3		A3,5;F1;G;H		
A777	2	3	H;I		
A778	3		A2;H;K1		
A779	3		A;C;F1;G;H;I		
A780	3		A;F2;G;H;I		
A781	3		A2;C1,2;G;H		
A782	NO DATA RETURNED				
A783	3		A;C2	starch found	starch iodine test
A784	3		A2;H;J;K1		
A785	5		A2;C;I	78.1% sodium pentobarb.; starch found	H, microscope
A786	1(3)		A;C;I		
A787	1(8)		A2,5;G;I;K1		
A788	3		G;I		
A789	3		A2,3;C1;G;H	45.9% using H	H
A790	3		H;K1		
A791	3		A2;F;G;H;I;M		
A792	NO DATA RETURNED				
A793	5		A2;C1;G		
A794	3		A1;C1,2,3,4		
A795	NO DATA RETURNED				
A796	10		A2,3;I		
A797	3		C2,4;H	no other drugs found	
A798	3		H;K1		
A799	3		A;C;G		
A802	NO DATA RETURNED				
A805	3		A;G;H;K1		
A806	3		A2;H;K1		
A807	3		A2,5,8,9,10;C1,2,3;I		
A809	2	11	A15;H		I
A810	3		A;C;K1		
A811	7		A;I		
A812	3		A2;C1,3;H	starch found	
A813	3		A;H;I;K1	67% ± 2% drug concentration; starch found	microscope, C,H
A814	2(6)		A3,5,6,11;C1,2,3;G;I;K1		
A815	2	3	A2;H		C6
A816	1		G;K1		
A817	NO DATA RETURNED				
A818	3		A3;C1,2,7;K1	no amphet., opiates, hallucinogens	
A820	3		A2;C1,4		H,K1
A821	3		A2,5;H;I;K1		
A822	3		A1;G;I		
A823	3		A;G;I		
A824	3		A2;G;H;K1;M	67% pentobarbital by weight	H
A825	1		A2;H		
A826	NO DATA RETURNED				
A827	3		G;H;I;K1		
A828	NO DATA RETURNED				
A829	11		A2;C1;G;I		
A830	3		A2;C6;H		
A831	4		I		
A832	3		A2,3;C1,2		K1
A833	3		C1;F1,2;H;I	starch found	polarizing microscope
A834	1		A2,3;C;G;H		
A835	3		A;C;H		
A836	1		D		
A837	3		C3,5;F1;H;K3;M	97% pentobarbital	H
A838	3		A;C;H		
A839	3		A;H;I;K1		
A841	3		A;H;I;K1		
A842	5		A;H;I;K1	75% sodium pentobarb.; no other drugs present	H
A843	14 (NO DRUG FOUND)		G;H		

( )- indicates that additional findings were reported.

TABLE 6
CONTINUED

LAB CODE	1a CONTROLLED SUBSTANCE FOUND (see table 1) I	II	1b INSTRUMENTS AND METHODS (see table 4)	2a ADDITIONAL INFORMATION REPORTED	2b ADDITIONAL INSTRUMENTS AND METHODS
A844	3		A;H;I	56.4% pentobarbital by weight	
A845	5		A2;H;I		
A846	NO DATA RETURNED				
A847	3		A2;H;I		
A848	3		A2;H;I		
A849	2		A2,3,5;H		
A850	3		A2,3;G		
A851	NO DATA RETURNED				
A852	3		A;H;I		
A853	2		A;G;H		
A854	3		A;L	starch found	microscope
A855	1	3	A2,3;G;I		KI
A856	3		A2;G;I;KI		
A858	1		A;G		
A859	5		A3;B;C		
A860	3		A;C;Fl;H;I	starch found	H,KI for quant.
A861	3		C;KI		
A862	NO DATA RETURNED				
A863	2		A5,15;H;I		
A864	NO DATA RETURNED				
A865	NO DATA RETURNED				
A866	2(3)		A3,15;H;KI		
A867	NO DATA RETURNED				
A868	3		A;I		
A869	3		A2;C;H		
A870	5		A;G;I;K2		H,C
A871	1(3)		G;H;I;K2		
A872	2(3)		A2;H;I	62.5% pentobarbital	
A873	3		A3;C	starch found	microscope
A874	3		A;C;I		
A875	3		A2,12;C1,2;G;H		
A876	3		A;H;I;KI		
A877	3		A13;H;KI		
A878	NO DATA RETURNED				
A879	NO DATA RETURNED				
A880	7		A;I;KI	starch found	C
A884	3		A2,3,4;C4		
A885	3		A2;H;I		
A886	3		A;H;I		
A887	NO DATA RETURNED				
A888	3		A;H;I		
A889	3		A2;C2,4;H		
A891	5		H;I		
A892	NO DATA RETURNED				
A894	5		A;C;E;F;H;KI		
A895	3		A;G;H;KI		
A897	5		A2;C1;I		
A898	3		A2;C6;I;K3		
A899	3		A2;C;H;KI		
A900	NO DATA RETURNED				
A902	3		A;C;H		
A903	9		A;H;KI		
A904	5		A2,3;H;I;KI		
A907	3		A;C;H;I;KI	11% drug concentration; starch found	KI quant.
A908	5		A;H;I;KI	starch found	HgI_2 + HCl
A912	NO DATA RETURNED				
A913	3		A2,5;C1,2,3,4;F2;G;H;J	starch found	micro-HOAC
A914	3		A2,3,5,9,10,14;C1,2,4		
A915	3		A;C;H;I	starch found	polarizing microscope
A917	3	5	A2;C1,2,3,4;G;H;I;K	80% sodium pentobarbital	H,K3
A918	2		A15;H		
A920	3		A;I		
A921	2		A5,6,9,11,15;H		
A923	5		A2;L		
A924	3		G;H;K2		
A925	3	5	H;I	82.2% sodium pentobarbital	L
A926	12(3)		A2,3,5,6,11;C1,4		
A927	7		C;I	78% as Na Salt	H
A931	2		A2;C6;H		
A932	3		A2;C1;I		

( )- indicates that additional findings were reported.

TABLE 6
CONTINUED

LAB CODE	1a CONTROLLED SUBSTANCE FOUND (see table 1) I	II	1b INSTRUMENTS AND METHODS (see table 4)	2a ADDITIONAL INFORMATION REPORTED	2b ADDITIONAL INSTRUMENTS AND METHODS
A935	3		A2;G;I;K1		
A937	3		A2;C;H	80%+ drug concentration	H
A938	3		A2;C1,6;F1;I	starch found	iodine test and microscope
A942	6		G;H;I;K2	88% by K1, 72% by H	K1,H
A944	NO DATA RETURNED				
A946	5		A3,13;F1;H;I;J;K1	no other substances found	C,G,M
A948	3		A2;C1;H;J		
A950	3		A2,5,9;C;I	weak positive for sugar	
A951	3		I;L		
A953	3		H;K1		
A958	2	3	H;I;K1		I,K1
A960	3		A2,3;H;K2		
A961	3		C1;I	58% drug concentration	
A964	3		A3;I;K1		
A966	2(3)		A2;C1,3,4		
A969	3		A2;C1,2;H;I		
A970	3		A;C;H;I	69% pentobarbital as free acid	H
A972	NO DATA RETURNED				
A973	3		A3,5,9;C1,2,3;G		
A974	5		A2;G;I;K1	35.7% pentobarb. sodium; starch found	K1, polarizing microscope
A975	3		A;H;I;K1;L	74% pentobarbital; starch found	H
A978	3		A2;H;I;K1;L		
A979	5		A;C;H;K1	starch found	starch test, microscope
A980	1	3	A1,3;C1		F1,G
A983	5		A;I;K1	75% pentobarb. by weight; starch found	I,K1
A985	3		A;C;H		K1
A986	2		A5,9,15;H;I		
A987	8		A2;G;H;I	no active contaminants	
A988	NO DATA RETURNED				
A989	3		C;H;I;K1	55% pentobarbital; 45% corn starch	H,K1, microscope
A992	2		A2;C1,2		
A994	3		A2;C;H;I;K1		
A995	4		A2;C1,2;G		
A998	3		A2;C,1,2,3		
A999	3		A;H;I		

( )- indicates that additional findings were reported.

March 7, 1975

END