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X-Ray Powder Diffraction Data for Some Drugs, Excipients, and Adulterants in Illicit Samples

The development of new compounds with the potential for drug abuse necessitates a continuous accumulation of analytical data in the forensic laboratory. Also, the identification of excipients and adulterants in drug samples provides a data base that can be used for intelligence purposes. Correlation of cases can provide investigative leads as well as being supporting evidence in conspiracy cases.

The purpose of the present paper is to present X-ray powder diffraction data not available in the literature. Included in the paper are data on compounds where more complete and accurate tabulations than those already published have been obtained.

The X-ray diffraction powder method has proven to be very effective and specific in the identification of crystalline substances. For example, different hydrates of the same compound, as well as polymorphic forms, have completely different X-ray diffraction patterns. Compounds in mixtures can be identified without separation. The Special Testing and Research Laboratory has been able to identify three components of a mixture from a single strip chart recording, which was a composite of the diffraction patterns of the substances involved.

Two modes of operation are utilized in the powder method, one using a camera and recording the diffracted X-rays on film, the other involving a diffractometer, detector, electronic panel, and chart recorder. The latter method is the superior one in that, among other advantages, the resulting pattern is spread out on a chart recording with much better resolution of lines. Triplets and doublets on the chart would appear as single (broad) lines on a film.

The Powder Diffraction File, published by the Joint Committee on Powder Diffraction Standards,[2] contains inorganic and organic sets of cards containing X-ray diffraction data on over 25,000 crystalline substances, accompanied by numerical and alphabetical index books. Annually, a new set of data (separated into organic and inorganic categories) is published. The new sets contain data on compounds not previously entered in the file, or replace cards in previous sets with more complete and accurate data. It is essential that complete sets of cards be available to the X-ray diffraction laboratory to realize the potential of the equipment.

Much of the data in the Powder Diffraction File, especially older data, has been taken from film. Many of the original cards have been deleted and replaced by more accurate tabulations. Of those earlier cards which remain, the data are often incomplete, especially for organic materials where the lines are confined to lower angles and are in

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close proximity. Resolution on film is improved somewhat by using CrK_α radiation, but still does not approach that obtained with the diffractometer. Therefore, data are included in the present paper for compounds already compiled in the Powder Diffraction File for which discrepancies have been found.

Experimental

Apparatus

A Philips wide-angle diffractometer, equipped with scintillation counter and using nickel-filtered copper radiation, was employed. Interplanar spacing d was based on $(CuK_\alpha) = 1.5405$ Å.

Sample Preparation

Reagent grade chemicals were used whenever possible, crushed to very fine powder, and packed in regular sample holders.

Operation

The goniometer scan (1 deg 2θ/min) was from 2 to 60 deg 2θ, higher for inorganic substances of small unit cell.

The X-ray tube was maintained at 30 kV and 10 mA. To obtain d-spacings from the strip chart recording, a scale [*1*] previously made from Plexiglas® was used. The scale shows the angles along the lower edge (1 in. = 1 deg 2θ), and the corresponding d-spacings above. The accuracy of the scale is equal to that of the conversion tables [*2*], and use of the scale has the advantage of being much more rapid with less chance of error. Relative intensities were calculated, giving the most intense peak for each pattern a value of 100.

Results

Table 1 presents the three most intense lines of each pattern, arranged in descending order according to the d-spacing of the most intense line. The data are arranged in groups corresponding to the Hanawalt search method [*3*] to facilitate selection of the most probable pattern. Relative intensities are tabulated along with the d-spacings. The final column in the table gives the number appointed to the compound of interest as it appears in Table 2.

In Table 2 are listed the complete X-ray diffraction data for each compound, arranged in alphabetical order and numbered accordingly.

Discussion

l-*Ascorbic Acid*

Ascorbic acid is often found in LSD preparations, sometimes with narcotics and cocaine. Occasionally, exhibits have been received which were ascorbic acid tablets (commercially available), the surfaces of which were treated with solutions of LSD.

Benzocaine (Ethylaminobenzoate)

Benzocaine is frequently found as an adulterant in combination with cocaine hydro-

TABLE 1—*Numerical (Hanawalt) index.*

d, Å			*I/I*$_1$			Pattern No.
			19.9-18.0			
18.4	4.84	4.35	100	31	18	9
18.3	4.81	4.31	>100	100	57	12
18.0	18.5	5.53	100	86	51	59
			17.9-16.0			
17.1	5.65	4.17	>100	100	38	13
17.1	3.48	6.10	100	35	28	57
16.9	4.23	3.87	100	56	45	11
16.6	3.37	3.49	>100	100	38	22
			15.9-14.0			
15.8	4.39	5.23	>100	100	55	10
15.5	4.32	5.40	>100	100	61	23
15.3	3.98	3.89	100	56	56	30
14.6	7.31	7.80	>100	100	67	66
			13.9-12.0			
13.8	3.48	6.96	>100	100	100	72
13.7	4.72	3.94	100	60	59	44
12.5	4.57	3.46	100	30	20	68
			11.9-11.0			
11.5	5.41	3.35	100	60	41	46
			10.9-10.0			
10.6	4.26	8.48	100	76	57	52
10.4	14.1	6.26	100	78	39	69
10.2	3.36	5.07	100	80	35	49
10.1	14.0	5.03	100	43	29	7
10.1	4.34	3.91	>100	100	49	4
10.0	5.16	5.02	100	67	34	63
			9.99-9.50			
9.99	14.1	12.0	>100	100	84	53
9.92	12.0	4.04	100	58	42	19
9.69	4.53	3.16	100	96	94	25
			8.99-8.50			
8.72	5.54	3.77	100	69	66	15
			7.99-7.50			
7.98	5.25	9.24	>100	100	86	17
7.82	3.69	5.26	100	68	66	32
7.52	3.38	3.30	>100	100	90	18
			7.49-7.00			
7.06	4.51	8.82	100	97	72	38
			6.99-6.50			
6.90	5.44	4.42	100	46	41	41
6.74	4.24	3.47	100	99	72	39
6.74	3.62	3.36	>100	100	99	48
6.50	7.25	4.47	100	85	82	55
			6.49-6.00			
6.15	8.68	4.08	100	58	41	54
6.13	4.56	7.40	100	43	43	62

TABLE 1—*Continued.*

d, Å			*I*/*I*$_1$			Pattern No.
			5.99-5.75			
5.88	11.7	4.68	100	53	29	47
5.87	5.39	5.74	100	77	72	35
5.80	10.8	9.44	100	49	44	40
			5.74-5.50			
5.73	12.6	4.23	100	60	29	61
5.73	4.88	3.81	100	96	86	1
			5.49-5.25			
5.34	7.80	4.36	100	83	77	29
5.34	6.85	5.77	100	91	87	33
			5.24-5.00			
5.14	5.11	3.79	100	97	92	31
5.13	9.82	4.79	100	47	20	64
5.07	3.73	6.32	100	100	90	2
			4.89-4.80			
4.85	3.26	3.91	100	58	42	8
			4.79-4.70			
4.78	4.24	3.87	100	74	43	5
4.77	4.08	3.68	100	74	53	70
4.76	5.57	3.40	100	88	54	45
4.75	4.53	5.07	100	90	67	50
			4.69-4.60			
4.61	4.39	6.82	100	64	29	21
			4.49-4.40			
4.46	4.54	4.64	100	50	39	37
			4.39-4.30			
4.37	2.92	8.70	100	100	45	71
4.33	6.33	4.18	100	93	80	27
4.31	4.74	2.49	>100	100	44	24
			4.29-4.20			
4.26	4.67	8.50	100	75	29	36
4.22	3.87	4.24	100	100	91	28
			4.09-4.00			
4.08	23.7	3.35	100	48	22	20
4.08	6.75	7.37	100	57	53	34
4.07	5.43	4.16	100	79	49	58
4.02	3.27	3.86	100	94	88	51
			3.99-3.90			
3.91	5.37	3.14	100	68	23	6
			3.89-3.80			
3.81	6.10	6.90	100	84	68	56
3.80	4.74	6.06	100	88	61	43
			3.79-3.70			
3.72	4.42	5.32	100	89	61	16
			3.69-3.60			
3.60	4.73	4.55	100	84	71	73

TABLE 1—*Continued.*

d, Å			*I*/*I*$_1$			Pattern No.
			3.59-3.50			
3.56	5.27	4.67	100	81	78	26
3.54	4.63	5.53	100	74	66	65
3.53	7.24	8.24	100	54	51	3
3.50	5.45	3.21	100	90	62	67
3.50	3.57	4.09	100	49	41	60
			3.19-3.15			
3.18	8.52	4.48	100	91	72	14
			3.14-3.10			
3.11	9.29	4.67	>100	>100	100	42

d = interplaning spacings of the three most intense lines
I/*I*$_1$ = relative intensities

chloride. Occasional narcotic samples also contained benzocaine. The X-ray diffraction data obtained closely follow those of Owen et al [*4*].

Caffeine

Caffeine has been found in combination with heroin and amphetamines. It is also found in many tablet samples reputed to be amphetamine or methamphetamine. Crystals of caffeine are very fine needles as seen microscopically. When packed for X-ray diffraction analysis the crystals exhibit preferred orientation, producing very intense lines at d = 7.42 and 7.52 Å (a doublet) and d = 3.38 Å, the remaining lines being of relatively weak intensity. Therefore, low concentrations of caffeine are indicated when lines appear having the d-spacings mentioned above, lines which would not be seen if there was little or no preferred orientation.

Calcium Salts

Inorganic and organic calcium salts have been found in a large number of exhibits. Perhaps the most common are calcium phosphate hydrate (brushite, $CaHPO_4 \cdot 2H_2O$), calcium magnesium carbonate [dolomite, $CaMg(CO_3)_2$], and the two forms of calcium carbonate (calcite and aragonite). Calcium sulfate dihydrate (gypsum) and hydroxyapatite [$Ca_5(PO_4)_3(OH)$], have also been encountered. Excellent data for these inorganic substances are found in the Powder Diffraction File. Calcium tartrate hydrate ($C_4H_4CaO_6 \cdot 4H_2O$) has been found in heroin samples and calcium lactate hydrate ($C_6H_{10}CaO_6 \cdot 5H_2O$) has been used as a filler in LSD tablets.

Procaine Hydrochloride

Cocaine and heroin samples are often adulterated with appreciable amounts of procaine hydrochloride. There are some differences between the tabulated data taken from film and those in the present paper.

Quinine and Quinidine Salts

Quinine salts which have been used as adulterants in heroin preparations are quinine hydrochloride ($C_{20}H_{24}N_2O_2 \cdot HCl \cdot 2H_2O$) and quinine sulfate ($C_{40}H_{48}N_4O_4 \cdot SO_4 \cdot 2H_2O$).

TABLE 2—*Complete powder diffraction data. (Two numbers bracketed together indicate a doublet; three indicate a triplet; B indicates broad lines).*

d, Å	*I*/*I*$_1$	*d*, Å	*I*/*I*$_1$	*d*, Å	*I*/*I*$_1$	*d*, Å	*I*/*I*$_1$
(1) Acetaminophen		5.07	100	{3.45	29	5.19	17
7.37	32	4.89	33	{3.42	31	5.09	11
6.46	23	4.76	18	{3.355	27	4.88	5
5.73	100	4.44	76	{3.335	32	4.68	7
5.65	16	4.18	28	3.270	25	4.54	23
4.88	96	3.88	62	3.185	21	4.48	14
4.71	12	3.73	100	3.090	12	4.41	26
4.36	52	3.41	37	3.055	11	4.34	100
4.28	6	3.32	8	2.920B	5	4.20	5
3.81	86	3.25	9	2.774	14	4.11	6
3.67	80	3.12	15	2.726	7	4.02	4
3.37	66	2.816	24	2.685	3	3.91	49
3.29	11	2.515	9	2.550B	6	3.87	24
3.22	3	2.465	8	2.445B	3	3.80	26
3.08B	8			2.370B	2	3.73	7
2.863	5	(3) Aminophylline Dihydrate		2.300	4	3.67	11
2.755	18	11.5	13	2.223B	2	3.58	12
2.484	6	10.7	3	2.055B	2	3.56	17
2.440	9	8.24	51	2.003B	3	3.47	5
2.404	4	7.43	23	1.963	3	3.43	23
2.340	6	7.24	54	1.896	4	3.35	8
2.290B	3	6.69	4			3.29	5
2.253	6	6.07	43	(4) Aminopyrine		3.25	8
2.183	6	5.76	6	11.2	3	3.08	15
2.135	3	5.66	7	10.1	261	2.998	7
2.085	3	5.52	4	8.00	7	2.959	5
2.010	2	5.26	13	7.13	24	2.898	4
1.980B	2	5.12	7	7.07	18	2.760	4
1.895B	4	4.62	3	6.88	42	2.736	2
1.800B	3	4.51	7	6.71	15	2.664	19
1.760B	2	4.09	8	6.63	23	2.560	2
		3.88	24	6.13	4	2.477	1
(2) 1-Acetyl-2-Phenylhydrazine		3.73	8	5.60	13	2.440	2
9.5	34	{3.66	11	5.55	20	2.319	2
6.32	90	{3.63	11	5.42	17	2.240	2
5.47	62	3.53	100	5.37	11	2.175	2

TABLE 2—*Continued.*

d, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1
2.130	2	2.376	5	(9) *d*-Amphetamine Phosphate		2.827	9
2.061	2	2.345	3	18.4	100	2.787	6
2.035	2	2.318	8	16.6	7	2.584	6
2.011	2	2.233	9	9.19	7	2.131	6
		2.210	5	6.11	17	2.068	6
		2.182	3	4.98	13		
(5) Amitriptyline HCl		2.142	3	4.84	31	(12) *dl*-Amphetamine Phosphate	
7.71	18	2.110	1	4.72	5	23.5	4
6.94	11	{2.025	4	4.57	14	18.3	484
6.60	4	{2.007	4	4.47	6	16.5	16
5.94	19	1.960	3	4.35	18	9.15	36
5.67	10	1.837	4	4.07	18	6.11	28
5.55	4	1.771	2	3.86	13	5.08	14
5.43	11	1.745	3	3.66	6	4.98	31
5.32	17	1.682	2	3.50	5	4.81	100
5.02	3	1.564	2	3.46	6	4.57	51
4.78	100	1.531	2	3.25	4	4.31	57
4.70	14			3.21	8	4.07	56
4.62	21	(7) Amobarbital Sodium		3.07	4	3.89	39
4.38	6	14.02	43	2.993	3	3.76	28
4.24	74	11.68	27	2.910	2	3.65	28
4.17	10	10.06	100	2.825	2	3.58	17
4.07	4	6.21	20	2.541	3	3.53	22
3.87	43	6.04	18	2.287	4	3.44	17
3.79	5	5.78	16	1.920	1	3.20	23
3.67	6	5.70	20			3.14	6
3.65	8	5.63	16	(10) *d*-Amphetamine Sulfate		3.07	10
3.57	3	5.54	12	15.8	2075	3.01	9
3.45	18	5.43	14	8.31	8	2.959	9
3.34	20	5.32	14	5.50	23	2.890	11
3.26	9	5.03	29	5.23	55	2.830	8
3.165	6	4.67	27	5.11	37	2.580	8
3.080	2	4.44	16	4.94	22	2.535	8
3.030	2	3.87	12	4.77	29	2.495	6
2.967	5	3.37	6	4.58	19	2.440	6

2.890	4
2.858	6
2.820	4
2.755	4
2.730	5
2.650	6
2.622	2
2.544	2
2.500	2
2.350	2
2.308	3
2.195	1
2.088	2
2.040B	<1
1.820	<1

(6) Ammonium Citrate Dibasic

7.49	8
7.38	4
5.68	21
5.56	4
5.37	68
5.05	8
4.35	4
4.28	2
4.06	7
3.91	100
3.73	2
3.70	11
3.63	13
3.14	23
3.05B	10
2.920B	6
2.855B	5
2.740B	3
2.665	13
2.596	9
2.547	4
2.463	6

(8) *d*-Amphetamine HCl

15.6	5
6.53	41
6.18	15
5.96	27
5.37	7
4.85	100
4.74	13
4.65	9
4.56	27
4.43	27
4.18	40
4.04	7
3.97	9
3.91	42
3.83	16
3.69	31
3.54	20
3.42	31
3.30	29
3.26	58
3.16	19
3.01	13
2.920	5
2.853	9
2.820	8
2.761	11
2.732	11
2.631	5
2.571	7
2.538	14
2.501	7
2.390	16
2.273	4
2.239	5
2.210	5
2.175	7
1.977	5
1.948	6
4.39	100
4.23	44
4.05	13
3.93	33
3.88	40
3.69	12
3.57	21
3.39	14
3.14	41
3.03	12
2.949	11
2.882	8
2.665	5
2.610	6
2.480	5
2.318	6
2.270	8

(11) *dl*-Amphetamine HCl

16.9	100
7.30	5
6.33	6
5.52	27
5.28	27
5.10	17
4.84	10
4.72	15
4.55	9
4.38	19
4.23	56
3.87	45
3.74	11
3.59	15
3.50	19
3.39	17
3.24	14
3.17	20
2.951	16
2.865	8
2.400	5
2.282	21
2.229	4
1.937	4
1.910	5
1.840	4

(13) *dl*-Amphetamine Sulfate

17.1	539
8.49	32
5.65	100
5.20	33
4.48	19
4.17	38
3.91	10
3.80	7
3.57	13
3.38	33
2.850	6
2.725	5
2.332	7
2.112	4
2.070	4

(14) *l*-Ascorbic Acid

8.52	91
6.32	11
5.66	16
5.55	9
5.10	44
4.48	72
4.23	14
4.18	6
3.79	10
3.53	39
3.33	16
3.285	14
3.180	100
3.030	6

TABLE 2—*Continued.*

d, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1
2.980	59	2.582	5	3.76	43	5.43	4
2.870	4	2.552	2	3.70	52	5.33	5
2.810	4	2.518	4	3.42	39	5.23	9
2.760B	3	2.500	3	3.38	100	4.86	5
2.695	6	2.478	2	3.30	90	4.75	17
2.635	3	2.400B	3	3.15	33	4.61	100
2.578	17	2.340BB	3	3.07	9	4.39	64
2.528	15	2.302	3	3.03	14	4.25	18
2.442	2	2.180B	3	2.753	8	4.14	20
2.400	7	2.110	3	2.510	10	4.06	22
2.274	3	1.898	2	2.466	16	3.99	4
2.260	3	1.815B	1	2.356	5	3.69	20
2.239	8	1.751B	2	2.179	6	3.60	22
2.175	6			2.046	10	3.565	23
2.140	8	(16) Benzocaine (Ethylamino-benzoate)				3.460	10
2.112	8			(19) Calcium Lactate Penta-Hydrate		3.380	24
2.092	3	10.4	16			3.330	2
2.056	4	7.65	22	12.0	58	3.185	6
2.020	5	5.32	60	9.92	100	3.140	4
2.008	9	5.22	61	8.12	21	2.978	7
1.980	6	4.74	28	5.96	25	2.918	3
1.834	2	4.42	89	5.16B	9	2.882	2
1.883	2	4.22	38	4.74	11	2.778	11
1.855	1	4.13	34	4.54	12	2.658	6
1.808	2	3.82	28	4.38	21	2.636	4
1.778	4	3.72	100	4.04	42	2.578	7
1.691	4	3.48	10	3.96	20	2.390	4
1.639	1	3.29	56	3.83	12	2.352	2
1.615	1	2.952	11	3.59	20	2.164	3
1.588	3	2.600	10	3.51	11	2.143	3
1.562	4	2.515	4	3.28	25	2.028	4
1.536B	1	2.410	4	3.08	11	1.935B	2
1.502B	1	2.280B	6	2.935	11	1.890B	2
1.408B	2	1.900	3	2.855B	9	1.840B	1

(15) Benactyzine HCl

15.9	36
8.72	100
7.79	14
7.58	20
7.04	10
6.68	33
6.20B	3
5.77	48
5.54	69
5.29	25
5.04	2
4.87	2
4.45	6
4.42	6
4.32	16
4.27	9
4.18	1
4.08	3
3.96	5
3.89	3
3.84	5
3.77	66
3.66	56
3.60	6
3.57	10
3.51	21
3.43	10
3.34	22
3.315	19
3.205	8
3.100	13
3.000	16
2.885	3
2.848	4
2.743	13
2.662	5
2.640	3
2.608	6
1.860	3
1.748	4

(17) Butabarbital Sodium

15.82	14
14.11	34
12.48	24
11.33	41
10.83	28
9.97	52
9.60	55
9.24	86
8.87	38
7.98	290
6.12	41
5.98	45
5.51	38
5.25	100
5.07	41
4.98	28
4.77	34
4.49	21
4.37	24
3.94	28
3.30	17
3.21	17
3.16	28

(18) Caffeine

10.67	10
8.28	9
7.52	654
7.42	612
7.12	37
6.15	14
5.63	7
4.52	7
4.33	23
4.27	6
2.750	6
2.690	5
2.620	6
2.575	10
2.475	7
2.440	14
2.270	9
2.185	15
2.003	10
1.884	7
1.853	6

(20) Chlorphenesin Carbamate

23.7	48
5.89	9
4.78	6
4.66	12
4.43	4
4.08	100
3.35	22
3.09	7
3.04	5
2.885	2
2.730	5
2.600	3
2.468	4
2.360	3
2.315	1
2.273	2
2.101	4
1.930	1
1.813	2

(21) Chlorpheniramine Maleate

9.48	2
8.80	2
7.23	12
6.82	29
5.77	2

(22) Clorazepate, Dipotassium Salt

16.6	300
13.4	5
8.44	30
6.06	8
5.63	45
5.27	4
4.98	15
4.76	12
4.32	5
4.22	36
4.17	8
4.08	4
4.01	3
3.75	3
3.64	5
3.49	38
3.37	100
3.28	11
3.25	22
3.20	4
3.08	5
3.05	6
2.975	8
2.925	6
2.875	6
2.813	28
2.768	7
2.658	6
2.620	6
2.570	3
2.458	9
2.412	23
2.258	4
2.170	3
2.090	7
1.876	11

TABLE 2—*Continued.*

d, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1
(23) Cyclamate Sodium		(25) Dextrose Monohydrate		(27) Dimethyltryptamine		(29) Diphenylhydantoin	
15.5	2864	9.69	100	9.58	33	10.30	60
6.18	15	8.58	3	9.06	16	7.80	83
5.4	61	6.99	49	8.05	19	6.85	32
5.18	35	6.11	16	7.44B	36	5.34	100
4.98	18	5.21	4	7.02	26	5.13	43
4.67	61	4.84	63	6.57	53	4.88	48
4.47	47	4.53	96	6.33	93	4.60	13
4.32	100	4.41	47	6.14	53	4.36	77
4.05	8	4.31	27	5.78	20	3.97	63
3.91	53	4.11	10	5.58	63	3.89	16
3.89	60	3.90	35	5.22	40	3.83	2
3.64	7	3.80	5	5.12	28	3.41	48
3.45	15	3.59	3	4.78	40	3.31	11
3.29	48	3.51	21	4.72	54	3.21	20
3.11	10	3.37	10	4.56	74	3.05	3
3.02	19	3.24	13	4.33	100	2.996	8
2.925	41	3.22	18	4.18	80	2.897	4
2.875	9	3.16	94	4.05	35	2.834	5
2.795	50	3.04	14	3.95	16	2.682	11
2.69	10	2.891	15	3.84	16	2.570	4
2.59	36	2.873	14	3.76	16	2.538	3
2.315	15	2.773	06	3.69	23	2.412	5
2.045	14	2.717	5	3.55	29	2.263	2
		2.685	9	3.41B	16	2.177	3
(24) Dextrose, Anhydrous		2.656	6	3.315	14	2.097	2
8.56	13	2.547	9	3.260	13	2.059	5
7.44	16	2.521	12	3.145B	13	1.997	3
6.06	39	2.435	20	2.990B	8		
5.21	30	2.410	8	2.890B	6	(30) Diphenylhydantoin Sodium	
4.74	100	2.242	20	2.735	10	15.3	100
4.50	18	2.178	19	2.625	5	13.2	47
4.31	532	2.144	8	2.584	4	9.17	13
3.85	14	2.051	10				

3.60	8
3.50	37
3.33	10
3.24	6
3.14	43
3.03	3
2.982	6
2.913	10
2.862	9
2.657	4
2.587	13
2.558	11
2.493	44
2.459	32
2.423	9
2.393	5
2.366	3
2.299	9
2.275	11
2.259	14
2.221	14
2.197	3
2.169	4
2.132	3
2.086	3
2.056	10
2.030	5
2.004	4
1.959	4
1.918	5
1.899	3
1.872	3
1.827	3
1.791	4
1.687	5
1.668	5
1.630	4
1.597	5
2.004	9
1.937	4
1.915	3
1.872	5
1.857	10
1.833	3
1.745	4
1.723	6

(26) Diethyltryptamine

8.75	69
7.80	47
7.18	19
6.19	27
5.54	69
5.27	81
4.84	31
4.67	78
4.37	47
4.25	43
4.00	58
3.90	15
3.81	7
3.56	100
3.415	36
3.350	6
3.240	22
3.185	6
3.085	3
2.945	15
2.898	9
2.754	6
2.718	5
2.652	4
2.328	4
2.265	8
2.213	2

(28) 2,4-Dinitrophenol

11.6	10
5.79	59
5.43	14
5.08	54
4.80	29
4.74	41
4.24	91
4.22	100
3.87	100
3.70	6
3.54	71
3.47	20
3.28	62
3.11	7
3.02	29
2.910	9
2.850	4
2.796	8
2.770	28
2.620	17
2.579	11
2.541	23
2.536	24
2.383	5
2.340	14
2.321	17
2.286	9
2.211	6
2.179	11
2.121	6
1.951	5
1.935	7
1.920	7
1.871	6
1.844	7
1.659	20
1.635	5
8.68	19
8.23	3
7.70	19
7.36	17
6.83	16
5.83	50
4.38B	15
3.98	56
3.89	56
3.85	11
3.69	15
3.60	12
3.53	15
3.40	17
3.09B	6

(31) Glutethimide

11.3	4
10.2	60
9.3	7
7.18	46
6.21	61
6.03	59
5.14	100
5.11	97
4.85	11
4.68	23
4.53	17
4.17	9
3.91	14
3.86	21
3.79	92
3.71	11
3.53	11
3.26	11
3.20	11
3.02	5
2.830	11

TABLE 2—*Continued.*

d, Å	*I*/*I*$_1$	*d*, Å	*I*/*I*$_1$	*d*, Å	*I*/*I*$_1$	*d*, Å	*I*/*I*$_1$
2.700	3	5.07	45	3.44	2	1.984	2
2.510	7	4.70	20	3.33	3	1.928	2
		4.5	31	3.245	4	1.899	3
(32) JB-318 (*N*-Ethyl-3-Pip-		4.33	31	3.185	8	1.845	2
eridyl Benzilate)		4.13	43	2.960	1	1.816	3
11.5	39	4.08	100	2.890	2	1.760	2
9.40	15	3.69	29	2.825	13		
9.08	19	3.53	16	2.770	1	(38) Lidocaine	
8.50	20	3.37	43	2.705	5	11.2	13
7.82	100	3.2	18	2.650	2	9.75	15
6.68	26	3.08	16	2.597	1	9.06	31
6.40	20	3.02	20	2.513	2	8.82	72
5.74	14	2.82	18	2.475	4	7.38	22
5.33	40	2.75	20	2.460	6	7.21	48
5.26	66			2.422	3	7.06	100
5.13	57	(35) JB-344 [1-Methyl-3-Pip-		2.402	5	6.53	26
4.99	57	eridyl *a*-(2-Thienyl) Mandelate HCl]		2.362	2	6.27	19
4.26	45	10.5	27	2.323	6	5.98	33
4.08	12	8.23	25	2.282	2	5.63B	16
3.91	66	7.38	45	2.260	3	5.35	19
3.69	68	6.95	23	2.230	3	4.73	53
3.59	43	6.47	39	2.182	4	4.51	97
3.52	51	6.12	43	2.168	3	4.24	33
3.39	22	5.87	100	2.146	2	4.07	43
3.08	9	5.72	72	2.110	3	3.92	36
3.01	9	5.39	77	2.061	1	3.71	16
2.873	22	5.14	42	2.022	2	3.56	24
2.835B	9	4.64	52	2.000	6	3.41	19
2.782	8	4.48	45	1.958	2	3.27	12
2.732	14	4.34	22	1.947	6	3.15	18
2.468	7	4.28	12	1.934	4	3.10	10
2.360B	8	4.24	15	1.897	1	3.02	8
1.995B	3	4.17	31	1.875	2	3.02	8
1.940B	5	4.11	58	1.810	3	2.945	5
1.845	3	3.98	22	1.743	3	2.780	5

(33) JB-331 [1-Ethyl-3-Piperidyl α-(2-Thienyl) Mandelate HCl]

8.62	36
6.85	91
6.66	34
5.85	40
5.77	87
5.34	100
4.96	30
4.87	19
4.74	8
4.47	30
4.30	52
4.14	42
4.07	21
3.96	53
3.83	11
3.56	34
3.53	47
3.44	70
3.17	25
3.13	34
3.09	36
3.05	13
2.960	13
2.880	8
2.655	8
2.585	6
2.478	8

(34) JB-336 (*N*-Methyl-3-Piperidyl Benzilate HCl)

8.97	47
8.1	37
7.37	53
6.75	57
6.04	35
5.4	33
5.23	14
3.65	13
3.61	15
3.54	13
3.37	67
3.31	47
3.28	31
3.13	13
3.08	29
2.985	12
2.865	9
2.708	52
2.602	11
2.588	9
2.568	14
2.420	7
2.402	9
2.358	5
2.315	7
2.248	16

(36) β-Lactose

8.50	29
6.72	3
5.70	5
5.44	2
5.04	6
4.98	3
4.67	75
4.56	7
4.47	20
4.31	38
4.26	100
4.13	3
3.99	5
3.76	10
3.72	11
3.62	20
3.57	6
3.50	23

(37) Lactose Monohydrate

7.10	24
5.41	19
5.19	4
4.64	39
4.54	50
4.46	100
4.28	16
4.20	28
3.91	9
3.75	13
3.53	3
3.49	8
3.41	3
3.25	5
3.17	4
3.13	3
3.08	2
2.882	4
2.846	2
2.744	1
2.700	3
2.597	4
2.569	6
2.543	3
2.520	2
2.479	5
2.435	7
2.395	11
2.354	8
2.333	5
2.285	5
2.285	4
2.260	4
2.220	3
2.162	1
2.135	2
2.104	2
2.050	2
2.670	4
2.570	2
2.418	5
2.375	5
2.290	2
2.235	2

(39) Lidocaine Sulfate

11.7	9
9.53	56
8.30	31
7.35	25
6.74	100
5.51	58
5.00	23
4.78	16
4.58	18
4.24	99
4.08	52
4.01	69
3.90	19
3.70	47
3.47	72
3.43	23
3.40	22
3.28	12
3.07	31
2.930	10
2.485	5
2.397	5

(40) Lysergic Acid Diethylamide Tartrate

16.9	12
10.8	49
9.44	44
8.43	32
5.80	100
5.71	61

TABLE 2—*Continued.*

d, Å	*I/I*$_1$	*d*, Å	*I/I*$_1$	*d*, Å	*I/I*$_1$	*d*, Å	*I/I*$_1$
5.50	10	2.043	5	1.816	2	2.243	4
5.17	30	2.013	5	1.742	2	2.214	7
5.04	13					2.080	3
4.78	30	(43) D-Mannitol		(45) Methaqualone		2.040	3
4.56	14	8.47	11	8.18	15	1.975	5
4.39	24	7.73	4	7.78	11		
4.12	14	6.74	1	7.38	42	(47) Methenamine Mandelate	
4.03	31	6.06	61	6.99	48	11.7	53
3.87	10	5.29	11	6.12	20	9.47	11
3.79	11	4.74	88	5.57	88	7.70	25
3.69	13	4.51	3	5.36	13	7.10	2
3.60	18	4.34	30	5.10	12	6.95	5
3.38	16	4.21	52	4.76	100	6.53	1
3.29	5	4.08	12	4.57	7	5.88	100
3.21	5	3.80	100	4.44	6	5.48	2
3.14	7	3.60	8	4.13	17	5.13	20
2.900	6	3.44	13	4.07	12	5.04	22
2.810B	4	3.36	4	4.00	12	4.88	19
2.740B	4	3.17	8	3.77	29	4.83	16
		3.15	11	3.67	23	4.68	29
(41) Lysergic Acid Diethyl-amide (Iso)		3.02	43	3.57	10	4.52	23
		2.928	2	3.53	9	4.41	16
11.0	21	2.854	6	3.395	54	4.27	17
6.90	100	2.819	8	3.260	9	3.92	6
5.44	46	2.739	9	3.190	10	3.69	6
5.25	22	2.670	18	3.125	25	3.55	1
5.02	15	2.614	7	3.055	13	3.43	2
4.67	3	2.568	4	2.950	5	3.32	2
4.42	41	2.493	9	2.895	4	3.22	1
4.22	12	2.327	12	2.865	3	3.17	5
4.08	12	2.247	1	2.730	3	3.14	3
3.97	10	2.162	2	2.580	5	2.908	5
3.79	20	2.111	2	2.510	4	2.880	7
3.69	18	2.083	3	2.390	3	2.825	2
3.62	10	2.054	12	2.280	3	2.770	1

3.32	5
3.28	10
3.22	8
3.11	4
3.04	2
2.925	2
2.780	1
2.600	1
2.455	2
2.305	2
2.095	2
1.965	2

(42) Magnesium Silicate Monohydrate

13.08	4
10.01	30
9.29	500
8.37	30
7.25	11
5.03	5
4.67	100
4.55	11
4.20	11
3.60	5
3.44	8
3.35	24
3.27	14
3.11	522
2.895	16
2.801	8
2.699	11
2.590	8
2.523	5
2.473	11
2.333	14
2.317	5
2.194	5
2.102	3
1.989	3
1.925	3
1.900	2
1.890	1
1.811	1

(44) Meprobamate

13.7	100
8.05	8
6.85	34
5.16	3
4.92	11
4.72	60
4.38	4
4.03	6
3.94	59
3.74	11
3.72	13
3.69	29
3.51	30
3.36	5
3.30	3
3.12	5
3.05	4
2.986	5
2.728	6
2.682	4
2.590	4
2.571	4
2.509	2
2.469	3
2.366	3
2.343	5
2.271	2
2.227	2
2.193	2
2.090	2
2.055	3
1.931	2
2.160	4
2.090	3
2.015	3
1.960	2
1.922	5

(46) Methaqualone HCl

11.5	100
9.18	3
8.57	11
8.05	31
7.38	2
6.91	13
6.18	29
5.78	41
5.41	60
5.01	27
4.54	4
4.29	3
4.23	7
4.03	21
3.87	17
3.76	5
3.65	32
3.53	33
3.47	20
3.42	12
3.35	41
3.22	19
3.17	12
3.12	9
3.08	9
2.902	21
2.783	3
2.750	3
2.680	7
2.652	12
2.532	3
2.315	3
2.580	4
2.515	2
2.410	1
2.355	3
1.955	2
1.918	1

(48) Mescaline Sulfate Dihydrate

20.53	43
10.20	14
7.51	5
6.74	523
6.31	61
6.10	9
5.72	36
5.30	12
5.04	46
4.96	55
4.84	98
4.60	3
4.47	8
4.24	32
4.08	47
4.00	77
3.90	12
3.87	11
3.78	8
3.69	18
3.62	100
3.47	66
3.36	99
3.30	72
3.24	10
3.17	48
3.02	10
2.910	14
2.882	9
2.848	5
2.704	6

TABLE 2—*Continued.*

d, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1
2.627	5	2.041	3	3.3	6	(55) Phenobarbital Sodium	
2.518	22	1.975	3	3.2	4	14.52	61
2.460	5			3.13	3	11.72	45
2.346	6	(51) Methylene Dioxyamphetamine HCl (MDA)		3.04	6	11.27	36
2.235	10			3.02	9	9.75	27
2.205	5	12.8	7	2.92	10	0.09	42
2.148	6	12.4	45	2.877	29	8.12	70
		6.55	7	2.835	32	7.99	21
(49) Methyldimethoxymethylphenylethylamine HCl		5.34	53	2.81	15	7.25	85
		5.22	10	2.644	13	7.08	55
10.23	100	5.03	6	2.525	6	6.83	36
6.77	32	4.85	6	2.374	7	6.65	52
6.28	7	4.74	1			6.50	100
5.85	4	4.60	26	(53) Pentobarbital Sodium		6.23	27
5.68	15	4.53	18	14.11	100	5.80	39
5.41	15	4.45	4	11.97	84	5.67	27
5.07	35	4.19	21	11.13	42	5.58	24
4.77	4	4.15	20	9.99	280	5.47	27
4.44	6	4.09	38	8.98	23	5.19	18
4.39	4	4.02	100	8.53	10	4.90	61
4.28	7	3.86	88	7.85	29	4.79	52
3.97	11	3.79	81	6.99	10	4.68	36
3.83	11	3.74	25	6.65	32	4.47	82
3.69	5	3.57	24	6.35	32	4.31	30
3.57	31	3.33	7	5.98	81	4.20	21
3.48	8	3.27	94	5.55	48	4.12	27
3.36	80	3.17	14	4.98	65	3.90	55
3.33	28	3.13	8	4.67	26	3.82	58
3.10	7	3.08	34	4.44	36	3.72	39
2.915	13	3.03	19	3.86	13	3.54	39
2.879	5	3.01	17	3.56	6	3.34	18
2.739	6	2.927	84	3.36	10	3.25	33
2.587	4	2.890	4	(54) Phencyclidine HCl		3.19	27
2.560	5	2.858	5	9.54	12	3.03	27
2.442	5	2.825	15	9.42	16	3.003	24

2.400	4
2.313	5
2.235	7
2.189B	3
2.150	6
2.010	2
1.947	2
1.820	4
1.745	3

(50) Methylenedioxyamphetamine Carbonate

18.0	26
11.1	17
9.42	15
9.05	11
8.15	14
7.02	13
6.05	18
5.55	17
5.21	36
5.07	67
4.75	100
4.53	90
4.43	37
4.27	22
4.08	52
3.81	37
3.75	37
3.65	50
3.53	28
3.38	20
3.25	32
3.04	15
2.930	9
2.780	8
2.713	6
2.629	8
2.210	7
2.664	6
2.623	5
2.590	11
2.550	10
2.470	4
2.420	5
2.385	3
2.363	6
2.333	6
2.221	34
2.193	4
2.160	12
2.083	3
2.062	4
2.014	3
1.954	4
1.928	6
1.888	7
1.867	3
1.820B	3
1.794	4
1.779	6
1.754	5
1.729	4
1.707	6

(52) Methylphenidate HCl

10.6	100
8.48	57
6.98	13
6.13	36
5.85	40
4.53	50
4.32	38
4.26	76
3.88	33
3.75	45
3.53	41
3.43	16
{8.90	14
{8.68	58
7.00	4
6.38	8
6.15	100
5.81	29
5.33	7
4.71	31
4.52	6
4.37	30
4.30	38
4.08	41
3.91	12
3.84	40
3.75	6
3.66	21
3.34	18
3.185	12
{3.150	11
{3.135	25
2.960	5
2.915	5
2.898	8
2.863	7
2.788	4
2.735	3
2.698	5
2.640B	3
2.470B	3
2.342	2
2.289	12
2.180	2
2.135B	2
2.103	2
2.055	2
2.008	2
1.943	4
1.880	3
1.805	1
2.978	24
2.962	21
2.890	18
2.799	15
2.753	18
2.554	9
2.453	9
2.348	6
2.263	9
2.231	6
2.149	9

(56) Phenylephrine HCl

6.90	68
6.10	84
5.82	4
5.58	3
5.35	8
5.15	9
4.84	65
4.73	10
4.30	40
4.20	15
3.92	15
3.81	100
3.62	5
3.53	22
3.45	33
3.41	21
3.30	28
3.25	18
3.13	15
3.07	17
3.03	8
2.899	15
2.875	10
2.820	10
2.733	3
2.635	4

TABLE 2—*Continued.*

d, Å	*I*/*I*$_1$	*d*, Å	*I*/*I*$_1$	*d*, Å	*I*/*I*$_1$	*d*, Å	*I*/*I*$_1$
2.590	7	(59) Probenecid		2.743	4	2.770	4
2.569	7	18.5	86	2.470	2	2.730	6
2.536	5	18.0	100	2.415	3	2.665	3
2.487	5	9.15	39	2.365	2	2.620	4
2.453	7	7.40	28	2.270	2	2.575	4
2.331	7	6.69	4	2.235	3	2.500	2
2.154	3	6.06	10	2.213	2	2.465	3
2.041	4	5.53	51	2.134	3	2.410	2
		5.11	10			2.330B	2
(57) Phenylpropanolamine HCl		4.98	9	(62) Psilocybin		2.230B	2
17.1	100	4.53	50	14.2	7	2.140B	2
8.63	7	4.17	29	10.0	33		
7.89	4	3.97	17	8.85	12	(64) Quinine Sulfate	
7.71	17	3.89	6	7.74	41	14.1	14
6.34	5	3.79	8	7.40	43	9.82	47
6.10	28	3.55	24	7.08	11	9.32	9
5.33	11	3.49	8	6.42	23	7.11	4
5.23	9	3.28	9	6.13	100	6.66	5
5.00	14	3.14	7	5.96	11	6.31	1
4.82	3	3.09	29	5.52	11	6.09	5
4.74	4	2.94	4	5.00	9	5.49	3
4.45	13	2.834	2	4.73	5	5.13	100
4.32	20	2.745	3	4.56	43	4.79	20
4.14	9	2.59	4	4.38	27	4.55	9
3.86	9	2.482	2	4.30	9	4.00	17
3.82	7	2.29	6	4.24	11	3.87	5
3.75	20	2.177	4	4.14	16	3.66	3
3.67	18			4.02	22	3.54	11
3.61	7	(60) Prochlorperazine Dimaleate		3.86	25	3.45	17
3.48	35	8.84	4	3.81	20	3.40	4
3.41	7	6.15	25	3.67	32	3.31	4
3.20	7	5.50	29	3.46	20	3.26	5
2.975	8	5.27	20	3.34B	7	3.11	3
2.931	12	4.97	26	3.21B	11	2.961	8
2.860	4	4.68	41	3.02B	14	2.893	1

2.803	4
2.535	4
2.430	6
2.335	5

(58) Procaine HCl

12.6	6
6.88	36
6.26	35
5.43	79
4.96	3
4.71	8
4.44	16
4.16	49
4.07	100
3.92	24
3.74	24
3.69	23
3.61	12
3.56	34
3.44	10
3.27	13
3.18	26
3.13	15
3.10	25
2.985	2
2.925	10
2.872	3
2.700B	9
2.660	13
2.580	13
2.500B	5
2.365	4
2.270	10
2.190	2
2.148	2
2.050	4
1.995	6
1.789B	4

4.49	19
4.33	11
4.09	41
3.94	17
3.57	49
3.50	100
3.34	18
3.26	15
3.06	15
2.936	5
2.829	7
2.688	13
2.611	11
2.220	5

(61) Pseudoephedrine HCl

12.6	60
6.37	4
6.24	4
5.73	100
5.14	5
4.67	11
4.64	17
4.42	7
4.23	29
4.12	6
3.99	7
3.78	2
3.46	5
3.42	2
3.33	2
3.21	13
3.17	14
3.12	7
3.01	12
2.950	14
2.916	5
2.875	8
2.843	5

2.875	7
2.750	7
2.660B	3
2.555B	3
2.480B	3
2.330B	3
2.240	5
2.175	5
2.120	7

(63) Quinine HCl

10.0	100
7.34	24
6.18	3
6.00	4
5.86	7
5.62	16
5.42	22
5.16	67
5.02	34
4.92	13
4.64	12
4.50	10
4.37	8
4.30	14
4.23	21
4.13	14
3.69	8
3.60	15
3.56	16
3.43	9
3.40	9
3.34	12
3.25	4
3.20	3
3.12	8
3.04	7
2.925	3
2.845	3

2.805	2
2.660	3
2.626	2
2.542	1
2.470	2
2.360	1
2.207	1
2.143	1
2.077	1

(65) Saccharin

9.25	17
6.12	4
5.94	12
5.73	16
5.53	66
5.12	3
4.92	3
4.63	74
4.42	33
4.13	16
3.89	22
3.73	29
3.54	100
3.45	20
3.25	25
3.19	5
3.09	4
3.04	11
2.925	5
2.890	5
2.783	8
2.650	6
2.612	4
2.560	7
2.480	4

(66) Saccharin, Soluble USP

14.57	582

TABLE 2—*Continued.*

d, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1	*d*, Å	I/I_1
11.10	20	1.890	1	1.835B	3	3.130	5
9.38	10	1.860B	2	1.802	5	3.040	3
7.80	67			1.754B	2	2.955	2
7.67	26	(68) Salicylanilide		1.718	2	2.855	5
7.46	13	12.5	100	1.698	3	2.788	25
7.31	100	6.07	10	1.654	2	2.750	1
6.71	19	5.15	4	1.645	2	2.630B	1
6.58	16	4.57	30	1.622	3	2.490B	1
5.79	10	4.45	12	1.511	5	2.364	2
5.54	41	3.90	2			2.324	11
5.32	29	3.59	5	(71) Sodium Borate Hydrate		2.245	1
5.19	24	3.46	20	8.70	45	2.216	6
5.00	53	3.43	18	7.01	18	2.140	2
4.80	28	3.35	6	5.55	21	2.120	1
4.57	14	3.24	3	4.68	30	2.070B	1
4.44	51	3.07	10	4.63	11	2.006B	1
4.35	41	3.04	7	4.37	100	1.960B	1
4.26	16	2.941	3	3.87	8	1.900B	1
4.06	9			3.58	9		
3.88	28	(69) Secobarbital Sodium		3.55	10	(73) Sucrose	
3.82	21	14.06	78	3.42	63	10.70	14
3.65	57	12.00	33	3.18	5	7.62	59
3.56	60	10.44	100	2.995	28	6.98	28
3.48	47	6.26	39	2.921	100	6.77	41
3.44	16	6.08	28	2.880	6	5.75	22
3.37	19	5.71	24	2.757	19	5.46	8
3.33	67	5.57	22	2.645	3	5.33	15
3.28	52	5.21	26	2.589	19	4.91	7
3.16	16	4.93	13	2.551	7	4.73	84
3.04	21	4.68	26	2.382	15	4.55	71
2.972	17	4.44	20	2.326	10	4.38	17
2.912	41	3.89	15	2.259	20	4.28	26
2.789	26	3.37	5	2.186	30	4.04	22
2.677	12	3.16	6	2.163	6	3.96	10
2.632	12	2.753	3	2.138	7	3.79	10

2.582	12
2.451	10
2.360	9
2.302	9
2.209	10
1.964	26
1.940	9

(67) Salicylamide

10.5	61
6.48	42
5.58	50
5.45	90
5.26	59
4.86	37
4.70	4
4.25	27
4.14	8
3.88	7
3.60	4
{3.50	100
{3.48	56
3.42	10
3.21	62
3.07	4
2.900	3
2.860	3
2.783	36
2.700B	7
2.635	3
2.410	11
2.320	3
2.270B	5
2.145	3
2.118	6
2.030B	2
1.985B	1
1.940B	3

(70) Sodium Ascorbate

9.52	33
4.77	100
4.26	10
4.08	74
3.98	40
3.90	23
3.68	53
3.54	4
3.49	5
3.375	43
3.180	16
{3.095	39
{3.050	26
2.977	5
2.940	9
2.910	12
2.780	21
2.750	8
2.722	10
2.593	31
2.546	4
2.438	6
2.400	8
2.382	6
2.328	5
2.293	12
2.277	4
2.246	27
2.214	7
{2.128	19
{2.116	21
{2.057	12
{2.043	10
1.965B	4
1.944	13
1.905B	5
1.860B	4
2.102	4
2.040	3
{2.011	11
{2.001	19
1.958	4
1.937	15
1.897	6
1.855	12
1.830	1
1.793	23
1.762	12
1.658	4
1.644	5
1.625	3
1.606	3
1.567	6
1.546	3

(72) Sodium Salicylate

{22.5	9
{20.3	9
15.3	24
13.8	2000
12.9	22
7.70	1
6.96	100
6.46	7
5.04	7
4.73	2
4.64	7
4.12	1
3.94	2
3.86	1
3.58	1
3.48	170
3.37	9
3.33	7
3.225	4
3.70	4
3.60	100
3.54	52
3.38	8
3.26	7
3.23	4
3.12	8
2.890	18
2.806	14
2.749	8
2.717	3
2.687	5
2.587	5
2.549	3
2.528	4
2.499	3
2.453	5
2.450	4
2.437	5
2.414	7
2.351	16
2.316	4
2.262	12
2.239	7
2.192	3
2.174	4
2.080	3
2.064	3
1.907	4
1.800	3
1.696	3

There are discrepancies between the data in the Powder Diffraction File and those presented here, such as *d*-spacings, extra lines in the data developed for the present paper, and differences in relative intensities. Quinidine hydrochloride ($C_{20}H_{24}N_2O_2 \cdot HCl \cdot 2H_2O$) has occurred in some heroin preparations in place of a quinine salt. Quinidine, a myocardial depressant, is much more toxic than quinine, and its action is cumulative. No data are available in the Powder Diffraction File for quinidine hydrochloride.

Saccharin and Soluble Saccharin

Saccharin or o-sulfobenzimide ($C_7H_5NO_3S$) and soluble saccharin ($C_7H_4NNaO_3S \cdot 2H_2O$), both tablets and powder, are used in illicit preparations. The tablet surfaces are often treated with solutions of LSD or other hallucinogens, and the powder has been found in cocaine preparations. Often sodium bicarbonate is also present, an ingredient in many saccharin tablets which accelerates their dissolution.

Starches

Starch grains have been found in practically all types of illicit preparations. Wheat flour has been present in several heroin exhibits. Other starches which are commonly found are corn and potato starch. Starch grains are only semicrystalline, having an internal regular arrangement of micelles. X-ray diffraction patterns of the starches show five or six broad maxima of low intensity, the most intense maximum occuring at $d \simeq 5$Å. The starches can be differentiated using a polarizing microscope. X-ray diffraction chart recordings of mixtures containing starch exhibit an elevated background, as is the case when amorphous or semi-amorphous materials are present.

Sodium Salts

Those sodium salts which have been found in combination with controlled drugs are listed below.

sodium acetate trihydrate (in cocaine)
sodium benzoate
sodium borate pentahydrate
sodium tetraborate decahydrate (borax)
sodium bicarbonate
sodium chloride
sodium cyclamate (in cocaine)
monosodium glutamate
soluble saccharin
sodium sulfate
sodium tartrate

Only those compounds not listed in the Powder Diffraction File, or where there are differences between the data file and the present study, are included in the tables of the present paper.

Sugars

By far the most common excipient in illicit preparations is lactose monohydrate. It has been found in heroin samples, either as the sole excipient or in combination with a

quinine salt, mannitol, dextrose monohydrate, sucrose, or many other less commonly occurring materials. The X-ray diffraction data for lactose monohydrate in the Powder Diffraction File, taken from film, list the two most intense lines having the *d*-spacings of 4.35 (I/I_1 = 100) and 4.21 Å (I/I_1 = 80), the former *d*-spacing designated as being broad. A diffractometer chart recording resolves the two lines into five distinct and characteristic peaks, as shown in Fig. 1, a group of three peaks followed by two peaks,

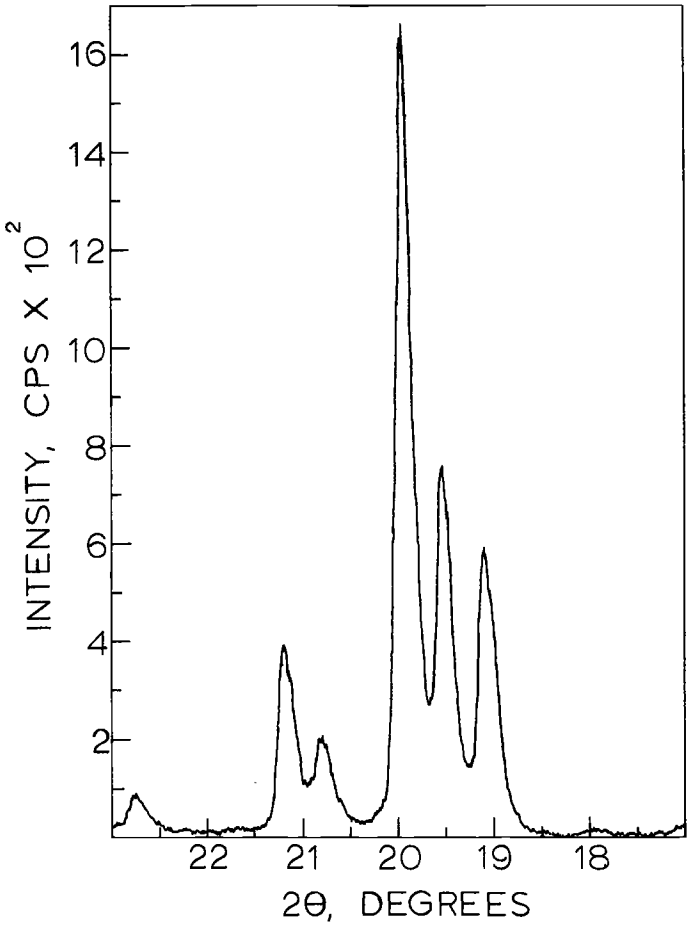

FIG. 1—*Characteristic peak groups for lactose monohydrate.*

each group without background resolution. The most intense peak is very close to 20 deg 2θ, and the three most intense lines make up the triplet; the *d*-spacings are 4.46 Å (I/I_1 = 100), 4.54.Å (I/I_1 = 50), and 4.64 Å (I/I_1 = 39). Crystals of lactose monohydrate are wedge-shaped and are randomly oriented when packed. There is no preferred orientation, even when the crystals are relatively large. Therefore, if a peak of another substance in the mixture coincides with one attributable to lactose monohydrate,

the effect is additive and easily recognized since the lactose peak would have a relative intensity greater than it would be for pure lactose. Dextrose monohydrate and sucrose occur frequently as cutting materials. Mannitol, a sugar alcohol, is another common excipient in heroin samples. Occasionally anhydrous dextrose and β-lactose have been found combined with controlled drugs.

Talc

Talc or magnesium silicate ($Mg_3Si_4O_{11} \cdot H_2O$) crystals orient preferentially, even when finely crushed. Two very intense peaks occur in the diffraction pattern as a result (d = 3.11 and 9.29 Å). As is the case for caffeine, indication that talc is present follows when intense lines occur having these two d-spacings. The lines appear even when a relatively small amount of talc is present in a mixture. Occasionally two different crystal forms of talc are present in the same preparation. Only one form of talc is included in this paper. Data for the other form, as shown in the inorganic powder diffraction file, are adequate for its identification.

Summary

X-ray powder diffraction data have been developed or refined for the identification of drugs, excipients, and adulterants found in illicit preparations.

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