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X-RAY DIFFRACTION POWDER DATA FOR STEROIDS: SUPPLEMENT VII

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X-ray diffraction powder analysis has proved to be a convenient method for use in establishing or confirming the identification of crystalline materials. In order to use the above method, however, it is necessary that standard pattern data be available for the particular compounds being investigated.

Steroids comprise a large group of compounds including hormones, bile acids, sterols, and synthetic and natural drugs. Naturally occurring steroids are important in the structure, growth and function of all biological systems. These compounds contain a fused reduced 17-carbon-ring system, cyclopentoperhydrophenanthrene. With this ring system as the nucleus, substitutions are made at various positions making possible a large number of different steroids.[4] Because steroids are insoluble in water and soluble in solvents such as acetone, ethyl alcohol, methyl alcohol and benzene, they are included with compounds classed as lipids.

The X-ray diffraction powder data program for steroids started in 1956 in the Physics Department of the Edsel B. Ford Institute for Medical Research. The first data report appeared in the Henry Ford Hospital Medical Bulletin in 1958.[1] Since that time six supplements to the above first steroid data report have appeared.[2-7] This paper brings the total number of steroids for which data has been assembled to 476.

A classified list of the 29 steroids included in this supplement is given in Table I. The melting point ranges of all these steroid specimens were carefully checked for agreement with the literature after recrystallization from ethanol. The method used for recrystallization of these steroids was described on page 368 of the original paper[1] in this series.

The data for the steroids included in this supplement are given in Table II with the numbers at the head of the data columns corresponding to specific steroids as listed in Table I. Data for each steroid consists of (1) the interplanar spacings

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(d,A) as measured and calculated from the X-ray diffraction powder pattern lines and (2) the relative intensity I/I_1 of each pattern line as determined using a Joyce-Loebl automatic recording microdensitometer. The strongest line of each set of data is recorded as 1.00 and the other lines are listed relatively as decimal fractions.

As has been the case with the earlier published data in this series, pattern photographs are included at the end of the paper for use in quick pattern comparison.

ACKNOWLEDGMENTS

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This work has been partially supported by grants from the Joint Committee on Powder Diffraction Standards which is a joint project of the American Crystallographic Association, The American Society for Testing and Materials, and the British Institute of Physics.

The Photography Department of Henry Ford Hospital reproduced the pattern photographs.

REFERENCES

1. Parsons, J., Beher, W. T., and Baker, G. D.: X-ray diffraction powder data and index for the steroids. Henry Ford Hosp. Med. Bull. 6:365-422, Dec., pt. II 1958.

2. Parsons, J., Beher, W. T. and Baker, G. D.: X-ray diffraction powder data for steroids. Henry Ford Hosp. Med. Bull. 9:54-62, Mar. 1961.

3. Parsons, J., Wong, S. T., Beher, W. T., and Baker, G. D.: X-ray diffraction powder data for steroids: Supplement II, Henry Ford Hosp. Med. Bull. 10:471-486, Sept. 1962.

4. Parsons, J., Wong, S. T., Beher, W. T., and Baker, G. D.: X-ray diffraction powder data for steroids: Supplement III. Henry Ford Hosp. Med. Bull. 11:23-52, Mar. 1963.

5. Parsons, J., Wong, S. T., Beher, W. T., and Baker, G. D.: X-ray diffraction powder data for steroids: Supplement IV. Henry Ford Hosp. Med. Bull. 12:87-120, Mar. 1964.

6. Parsons, J., Wong, S. T., and Beher, W. T.: X-ray diffraction powder data for steroids: Supplement V. Henry Ford Hosp. Med. Bull. 12:459-475, Dec., pt. I, 1964.

7. Parsons, J., Wong, S. T., and Beher, W. T.: X-ray diffraction powder data for steroids: Supplement VI. Henry Ford Hosp. Med. Bull. 13:303-316, Sept. 1965.

Table I

Index To Steroid X-ray Diffraction Powder Data

Pattern Number	Name	Molecular Formula	Melting Point (Uncorr.) °C
	Carbon-18 steroids and derivatives		
1	$\triangle^{1,3,5(10),6}$-Estratetraen-3β-ol-17-one	$C_{18}H_{20}O_2$	261-263
	Halogen substituted steroids		
2	$\triangle^{5}$-Pregnen-3β,17α-diol-20-one-16β-bromo	$C_{21}H_{31}O_3Br$	194-196
	Dihydric alcohols		
3	5α-Androstan-3β,17α-diol	$C_{19}H_{32}O_2$	212-213
	Monoketones		
4	5β-Pregnan-20-one	$C_{21}H_{34}O$	115-117
	Triketones		
5	5α-Androstan-3,11,17-trione	$C_{19}H_{26}O_3$	178-180
6	5α-Pregnan-3,6,20-trione	$C_{21}H_{30}O_3$	234-236
	Monohydroxy-monoketones		
7	$\triangle^{4,9(11)}$-Androstadien-17β-ol-3-one-17α-methyl	$C_{20}H_{28}O_2$	168-169
8	5α-Androstan-17β-ol-3,3-dimethoxy-17-hexahydrobenzoate	$C_{28}H_{47}O_4$	134-136
9	5β-Androstan-17β-ol-17α-methyl-3-one	$C_{20}H_{32}O_2$	117-118
10	$\triangle^{5}$-Pregnen-3β-ol-20-one-3-hemisuccinate	$C_{25}H_{36}O_5$	171-173
	Dihydroxy-monoketones		
11	5β-Androstan-3α,11β-diol-17-one	$C_{19}H_{30}O_3$	239-241
12	$\triangle^{4}$-Androsten-6β,17β-diol-3-one	$C_{19}H_{28}O_3$	213-214
13	5β-Pregnan-3α,6α-diol-20-one	$C_{21}H_{34}O_3$	192-193
	Trihydroxy-monoketones		
14	$\triangle^{5}$-Pregnen-3β-ol-20-one-16α,17α-epoxy	$C_{21}H_{30}O_3$	190-192
15	$\triangle^{5}$-Pregnen-3β-ol-20-one-16α,17α-epoxy-16β-methyl	$C_{22}H_{32}O_3$	188-190
	Tetrahydroxy-monoketones		
16	5β-Pregnan-3α,17α,20α,21-tetraol-11-one	$C_{21}H_{34}O_5$	208-211
	Monohydroxy-diketones		
17	5β-Androstan-3α-ol-11,17-dione	$C_{19}H_{28}O_3$	189-190
18	5β-Pregnan-21-ol-3,20-dione	$C_{21}H_{32}O_3$	152-154
19	$\triangle^{4}$-Pregnen-11α-ol-3,20-dione-11-acetate	$C_{23}H_{32}O_4$	177-179
20	5β-Pregnan-17α-ol-3,11,20-trione	$C_{21}H_{30}O_4$	204-206
	Trihydroxy-diketones		
21	5α-Pregnan-11β,17α,21-triol-3,20-dione	$C_{21}H_{32}O_5$	233-234
22	$\triangle^{4}$-Pregnen-11α,17α,21-triol-3,20-dione	$C_{21}H_{30}O_5$	210-212
	Carboalkoxy acids		
23	$\triangle^{5}$-Androsten-3β-ol-17β-carboxylic acid	$C_{20}H_{30}O_3$	277-279
24	$\triangle^{5}$-Androsten-3β-ol-17β-carboxylic acid-3-acetate	$C_{22}H_{32}O_4$	238-240
25	5α-Cholanic acid-3,6-dione	$C_{24}H_{36}O_4$	161-163
26	5β-Cholanic acid-3α-ol-7-one	$C_{24}H_{38}O_4$	203-205
27	$\triangle^{5}$-Cholenic acid-3β-ol	$C_{24}H_{38}O_3$	228-230
	Carboalkoxy esters		
28	5β-Cholanic acid-3α,12α-diol-methyl ester	$C_{25}H_{42}O_4$	81
29	5β-Cholanic-3α-ol-12-one-methyl ester-3-benzoate	$C_{32}H_{44}O_5$	127-130

TABLE II

1		2		3	
d,A	I/I_1	d,A	I/I_1	d,A	I/I_1
10.94	.05	12.41	.05	11.57	.57
7.93	.08	8.69	.25	10.33	.30
6.68	.20	6.27	.15	7.75	.27
6.44	1.00	6.04	.29	7.58	.32
5.77	.25	5.59	1.00	6.83	.32
5.00	.11	5.23	.26	6.04	.86
4.54	.52	5.01	.16	5.96	1.00
4.24	.77	4.32	.16	5.20	.95
3.96	.17	4.18	.43	4.99	.30
3.79	.11	3.93	.11	4.82	.75
3.69	.13	3.83	.12	4.55	.14
3.57	.13	3.67	.17	4.20	.20
3.36	.11	3.47	.15	3.57	.11
3.25	.10	3.39	.24	3.41	.16
3.14	.11	3.25	.13	2.64	.14
3.03	.15	3.13	.11	2.18	.11
2.89	.07	3.01	.21		
2.79	.13	2.94	.14		
2.74	.07	2.80	.11		
2.68	.06	2.68	.13		
2.57	.06	2.47	.11		
2.32	.11	2.40	.05		
2.27	.04	2.35	.04		
2.22	.04	2.25	.05		
2.15	.04	2.09	.05		
2.11	.06	2.02	.04		
2.05	.03	2.01	.04		
		1.92	.06		
		1.81	.04		

4		5		6	
d,A	I/I_1	d,A	I/I_1	d,A	I/I_1
10.81	.12	12.48	.06	11.98	.07
9.87	.11	7.36	.10	7.82	.13
8.71	.05	6.32	.46	7.33	.25
7.73	.39	5.81	.26	6.86	.14
7.52	.29	5.25	1.00	5.92	1.00
7.20	.21	4.94	.14	5.72	.37
6.79	.27	4.60	.49	5.04	.78
6.20	.94	4.30	.17	4.65	.22
6.03	.20	4.07	.14	4.12	.15
5.74	.62	3.71	.17	4.02	.14
5.39	1.00	3.45	.15	3.71	.20
5.22	.19	3.34	.14	3.64	.19
5.06	.30	3.24	.16	3.43	.16
4.90	.38	3.17	.15	3.25	.19
4.38	.28	3.07	.27	3.05	.20
4.10	.48	2.90	.17	2.97	.15
3.96	.07	2.76	.14	2.88	.14
3.80	.11	2.64	.10	2.77	.13
3.55	.19	2.53	.09	2.56	.06
3.42	.07			2.46	.06
3.26	.11			2.37	.07
3.18	.07				
3.10	.08				
3.04	.06				
2.94	.07				
2.87	.05				
2.80	.07				
2.76	.06				
2.62	.08				
2.53	.10				

7

d,A	I/I_1
10.60	.03
6.18	1.00
5.94	.65
5.73	.39
5.52	.79
5.30	.20
4.99	.32
4.68	.17
4.34	.48
4.09	.42
3.88	.17
3.76	.17
3.48	.16
3.43	.24
3.38	.19
3.29	.13
3.20	.11
3.06	.17
2.96	.11
2.83	.10
2.75	.21
2.68	.10
2.60	.06
2.57	.06
2.51	.03
2.44	.05
2.39	.04

8

d,A	I/I_1
14.03	.06
9.78	.05
8.54	.05
6.05	.41
5.87	.55
5.75	.50
5.42	1.00
5.20	.70
5.04	.50
4.91	.39
4.25	.18
4.11	.12
3.64	.08
3.45	.08
3.08	.07

9

d,A	I/I_1
12.42	.24
10.81	.08
9.19	.10
7.97	.34
7.40	.23
6.81	.68
6.07	1.00
5.54	.87
5.17	.81
5.06	.54
4.80	.34
4.60	.24
4.43	.12
4.17	.20
3.99	.10
3.83	.09
3.68	.11
3.45	.13
3.15	.09

10

d,A	I/I_1
9.55	.09
7.65	.05
7.10	.17
5.94	.23
5.73	.39
5.29	.84
4.83	1.00
4.54	.22
4.16	.39
3.68	.16
3.44	.16
3.31	.18
2.96	.08

11

d,A	I/I_1
7.86	.47
6.82	.04
6.13	.56
5.66	1.00
5.40	.29
5.27	.60
4.91	.45
4.55	.40
4.39	.11
4.26	.31
4.09	.05
3.93	.12
3.79	.12
3.64	.12
3.45	.14
3.20	.07
3.11	.17
2.93	.08
2.87	.04
2.76	.05
2.70	.06
2.58	.25
2.51	.05
2.43	.06
2.39	.04
2.33	.08
2.26	.03
2.22	.06
2.11	.12
1.98	.12

12

d,A	I/I_1
10.35	.08
9.04	.03
7.96	.06
7.20	.09
6.78	.10
6.16	.26
5.95	1.00
5.35	.13
5.11	.65
4.65	.33
4.30	.08
4.15	.07
3.98	.09
3.59	.08
3.50	.06
3.38	.09
3.25	.06
3.09	.06
2.95	.06
2.80	.04
2.49	.06
2.42	.05

13

d,A	I/I_1
8.30	.17
6.98	.62
6.63	1.00
5.66	.99
5.33	.12
5.17	.95
4.65	.51
4.49	.23
4.24	.31
3.93	.17
3.74	.13
3.51	.17
3.42	.10
3.34	.14
3.29	.14
3.17	.19
3.07	.09
2.99	.14
2.80	.14
2.70	.08
2.62	.09
2.56	.09
2.50	.09
2.31	.09
2.25	.04
2.22	.04
2.17	.04
2.10	.08
2.04	.06
2.00	.05

14

d,A	I/I_1
13.07	.07
8.76	.07
7.58	.05
6.68	.03
6.43	.06
5.77	1.00
5.42	.12
4.93	.14
4.66	.55
4.41	.13
3.97	.10
3.83	.19
3.63	.06
3.53	.12
3.42	.02
3.30	.05
3.18	.07
3.08	.08
2.95	.08
2.82	.03
2.78	.06
2.60	.02
2.53	.03
2.42	.01
2.32	.04
2.22	.03
2.17	.04
2.13	.02
2.07	.03
2.00	.02
1.94	.03
1.89	.03
1.81	.03

15

d,A	I/I_1
12.85	.19
11.82	.06
10.94	.07
10.16	.07
9.16	.11
8.41	.10
7.28	.09
6.93	.13
6.53	.12
6.13	.43
5.86	1.00
5.59	.80
5.03	.62
4.82	.16
4.64	.34
3.95	.38
3.79	.16
3.46	.16
3.37	.15
3.08	.11
2.95	.14

16

d,A	I/I_1
11.82	.03
10.50	.08
8.27	.03
6.48	.26
6.27	.12
5.83	.67
5.69	1.00
5.26	.20
4.98	.03
4.70	.32
4.44	.21
4.34	.19
4.15	.17
3.90	.06
3.68	.03
3.28	.12
3.12	.04
2.93	.10
2.84	.04
2.74	.05
2.66	.09
2.62	.11
2.42	.03
2.40	.03
2.23	.02
2.20	.02
2.13	.03
2.07	.10

17

d,A	I/I_1
8.97	.29
8.43	.09
7.67	.07
7.16	.08
6.76	.52
6.03	.76
5.76	.31
5.35	.33
5.20	1.00
4.85	.16
4.73	.34
4.47	.19
4.07	.23
4.03	.11
3.94	.12
3.83	.19
3.71	.15
3.48	.10
3.33	.14
3.19	.06
2.98	.13
2.89	.06
2.83	.06
2.77	.06
2.72	.04
2.57	.08
2.49	.06
2.40	.06
2.32	.07
2.26	.07

18

d,A	I/I_1
7.55	.14
6.68	.55
6.38	.66
6.06	.42
5.87	.75
5.38	.13
5.14	1.00
4.90	.35
4.73	.58
4.52	.27
4.01	.63
3.74	.56
3.68	.39
3.56	.22
3.25	.25
3.11	.20
3.03	.20
2.94	.14
2.88	.16
2.79	.17
2.50	.08
2.46	.07
2.35	.14
2.28	.11
2.24	.06
2.21	.07
2.18	.05
1.96	.08
1.89	.08

19

d,A	I/I_1
9.57	.04
8.88	.07
7.34	.40
6.92	1.00
5.84	.06
5.50	.36
5.26	.30
5.01	.14
4.76	.66
4.42	.13
4.28	.13
4.11	.13
4.00	.23
3.70	.15
3.60	.20
3.37	.27
3.22	.14
3.14	.11
3.02	.14
2.85	.09
2.81	.11
2.63	.06
2.50	.04
2.44	.03
2.39	.03

20

d,A	I/I_1
11.08	.16
9.67	.25
9.02	.24
7.94	.17
7.50	.15
6.99	.15
6.29	.84
5.92	1.00
5.67	.52
5.39	.59
4.93	.14
4.83	.38
4.69	.13
4.54	.11
4.33	.13
3.89	.08
3.74	.14
3.63	.15
3.48	.11
3.37	.06
3.32	.08
3.21	.09
3.14	.06
3.07	.15
2.95	.06
2.86	.06
2.76	.15
2.68	.10
2.57	.06
2.43	.08

21

d,A	I/I_1
11.80	.03
9.59	.05
8.16	.03
6.72	.04
6.08	1.00
5.80	.05
5.46	.12
5.03	.69
4.66	.15
4.51	.10
4.37	.08
3.85	.06
3.67	.05
3.21	.05
3.04	.09
2.93	.04
2.45	.05
2.42	.04
2.08	.05

22

d,A	I/I_1
12.45	.20
10.14	.08
8.54	.08
7.33	.20
6.40	.38
5.96	.37
5.43	1.00
5.18	.22
4.83	.23
4.47	.10
4.26	.17
4.07	.13
3.62	.11
3.38	.13
3.11	.10
2.91	.09
2.86	.04

23

d,A	I/I_1
12.14	.08
5.85	1.00
4.83	.55
4.66	.35
4.06	.07
3.73	.15
3.39	.07
2.95	.09

24

d,A	I/I_1
12.48	.08
9.72	.08
8.79	.07
8.56	.12
7.46	.12
6.93	.33
6.05	1.00
5.84	.18
5.57	.25
5.42	.60
5.04	.16
4.61	.96
4.18	.32
4.02	.21
3.82	.18
3.78	.15
3.55	.19
3.50	.15
3.33	.20
3.13	.20
2.99	.14
2.84	.12
2.65	.10
2.56	.05
2.51	.05
2.36	.05
2.30	.07
2.21	.06
2.18	.08

25

d,A	I/I_1
6.46	.90
5.89	1.00
5.35	.93
5.15	.71
4.58	.39
4.44	.46
3.99	.49
3.36	.34
3.11	.27

26

d,A	I/I_1
12.92	.27
10.38	.02
8.97	.06
7.13	.30
5.99	.41
5.79	1.00
5.46	.37
4.94	.24
4.74	.23
4.45	.33
4.24	.25
4.19	.23
3.98	.20
3.85	.17
3.73	.17
3.57	.15
3.45	.21
3.33	.18
3.21	.14
3.16	.13

27

d,A	I/I_1
14.35	.05
7.67	.13
6.03	.19
5.66	.99
4.86	1.00
4.54	.16
4.40	.20
4.26	.16
4.18	.15
4.02	.19
3.85	.22
3.75	.20
3.69	.21
3.52	.19
3.42	.19
3.34	.16
3.16	.19
2.97	.16
2.89	.27
2.84	.19
2.55	.08
2.43	.09
2.37	.06
2.32	.06
2.21	.05
2.10	.06
2.07	.05
2.02	.05
1.97	.05

28

d,A	I/I_1
12.05	.09
10.63	.09
9.62	.23
8.78	.16
8.04	.53
7.20	.28
6.55	.15
6.12	.52
5.69	.14
5.44	.97
5.32	.36
4.99	.26
4.86	.73
4.81	1.00
4.40	.22
4.30	.39
4.15	.15
4.01	.18
3.88	.23
3.76	.20
3.61	.22
3.56	.22
3.50	.20
3.38	.22
3.29	.07
3.20	.15

29

d,A	I/I_1
11.98	.21
11.05	.43
9.35	.29
8.12	.59
7.10	.16
6.83	.22
6.38	.18
5.52	1.00
5.33	.41
5.02	.29
4.81	.59
4.68	.71
4.47	.50
4.01	.40
3.85	.28
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3.57	.41
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3.13	.17
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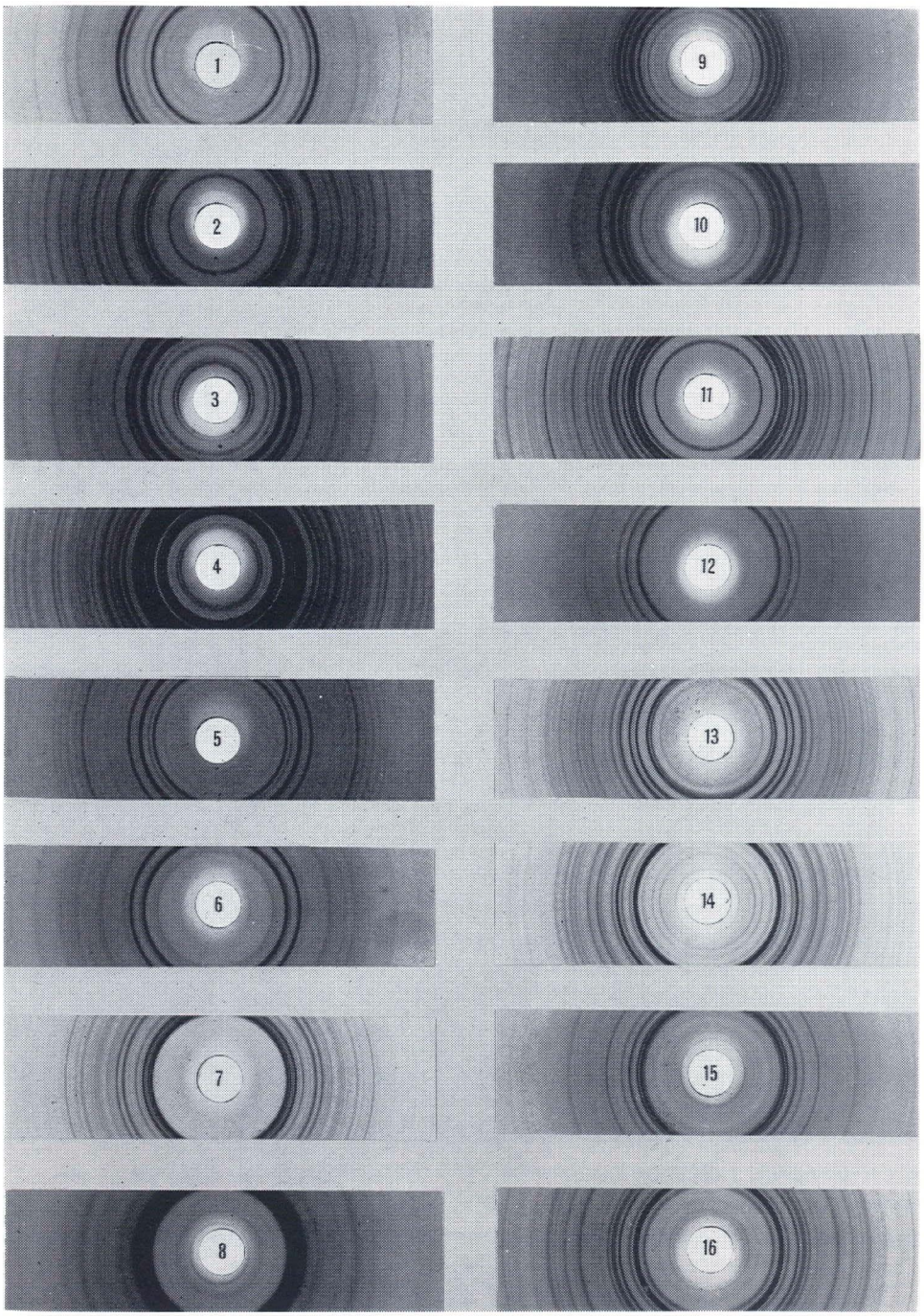
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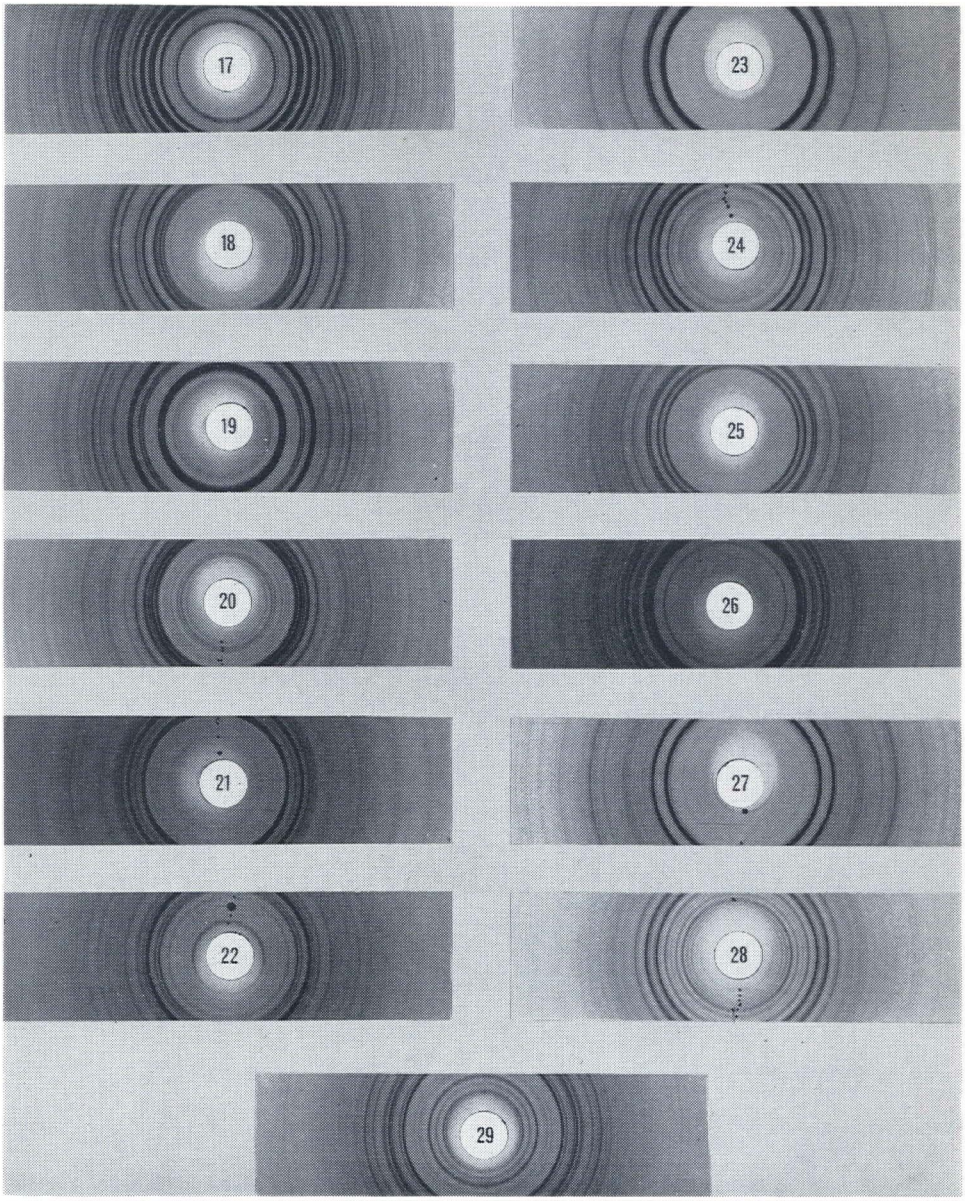
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