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A Comparison of Various Commercially Available X-ray Films*

(*Received* 6 *February* 1956)

The speeds for Cu and Mo radiation, the fog density, the granularity, the absorption of Cu radiation, and the density versus X-ray exposure curve have been determined for forty-one commercially available types of X-ray film. The results are tabulated and a short description is given of the experimental procedure followed.

1. Introduction

Manufacturers of X-ray film describe in their brochures only the properties of their own films; moreover, the information given is usually rather qualitative. Consequently, X-ray crystallographers commonly choose their X-ray film in the absence of reliable information about the relative merits of the various types of film which are commercially available. To the Commission on Crystallographic Apparatus of the International Union of Crystallography it therefore seemed desirable to have the properties of X-ray films of different makes and types compared.

Grants from the UNESCO and the International Union of Crystallography made it possible to realize this project. It has been carried out in the Laboratory of Inorganic and Physical Chemistry of the University of Groningen, The Netherlands, under the supervision of D. W. Smits and E. H. Wiebenga. Most of the experimental work was done by A. A. de Sturler, part of it by W. Drenth. Assistance in the computational work was rendered by A. P. Mosterman and H. Schürer.

Forty-one different types of X-ray film were collected and examined; four of these films were single coated. Some types of film were supplied directly by the manufacturers; others were obtained through the usual commercial channels.

2. Experimental

(*a*) *Intensity scales*

Most film samples had an approximate size of 13×18 cm. As shown in Fig. 1, there were printed on each film:

(i) A set of 35 equally exposed spots, which were made using Mo radiation.
(ii) Two sets of two identical intensity scales, each scale consisting of 20 circular spots with diameters of 2 mm., of which the exposures formed an arithmetical series. One of the two sets of intensity scales was made with Mo radiation, the other set with Cu radiation.

The equal exposures were obtained in the following way. The film was mounted between two brass plates, one of which was provided with a series of 35 holes.

* This paper describes the results of an investigation sponsored by the Commission on Crystallographic Apparatus of the International Union of Crystallography.

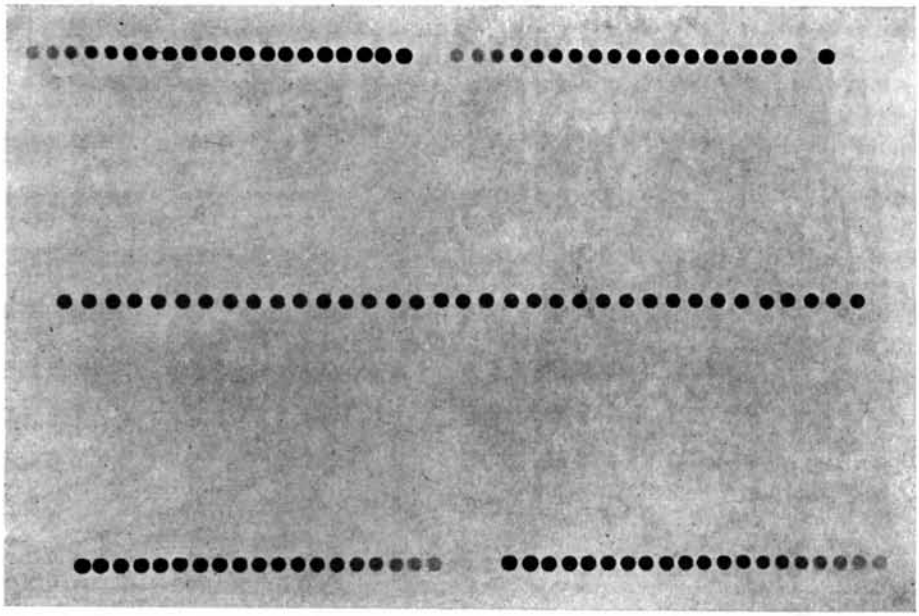

Fig. 1. Middle part of exposed film sample. In one of the intensity scales for Cu radiation the 19th spot was omitted in order to distinguish them from the Mo scales.

The plates and film were moved with a uniform speed past a slit (dimensions 5×15 mm.) in a large lead screen, using a synchronous motor. Radiation from an X-ray tube at a distance of 1·5 m. from the lead screen passed through the slit and then struck the film through the circular holes in the filmholder.

The input of the high-voltage generator was stabilized, and the high voltage obtained was rectified and smoothed by a condenser; the filament current as well as the tube current were electronically stabilized. This made the output of the X-ray tube constant to within 1 or 2%, as was checked by means of a Geiger counter. Since, moreover, the film holder made several runs, between 2 for the fastest films and 20 for the slowest ones, it may be assumed that the exposures of the 35 spots were equal to about 1%.

The intensity scales were made in a similar way. Again the film was moved past a slit in a lead screen, through which the X-rays entered. The radiation struck the film through two sets of 20 holes in a brass screen mounted in front of the film. The increasing exposures of the spots were obtained by interrupting the X-rays, which had passed through the slit in the lead screen, by a fast rotating cylinder. In this cylinder, which was driven by a non-synchronous motor, series of steps of increasing angular opening were cut out. The film, the screen and the rotating cylinder were mounted on the same carriage, which was driven by a synchronous motor. For further details, see Smits (1952).

The Mo radiation, generated at 35 kV., was filtered by 0·1 mm. Zr and 0·4 mm. Al; the Cu radiation, used for the second set of intensity scales, was generated at 15 kV. and filtered by 0·03 mm. Ni.

(b) Processing

Processing was carried out in thermostatically controlled tanks with the films hung vertically and with the rows of spots horizontal. The recommendations of the manufacturers with respect to developer and developing temperature were followed, except in a few cases where these data were not known to us. For details concerning the developing times, see Table 1. During development the films were gently moved up and down every minute for about 10 sec.

After developing, the films were rinsed in distilled water for about 30 sec., fixed in an acid fixing bath for 10 min., rinsed in tap water for an hour, and dried in the air at room temperature.

(c) Density measurements

The densities were measured relative to reference strips which were obtained by fixing, rinsing and drying a strip of each type of film without previous development. They are thus defined by $D = \log_{10} I_0/I$, in which I_0 and I are the intensities of a light beam transmitted respectively by the reference strip and the film. The measured densities D of the exposed spots were corrected for the fog density D_F, which is the density of the non-exposed part of the developed film. This correction was made by simply subtracting D_F from D; the differences are called the effective densities $D_{\text{eff.}}$.

Since the density depends somewhat on the geometry of the optics of the densitometer, the latter is schematically shown in Fig. 2.

The filament of a lamp L with stabilized lamp current was imaged by a microscope objective lens O_1

Table 1

No.	Type of film	Development: Developer	Development: Time* (min.)	Development: Temp. (°C.)	Speeds: Cu	Speeds: Mo	Fog density	$10^3 \times G$	F	Characteristic curve; relative exposures required for $D_{eff.}$ = 0·2	0·4	0·6	0·8	1·0	1·2	1·4	1·6	1·8	2·0
	Agfa, Germany																		
1	Super Spezial S. F.	Ilford ID 42	10(*d*)	20	14	10	High	7	1·65	17	34	54	75	100	127	157	188	222	—
	Ceaverken, Sweden																		
2	Textur X Direct	Ilford ID 42	5(*d*)	20	50	44	Medium	12	2·25	17	35	55	77	100	124	151	180	—	—
	Dupont, U.S.A.																		
3	Type 502 (Par Speed)	Dupont Xtra-Fast	5(*b*)	20	26	19	Medium	7	1·75	16	33	52	75	100	128	160	195	231	—
4	Type 504 (Screen Film)	*idem*	5(*b*)	20	26	17	Medium	8	1·75	17	35	55	76	100	127	—	—	—	—
5	Type 506 (Fine Grain)	*idem*	10(*a*)	20	11	8	Very low	6	—	20	40	60	80	100	120	143	166	191	217
6	Type 507 (Photo Roentgen, single coated)	*idem*	8(*b*)	20	22	14	Medium	9	1·75	17	34	52	74	100	128	159	196	239	287
7	Type 508 (Xtra Fast)	*idem*	5(*b*)	20	22	16	Medium	8	1·75	16	32	53	75	100	128	160	196	235	—
8	Type 510 (Fine Grain)	*idem*	10(*d*)	20	5	5	Very low	4	2·25	20	40	60	80	100	120	140	160	180	200
	Fuji, Japan																		
9	Industrial, Type 200	Fuji Rendol FD-R	10(*c*)	20	100	100	High	12	3·10	18	37	56	78	100	124	149	176	203	—
	Gevaert, Belgium																		
10	Osray	Gevaert G 230	6(*b*)	18	46	46	Low	12	3·10	19	38	58	79	100	122	144	167	191	—
11	Structurix D 7	*idem*	10(*c*)	18	13	12	Low	8	2·30	20	40	60	80	100	122	144	168	193	—
12	Structurix D 10	*idem*	6(*b*)	18	48	48	Medium	14	3·35	18	36	56	78	100	124	149	176	—	—
13	Structurix S	*idem*	5(*b*)	18	30	26	Medium	12	1·95	17	34	54	76	100	125	153	186	230	—
	Guilleminot, France																		
14	Radioguil	Kodak D 19*b*	15(*e*)	20	19	16	Very low	12	1·90	23	45	65	82	100	119	139	160	182	205
	Ilford, England																		
15	Ilfex	Ilford ID 42	7(*b*)	20	55	50	Medium	13	2·75	19	38	57	78	100	123	146	170	197	—
16	Industrial B	*idem*	8(*b*)	20	19	22	Medium	8	2·60	19	38	58	79	100	121	142	164	—	—
17	Industrial C	*idem*	12(*a*)	20	8	8	Very low	6	2·30	21	42	62	81	100	119	137	155	173	190
18	Industrial G	*idem*	8(*b*)	20	60	55	Medium	12	2·70	20	39	59	79	100	121	144	168	193	—
19	Red Seal	*idem*	4(*b*)	20	32	24	Medium	15	1·80	18	36	55	76	100	127	156	187	—	—
20	Single coated	*idem*	7(*b*)	20	28	19	Medium	9	1·80	14	32	53	75	100	127	157	191	228	—
	Kodak, England																		
21	Crystallex (Code 2)	Kodak D 19*b*	12(*a*)	20	14	14	Very low	6	2·60	19	39	59	79	100	122	145	169	195	—
22	Industrex D	*idem*	8(*b*)	20	36	32	Medium	11	2·65	19	38	58	79	100	121	143	165	—	—
23	Industrex S	*idem*	7(*b*)	20	30	24	Medium	10	2·05	18	36	55	77	100	126	155	—	—	—
24	Kodirex (Code 1)	*idem*	8(*b*)	20	38	32	Medium	11	2·65	18	36	56	77	100	124	148	174	200	—
	Kodak, France																		
25	Definix	Kodak D 19*b*	15(*e*)	20	18	16	Very low	6	2·30	20	40	60	80	100	121	143	165	188	211
26	Kodirex Monocouche	Kodak Rapid	10(*d*)	20	38	26	Medium	9	1·80	17	36	55	77	100	125	152	181	212	245
27	Regulix	Kodak D 19*b*	5(*b*)	20	26	20	Medium	9	1·65	17	35	55	77	100	125	152	181	211	242

	Kodak, U.S.A.																		
28	Blue Brand (Medical)	Kodak Rapid	10(*c*)	20	38	26	Medium	11	1·80	16	32	51	74	100	130	164	202	244	—
29	Industrial, Type A	*idem*	10(*a*)	20	14	12	Low	5	2·15	20	40	60	80	100	121	142	164	188	212
30	Industrial, Type F	*idem*	5(*b*)	20	28	20	Medium	7	1·80	16	33	53	75	100	128	159	—	—	—
31	Industrial, Type K	*idem*	10(*a*)	20	80	75	Medium	11	3·40	19	38	58	79	100	123	147	172	198	—
32	Industrial, Type M	*idem*	10(*a*)	20	5	4	Very low	3	2·10	20	40	60	80	100	120	140	160	—	—
33	No Screen (Medical)	*idem*	10(*c*)	20	50	55	Low	12	3·80	20	40	60	80	100	120	140	162	185	—
34	Single coated (Blue sensitive)	*idem*	7(*b*)	20	26	17	Medium	7	1·75	14	30	50	73	100	132	168	—	—	—
	Lumière, France																		
35	Radiofilm	Lumière Radios	10(*c*)	18	24	20	Low	11	1·70	16	33	53	75	100	127	159	190	223	—
	Manufacturas Fotograficas Españolas, Spain																		
36	Mafe Ultra Rapida	Ilford ID 42	5(*b*)	20	18	16	Very high	8	1·80	14	30	50	74	100	—	—	—	—	—
	Perutz, Germany																		
37	Type K	Ilford ID 42	10(*d*)	20	14	10	Medium	8	—	16	33	52	74	100	129	161	197	243	—
38	Type S	*idem*	5(*d*)	20	24	18	High	8	1·75	16	33	53	75	100	128	159	194	232	—
39	Type X	*idem*	10(*d*)	20	32	28	Low	8	2·25	18	37	57	78	100	123	147	173	201	231
	Schleussner, Germany																		
40	Doneo	Schleussner Adox	10(*c*)	19	24	24	Low	10	2·05	19	39	59	79	100	121	144	167	193	—
	Typon, Switzerland																		
41	Progress (Ultra Rapide)	Typon	5(*d*)	19	28	22	Medium	10	1·75	16	34	54	76	100	127	157	190	227	—

* Notes

(*a*) Developing time according to, or almost according to, the recommendations of the manufacturer.

(*b*) Developing time according to the recommendations of the manufacturer. An increase of the developing time to 10 min. did not increase the speeds by more than about 20%.

(*c*) Although a developing time of about 5 min. was recommended by the manufacturer, the results have been listed for 10 min. development, because at this development the speeds were 20% or more higher than at the recommended period.

(*d*) The recommended developing time was not known. Developments of 5 and 10 min. were tried. When the increase in the speeds was 20% or more, the results have been listed for a developing time of 10 min.

(*e*) The recommended developing time was not known, but likely to be longer than for the films belonging to group (*d*). Developments of 10 and 15 min. were tried. When the increase in the speeds was 20% or more, the results have been listed for a developing time of 15 min.

Note added in proof, 6 *May* 1956.—When the manuscript was submitted for publication, copies of it were also sent to the manufacturers to enable them to comment on the paper. We are very grateful for the answers received, of which the following deserve special attention:

1. *Messrs Agfa, Germany*, state that at the present time they are manufacturing films which are probably more suitable for X-ray crystallography than the one listed.

2. *Messrs Ceaverken, Sweden*, remark that it would have been preferable to list the *speed of fog increase* with storing time instead of the fog at a given age, because the exact date of manufacture is mostly not known to the user. The indications of the degree of fog, as given in the table, might therefore be misleading in some cases.

Messrs Ceaverken also suggest stating explicitly that in view of the different exposure conditions (shorter wavelengths; intensifying screens) the results listed are not valid for radiological applications of the films. The same remark was made by *Messrs Perutz, Germany*.

3. *Messrs Fuji, Japan*, write that in addition to the film listed they manufacture three other types of X-ray film, namely Industrial Type 400 (screen type), Industrial Type 80 (no screen type) and Medical (screen type). It appears from the data submitted by *Messrs Fuji* that these types of film are slower than their film Industrial Type 200 (no screen type) which has been included in the above investigation.

4. *Messrs Typon, Switzerland*, write that they have lately started the production of a new type of X-ray film ('Ferix') which is especially suitable for X-ray crystallography.

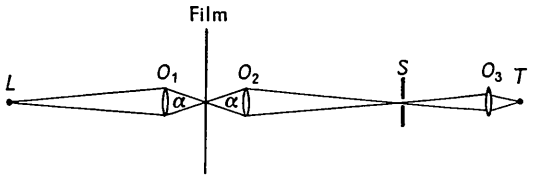

Fig. 2. Optics of densitometer.

on the film, the magnification being $\frac{1}{4}$. An identical objective lens O_2 gave a fourfold enlarged image of the illuminated film on a screen S; the angles α were 40°. A circular hole of 0·8 mm. diameter in the screen S determined on the film the area of which the transmittance was measured; this area thus amounted to $\pi(\frac{1}{4}\times 0{\cdot}4)^2 = 0{\cdot}03$ mm.^{2}. The light passing through the hole in the screen S was focused by an objective lens O_3 on a vacuum thermocouple, which was connected with a Zernike galvanometer. The readings of the galvanometer were perfectly proportional to the light intensity falling on the thermocouple. This was checked by interrupting a light beam of constant intensity by fast rotating sectors of various opening angles.

3. Results

The results have been summarized in Table 1. Although for each type of film only one set of data has been given, many more experiments were made. For some types of film different shipments were compared. The observed differences were not large enough to be considered significant, except for those in the 'film factor' (see under (*e*)). This factor in a few cases showed significant differences, which, however, did not exceed 10 %. Variations in developing time were tried on all films; on some, different developers were also used. For several films, measurements were repeated after a few months to examine possible changes of properties with age.

For many films the data presented in Table 1 are averages of the results obtained in two or more experiments at the same developing conditions. For each film the results have been listed for only one time of development, the choice of which has been explained in the footnotes of Table 1.

(*a*) *Homogeneity of the films*

The homogeneity of each type of film was tested by determining the root-mean-square deviation of the densities of the 35 equally exposed spots from their mean value. Generally speaking, the homogeneity of the films appeared to be satisfactory. The root-mean-square deviations were mostly less than 2%, but in some cases root-mean-square deviations up to 5% were found. Since the deviations observed were probably not characteristic for the type of film, they have not been listed in Table 1.

(*b*) *Relative speeds*

The film speed has been defined as the reciprocal of the X-ray exposure required to produce an effective density of 1·0. For each of the two kinds of radiation the speed of the most sensitive film was arbitrarily put equal to 100. From the results of different, comparable, experiments it was estimated that the standard deviation in the speed numbers of Table 1 amounts to less than 10%.

The film speeds increased with increasing development time. The increase was, however, quite different for the different types of film; for some films it was hardly perceptible, for others it amounted to even 50%, when the developing time was increased from 5 to 10 min. This made it somewhat difficult to decide for which developing time the results were to be listed (see the footnotes of Table 1).

(*c*) *Fog density*

In general the fog density D_F appeared to be higher for the fast films than for the slower ones. It increases with the developing time and with the age of the film. For fast films especially, the increase with age is quite considerable; it can amount to more than 0·05 per month when the films are stored at room temperature.

Because of their dependence on the age of the films, the fog densities are indicated only qualitatively in Table 1. The expressions of quality refer to films which had been stored in our laboratory at room temperature for about 2 months after being received. Although it may be expected that fresh films had been sent to us, the true age of the films was, of course, rather uncertain. The quality 'very low' indicates fog densities of less than about 0·05 and 0·10 at developing times of 5 and 10 min. respectively; 'very high' indicates fog densities higher than about 0·30 and 0·60 at similar developing times.

(*d*) *Granularity*

The granularity of the films was determined in the following way, making use of the middle ten of the thirty-five equally exposed circular spots. In each of these ten spots the density was measured at ten different points, and the root-mean-square deviation of the ten measurements from their mean value was calculated for each spot separately. The root-mean-square of these ten root-mean-square deviations was taken as representing the granularity G of the film. The values of $G\times 10^3$ are listed in Table 1. The film granularity thus defined probably represents as closely as possible the standard deviation of 100 density measurements from their mean value, irrespective of a possible long-range inhomogeneity of the film.

G depends on the scanning area, and is related to the Selwyn granularity G_S by the equation $G_S = G\sqrt{(2a)}$, in which a represents the scanning area (Selwyn, 1935).

As was mentioned in § 2(*c*), the scanning area used in our densitometer was 0·03 mm.^{2}.

The densities at which G was determined varied from 0·6 to 1·0 for the different films. Since G depends somewhat on the density, and may also depend on the age of the film, the values listed in Table 1 should only be taken as semi-quantitative indications. It is evident, however, that in general the granularity increases with the film speed, and that, when a scanning area of 0·03 mm.^{2} is used, the density fluctuations caused by the film grain are only 1–2 %, even for fast films.

(*e*) *Absorption of* Cu *radiation*

The 'film factor', F, by which the intensity of monochromatic X-rays is reduced when passing through the film, is of interest when the multiple-film technique is applied. This factor was measured by means of a Geiger counter for Cu radiation reflected by a monochromator crystal. The values of F are listed in Table 1 for the various types of film; the standard deviation in these values is approximately 2%. The data refer to films without envelopes or interleaving papers; the absorption of a sheet of paper amounts to about 5%.

The absorption of Mo radiation is much smaller than that of Cu radiation, and has not been measured. When using the multiple-film technique with Mo radiation, however, films of different speeds may be superimposed (Iball, 1954).

(*f*) *Characteristic curves*

Since for low densities the effective density $D_{\text{eff.}}$ is approximately proportional to the X-ray exposure E, $D_{\text{eff.}}$ was plotted against E, and not against $\log E$. The shapes of the characteristic curves are indicated in the last columns of Table 1, in which the relative exposures required to obtain effective densities of 0·2, 0·4, ..., 2·0 respectively are listed. For each film the exposures were scaled in such a way that the exposure corresponding with the effective density 1·0 became equal to 100. A perfectly linear relationship between $D_{\text{eff.}}$ and E is thus represented by the numbers 20, 40, ..., 200. An impression of the deviation from linearity can be obtained by looking at the numbers listed, although it may be preferable to plot them against the $D_{\text{eff.}}$ values. The accuracy of the numbers listed is different for the various effective densities, and is such that, for instance, the difference between the characteristic curves of the films 39 and 40 is at the limit of significance.

For practically all films it appeared that the $D_{\text{eff.}}$ versus E curve had the same shape for Cu and Mo radiation. Only in four cases was there an indication that the curve for Mo radiation was more nearly linear than that for Cu radiation. The numbers listed in Table 1 refer to Cu radiation.

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X-ray Study of the Allotropic Modifications of Calcium Metal

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It is shown by means of Debye–Scherrer photographs that for the metal calcium three allotropic modifications occur, namely α-Ca, cubic face centred; β-Ca, hexagonal close packed; and γ-Ca, cubic body centred, the transition points lying at about 250° C. and 450° C. The observed lattice constants of the three modifications are closely related to those of the analogous modifications of strontium.

1. Introduction

Earlier investigations concerning the structures of the allotropic modifications of calcium were carried out by Graf (1933, 1934); Ebert, Hartmann & Peisker (1933) and Bastien (1934, 1935). In his first paper Graf reports two modifications, a cubic face-centred (f.c.c.) and a cubic body-centred (b.c.c.) structure, stable respectively below and above 450° C. Ebert *et al.* also found two modifications, a f.c.c. structure stable below 450° C. and a hexagonal close packed (h.c.p.) structure stable above this temperature.

In his second paper Graf indicates that there are three modifications, the transition temperatures being 300° C. and 450° C. Again the α-modification, stable below 300° C., is f.c.c., whereas the γ-modification, stable above 450° C., is h.c.p. The structure of the β-phase, which is stable between these temperatures,