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Characteristics of Ion-Sensitive Emulsions for Mass Spectrography—II

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Characteristics of Ion-Sensitive Emulsions for Mass Spectrography—II

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Abstract

An earlier investigation of the properties of ion-sensitive plates has been extended to include additional types of spectrographic emulsions and to obtain more extensive data on the widely used Ilford Type Q2 emulsion. Log-probability response plots are used to compare the different emulsions and emulsion batches.

The effects of variations in exposure and processing conditions on the Type Q2 plates have been investigated. These effects are correlated, where possible, with the coating analyses and microstructure of the plate emulsion.

Introduction

Although sensitivity for the detection of kilovolt-energy-range ions is the most important property of the ion-sensitive plates generally used as detectors in spark-source solids mass spectrometers, the outgassing properties, plate uniformity, and other factors can profoundly influence the over-all usefulness of a type of plate as a mass spectrographic detector. In a previous paper,1) the author described methods for evaluating the sensitivity, contrast, pumpability, and coating characteristics of ion-sensitive emulsions. These methods have been used to evaluate several different emulsion number lots of the widely used Ilford Type Q2 plates and three other ion-sensitive products.

Post-S4325 Q2 Plates

Ilford Ltd. introduced an improvement in the Q2 emulsion as a running change at emulsion No. S4325.2) For the earlier work,1) only one sample lot (S4358) of the improved product was available. Six additional post-S4325 Q2 emulsions have now been evaluated. Three of these emulsion lots were the standard 2- by 15-inch plates normally used in the Consolidated Type 21-110 double-focusing spark-source mass spectrometer, and the other three were in an emergency shipment of shorter plates in the sizes (2 by 10 or 2 by 12 inches) normally used for the Associated Electrical Industries MS-7 mass spectrometers. Although Ilford now cuts the 2- by 15-inch Q2 plates by a procedure that eliminates the bar defects referred to by them as "coated edges",[3)] such a procedure has definitely not been used for the 2- by 10-inch plates. All 2- by 10-inch plates examined were from emulsion lot S4488, and all had pronounced end bars of the type previously reported in a study of plate uniformity.[4)] The uniform area of these plates was approximately 2 by 8.5 inches. The appearance of the uniform regions of all plates examined was good except for lot S4495, in which some plates were blotched with white opaque regions.

Examination of the coatings, Table I, has shown that the silver-gelatin ratio of the Q2 plates is distinctly different from that of conventional spectrographic types such as Kodak Type 1 or 103-a. The coating density varies from the light coat of S4460 plates to the heavy coat of lot S4399. After a pro-ration for plate area in the manner justified by earlier data,[1)] pumping characteristics of every lot were nearly identical. The microscopic appearances of all Q2 plates except lot S4409 were similar. The plates in lot S4409 exhibited an extreme uniformity of surface and a uniform grain distribution; the other Q2 emulsions showed a definite but limited surface irregularity. This difference between S4409 and the remainder of the Q2 plates may be distinguished in Figure 1; it is plainly evident in the original photomicrographs.

The response characteristics of Q2 plates processed in ID19 developer under standardized conditions, Table II, reveal the unexpected fact that the very uniform S4409 plates have undesirable properties. The 79 percent background transmittance for S4409 is much less acceptable than the 85 to 95 percent typical of the other Q2 emulsions. The definition of strong ion and photon images is visibly poorer on S4409 plates than on typical Q2 plates. Plates for mass spectrographic use do not have an antihalation backing, and the poor image definition of strong photon images results from halation, the exposure of the emulsion by scattered light from the primary image undergoing partial and total reflection at the second surface of the emulsion support.

Because ions and secondary light in the ultraviolet cannot penetrate the glass for reflection, the exposure induced in regions adjacent to the primary image of a strong ion exposure is not halation. But the extent of such induced exposures in Q2 plates does increase with the susceptibility of the plates to halation. Thus the intensity of halation about an image produced by a heavy exposure of visible light may be used as an empirical measure of image definition expected in mass spectrography.

In the microphotometer trace of a heavily exposed, processed image of a tungsten-light band on a Q2 plate, Figure 2, the secondary absorption maxima adjacent to the main band are halation effects. For a given difference in indexes of refraction across the second surface of the support, the separation of the secondary side bands is proportional to the thickness of the support. The secondary maxima are completely suppressed by backing the Q2 plate with a red lacquer, Figure 2. A glycerol-wetted piece of flat-black paper on the second surface also suppresses halation. Another wetting liquid can be used if it has a refractive index almost the same as or greater than that of glass. The emulsion structure affects the intensity of the secondary maxima by affecting the scattering of the light transmitted into the support. The response plot for the secondary maxima on logarithmic probability paper turns out to be nearly linear and parallel to the response plot of the main band, Figure 3. So long as a support medium of the same thickness and index of refraction and a light band of given width are used, the halation tendency of the emulsion may be measured by the ratio of exposures required in the main and secondary bands to attain the same level of absorption. To provide a simple calculation with the effects of small slope differences eliminated, the average of the exposure ratios at the relative absorption levels of 5, 10, and 20 percent was computed as an index of halation. The primary light band was 0.57 mm wide. On this basis, S4409 plates are clearly inferior to those prepared from other Q2 emulsions.

The ion responses of the various Q2 batches listed in Table II show considerable variation in the slope, or contrast term G,[1)] and in the level of absorption achieved with a saturation exposure of Fe^+ ions. The exposure necessary for 95 percent relative transmittance that corresponds fairly closely with the "just visible line" shows fair precision within lots. The ion response curves for different Q2 emulsions tend to pivot about a region near the 10 percent transmittance level, Figure 4. This may explain R. Brown's and the author's observations that the visual results from mass spectrographic plates usually compare favorably in accuracy and precision with data obtained from the plates by photometric means.[5)]

After 15 months' experience with post-S4325 Q2 plates, one is left with the impression that the emulsion change has not yielded as many benefits as was anticipated from the initial evaluations. The contrast G for ions in all lots except the sample S4358 and lots S4409 and S4460 is low enough to overlap the range of pre-S4325 plates. Although microporosity has been substantially eliminated from the Q2 plate, the saturation transmittance level for Fe^+ ion exposure rises to the 5 percent range in lots S4399, S4462, and S4488. A variation of saturation level with energy or charge level, similar to that reported for the Q1 plate,1) is also noticeable in these emulsion lots. None of this variability in saturation level appears to correlate with physical or chemical properties of the coating. The poor image definition in S4409 plates does appear to correlate with an overly smooth microscopic appearance of the emulsion.

The survey of the seven Q2 lots shows that emulsion calibration data must be obtained for every emulsion lot if photometric methods are to be used for calculation of analytical data from the plates. The consistency observed within a lot indicates that a common calibration plot for plates of each lot is sufficient for routine work over two or three weeks. Over longer periods or for special investigations, additional calibration plots should be prepared.

Processing of Q2 Plates

The 2.5 min development in ID19, followed by 2.5 min of rapid fixing at 25 C were selected from a limited survey of processing conditions with four developers.1) Further investigation of processing conditions, Table III, confirms that the 2.5 min development time gives good sensitivity with low processing fog. The ID19 developer permits considerable latitude in choice of 25 C processing times.

A few experiments with ID19 at 28.7 C again showed good latitude, but were discontinued because of severe staining. Microdol X, a developer suggested in a mass spectrometer newsletter,6) was tested. In addition to severe staining of Q2 plates, developer depletion evidently caused erratic results. All work with Microdol X was discontinued as soon as the consistency and stain-free properties of the sulfite-free developer suggested by Kennicott7) were ascertained. For Type Q2 plates processed in the sulfite-free developer, the response data are nearly linear when plotted on logarithmic probability paper. The contrast G is almost the same as that obtained with ID19 developer. The development background is substantially less than the background obtained with ID19 developer, but this feature is attained at the price of a lessened sensitivity. Although the data of Table III show that it takes about twice the exposure to produce the same relative transmittance value on a Q2 plate developed in sulfite-free developer as on a plate processed in ID19, the noise level on the microphotometer traces of the former plates is about half that encountered with traces of the latter. Accordingly the detectability of weak lines with sulfite-free developer is at least as good as it is with ID19.

The sulfite-free developer also decreases the intensity of the fog patches that lie on the high-mass side of very intense lines and, that probably result from secondary particles generated at the position of these lines. It is therefore superior to ID19 for study of lines located in these fog patches. The photometer traces for the same iron exposure after processing Q2 plates in ID19 and sulfite-free developer, Figure 5, show clearly that the background, noise, and fog patch near the Co^{59+} and Ni^{60+} lines are lower on the plate developed in sulfite-free developer. The three-way improvement of background, noise, and fog patch clearly more than offsets the loss in development sensitivity; accordingly the author intends to use the sulfite-free developer for analytical determinations of cobalt and nickel in high-purity iron. Although the sulfite-free developer offers a distinct advantage in the determination of nickel and cobalt, and also in reduction of the Fe^{+} background at the Mn^{55+} line position for general plate processing, the author intends to continue as before with a 2.5 min development at 25 C in the more sensitive ID19.

To compare the developed-grain characteristics, light-exposed areas of similar image transmittance on plates processed by different techniques were photomicrographed, Figure 6. Although there is a suggestion that fine grain developers such as

Microdol X may produce less well consolidated silver aggregates, differences in developed texture are barely apparent and not nearly as prominent as the differences in unprocessed emulsions, Figure 1.

Evaluation of Emission Spectrographic Plates

Dempster's mass spectrometer group at Chicago used Kodak Type 111 plates as ion detectors[8,9] when Q plates were virtually unobtainable.[10] Subsequent work by Schönheit[11] on Perutz plates supports the Hayden and Lewis observation[9] that ultraviolet-sensitized Type III plates are about ten times more sensitive to ions than the unsensitized plates. Rudloff has recently studied SWR film and a composite detector consisting of a highly sensitive phosphor and rapid X-ray film.[12] Although he did not directly study the ion response of ultraviolet-sensitized SWR film, he suggested that ultraviolet sensitization offered advantages only for protons and other ions of very light mass. Because the most ion-sensitive member in a series of similar emulsions is almost certainly also going to be the one with the largest grains and greatest sensitivity to light, the author evaluated Kodak Types 1 and 103a spectroscopic plates. Type 1 plates are about 10 times as sensitive to tungsten light as Type III plates, and Type 103a is a very fast finer-grain-class plate with a sensitivity close to that of Type 1.[13]

Coating and pumpability data were determined for the Type 1 and 103a plates, nonsensitized or "Class O" sensitivity, Table I and Figure 7. The large grains of the Type 1 emulsion can be clearly distinguished in Figure 1. The outgassing characteristics of Type 1 and 103a plates are unsatisfactory compared to those of Type Q2, and necessitate long pumping times prior to the determination of ion-response data. After it was ascertained that the slightly less light sensitive 103a plate had about the same sensitivity to ions as the Type 1, the experiments on ultraviolet sensitization of spectroscopic plates were confined to Type 1 plates.

Because Kodak discontinued factory supply of ultraviolet-sensitized spectroscopic plates in March 1961, the user must now sensitize the plates as required prior to use.14) A solution of "Ultraviolet Sensitizer No. 2" (Eastman Cat. No. 8269) may be applied to Type 1 plates by swab coating in the manner recommended by Kodak,15) but the author chose to shift from the highly flammable cyclohexane to a nonflammable, low toxicity degreasing solvent consisting mainly of 1,1,1-trichloroethane (Dow Chemical "Chlorothene NU"). Type 1 plates were also successfully sensitized with the Ultraviolet Sensitizing Solution (Cat. No. 3177) by swab coating followed by a subsequent rubdown of the emulsion surface with a stiff rubber wiper to distribute the fluorescent oil droplets; this solution is not recommended by the author or by Kodak for coating of spectroscopic plates. The Sensitizer No. 2 was used to sensitize Q2 plates, which had to be dip-coated because the emulsion is too fragile to stand up satisfactorily to swab coating. The sensitizer was removed from the glass rear surface of dip-coated Q2 plates by swabbing with excess solvent before the plates were finally dried for use.

The pumping characteristics of the sensitized plates were influenced by the thoroughness of solvent removal during drying. A well-dried sensitizing coat made very little difference to the pressure-time curves for Type 1 plates but substantially altered the curves for Q2 plates, Figure 7. Ultraviolet sensitization increases the ion-detection sensitivity of the Type 1 emulsion about one hundredfold, Table IV. The sensitizer acts as an ion-to-light energy transducer for the Type 1 emulsion. The net result is high contrast, negligible image transmittance at saturation, and response plots that are fairly linear on logarithmic probability paper, with more deviation at high exposure than is encountered with unsensitized Q2 emulsions.

Dip coating of the Q2 emulsion may be the indirect cause of a substantial reduction in ion-detection sensitivity.

The sensitizing layer may act as a desensitizing layer because it is so thick that it shields the emulsion from ions and partly from fluorescent ultraviolet light as well. The sensitizer-coated Q2 plates are not only less sensitive than uncoated ones, but are also less sensitive than sensitized Type 1 plates. These results confirm not only the correctness of the work by Hayden and Lewis[9)] on Type 111 plates but also Rudloff's opinion that ultraviolet sensitization is of little practical use as a technique for producing an ion sensitivity greater than that found in SWR emulsions.

Centrifuged-Emulsion Film, Type SC.5

Type SC.5 film for spectrographic work in the far ultraviolet is one of three C-type or centrifuged-emulsion films made experimentally in the Paris research laboratories of Kodak-Pathe. It is distributed on a limited commercial basis in the United States by Eastman Kodak, and is used extensively in rocket-borne spectrographs for study of the ultraviolet region of the solar spectrum. The ultraviolet sensitivity of the SC.5 film above 170 Å is reported to be about eight times that of the DC.3, another centrifuged emulsion with a sensitivity close to that of SWR.[16)] Because of the excellent ion sensitivity of SWR plates,[1)] a series of ion exposure tests were made on SC.5 film. They showed an extremely high sensitivity to ions.

The grains of the SC.5 emulsion are spherical and stand out clearly under the microscope. A wide range of grain diameters was present in the emulsions examined. Centrifuging improves the accessibility of the grains to ultraviolet light and ions by reducing gelatin shielding. The pumping curve for SC.5 film in Figure 7 shows that the cellulose acetate base of the emulsion is gassy. The outgassing of the base is accompanied by embrittlement and distortion, and on more than one occasion one edge of an SC.5 film actually sprang out of the 35-mm adapter used in the mass spectrometer plate holder. In spite of the handling difficulties occasioned by the acetate base, SC.5 film detected Fe^{54+}, Fe^{57+}, and Fe^{58+} ions in quantities between 1/5 and 1/20 of those just detectable with Q2 plates. The iron-ion-response plots on logarithmic probability paper are nearly linear. An interesting variation in the mass spectral background was noted with the SC.5 film; the secondary fog patch on the high-mass side of the intense Fe^{56+} line shifted towards higher mass compared with its position on Q2 plates. The Co^{59+} and Ni^{60+} lines are free of interference from this patch on SC.5 film, but the Cu^{63+} and Cu^{65+} lines are affected.

The variation in sensitivity and contrast (G) cannot be assigned to the emulsion, because handling problems caused by the acetate base may be producing part of the variation. Results on SC.5 film verify the intuitive reasoning[1)] on the possibility of an emulsion with a sensitivity to ions about ten times greater than those of Q2 and SWR emulsions. The desire for such ion-sensitive emulsions is long standing; Aston presented his ideas on the subject in 1933.[17)] The increased ion sensitivity of the SC.5 emulsion is useful to the researcher only for special problems because the objectionable outgassing and distortion of the cellulose acetate support preclude routine use. The SC.5 emulsion on a dimensionally stable polyester or glass support would offer the possibilities of both faster routine analysis and analytical accessibility of concentration levels about one-tenth of those now attained with Q2 plates. This means accessibility below the part per billion level for those spark-source mass spectrometers now working in the 1 to 10 part per billion range.

Summary and Conclusions

Several emulsion lots of Ilford Q2 plates and development procedures for these plates have been evaluated. Image definition was significantly poorer on the particularly uniform S4409 emulsion lot of Q2 plates. Ultraviolet sensitization greatly decreased the ion sensitivity of the Q2 plate, and although it greatly increased the ion sensitivity of the Type 1 plate it did not bring the sensitivity of the Type 1 plate up to the level of the unsensitized Q2 plate. Further work on ultraviolet sensitization is unlikely to yield a detector more sensitive to ions than the unsensitized Q2 plates. The very high ion sensitivity of SC.5 film shows that the centrifuged emulsions may become useful as ion detectors with sensitivities greater than those of Q plates.

None of the emulsion-processing combinations studied meet all the qualifications for ideal ion-sensitive plates suggested previously.1) The Q2 plate developed in ID 19 developer is the best all-round compromise of detector properties for routine mass-spectrographic applications. In special situations the researcher may gain advantages through low-noise development of Q2 plates with a sulfite-free developer, or from the very highly sensitive ion detection provided by SC.5 film.

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Table I

Coating Properties of Ion-Sensitive Plates and Films

Identification	Size, in.	Coating, mg/cm^2		Ratio Ag/Tot	Thickness, μ, Calc	Microscopic Structure	
		Total	Silver			Grain Diameter, μ	Appearance
Q2 S4358	2 x 15	0.53	0.19	0.36	2.0	0.8	Almost uniform
S4399	2 x 15	0.63	0.24	0.38	2.3		Almost uniform
S4409	2 x 15	0.52	0.19	0.37	2.0		Uniform
S4460	2 x 12	0.47	0.17	0.36	1.7		Almost uniform
S4462	2 x 12	0.54	0.19	0.35	2.2		Almost uniform
S4488	2 x 10	0.58	0.18	0.31	2.7		Almost uniform
S4495	2 x 15	0.53	0.19	0.36	2.1		Almost uniform
103a-0 ClB2	4 x 10	3.9	0.79	0.20	22	1.0	Uniform
1-0 ClL4	2 x 10	4.8	1.1	0.21	25	3	Clustered, voids in top layer
SC.5 101264	1.35 x 7.1*	2.0	0.32	0.16	12	0.8	Densely packed, wide range of diameters

* Supplied as a film, dimensions 35 x 180 in mm.

Table II

Response Characteristics of Post-S4325 Type Q2 Plates

Emulsion No.	Absolute Percent Transmittance			Exposure for 95 Percent Relative Transmittance		Contrast G, transforms/cycle		Index of Halation
	of Typical Background	at Saturation for Photon	at Saturation for Fe$^+$ Ion	Seconds of Photons	Picocoulombs of Fe^{56+}	Photons	Fe$^+$ Ion	
S4358	89	0.2	2.0	0.21	0.010	1.50	1.33	0.022
S4399	90	1.7*	5.2	0.12*	0.0104	1.49*	0.89	
S4409	79	0.4	0.8	0.10	0.0051	1.73	1.22	0.031
S4460	93	1.3	2.6	0.18	0.0166	1.38	1.28	0.022
	90	1.0	2.8		0.0091		1.23	
S4462	92	1.2	5.6	0.15	0.0079	1.22	0.97	0.024
	87	0.9	5.4		0.0127		1.04	
S4488	95	1.4	5.1	0.22	0.0110	1.22	0.99	0.014
	92							
S4495	91	0.6	2.9	0.13	0.0105	1.17	1.03	0.017
	89	1.2	2.3	0.15	0.0090		1.02	
	87		3.2		0.0080		0.86	

* Data for Microdol X developer, data for ID19 not obtained.

Table III

Processing Properties of S4495 Q2 Plates at 25 C

Developer Type	Minutes in		Transmittance % Absolute at		Exposure for 95% Relative Transmittance	Contrast G, transforms per cycle
	Developer	Fixer	Background	Saturation		
Tungsten Light (Exposure unit, seconds)						
ID19	1.5	2.5	96	1.1	0.132	1.43
	2.0	2.5	94	1.3	0.113	1.42
	2.5	2.5	94	1.2	0.119	1.41
	3.2	2.5	91	1.0	0.095	1.39
	6.0	2.5	86	1.0	0.099	1.44
	10.0	2.5	80	0.6	0.079	1.52
ID19*	2.5	1.5	90	1.1	0.117	1.36
	2.5	2.5	88	0.7	0.110	1.38
	2.5	4.0	89	0.9	0.117	1.54
Sulfite-free	3.2	2.5	96	1.6	0.225	1.52
	5.5	2.5	94	1.6	0.209	1.52
Ion Exposure (Exposure unit, picocoulombs of Fe^{56+})						
ID19	1.5	2.5	95	3.6	0.013	0.99
	2.5	2.5	89	2.3	0.009	1.02
	6.0	2.5	84	1.2	0.013	1.15
Sulfite-free	3.2	2.5	95	4.7	0.022	0.90
	3.2	2.5	96	7.7	0.025	0.88

* A fresh ID19 solution was used for the second series of tungsten-light exposures.

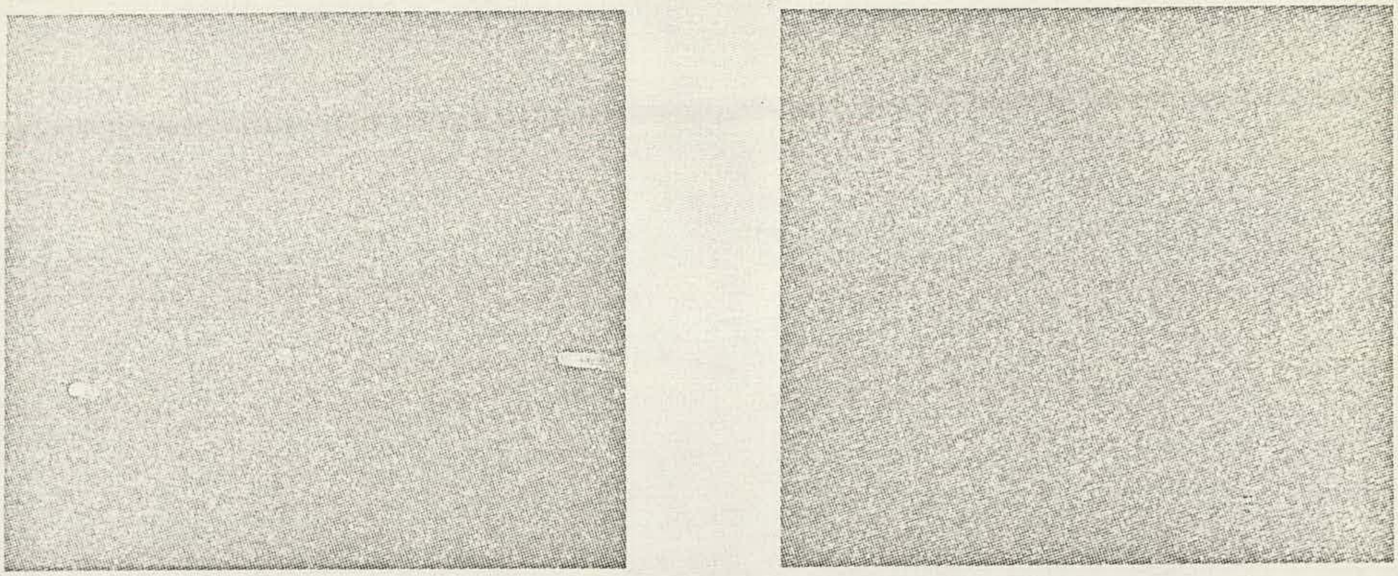
Table IV

Response Characteristics of Various Ion-Sensitive Emulsions

Emulsion		Emulsion Transmittance % Absolute for		Picocoulombs of Fe^{56+} for 95% Relative Transmittance	Contrast G, transforms per cycle	Sensitizing Material and Method
Type	Number	Background	Fe^+ Saturation			
Q2	S4495	88	2.8	0.0090	1.00	None
	S4495	86	0.3	7.8	1.69	Cat. No. 8269, dip
1-0	10C1L4	75	0.4	52	1.80	None
	10C1L4	77	0.1	0.65	2.44	Cat. No. 3177, swab-draw
	10C1L4	83	0.3	0.65	2.07	Cat. No. 3177, swab-draw
	10C1L4	79	0.3	0.9	1.70	Cat. No. 8269, swab
103a-0	103A0C1B2	48	0.5	59	1.20	None
SC.5	101264	91	1.3	0.0036	1.06	None
	101264	57	0.2	0.0007	1.25	None*
	101264	83	1.1	0.0006	1.51	None*
	101264	58	0.2	0.0019	1.12	None**

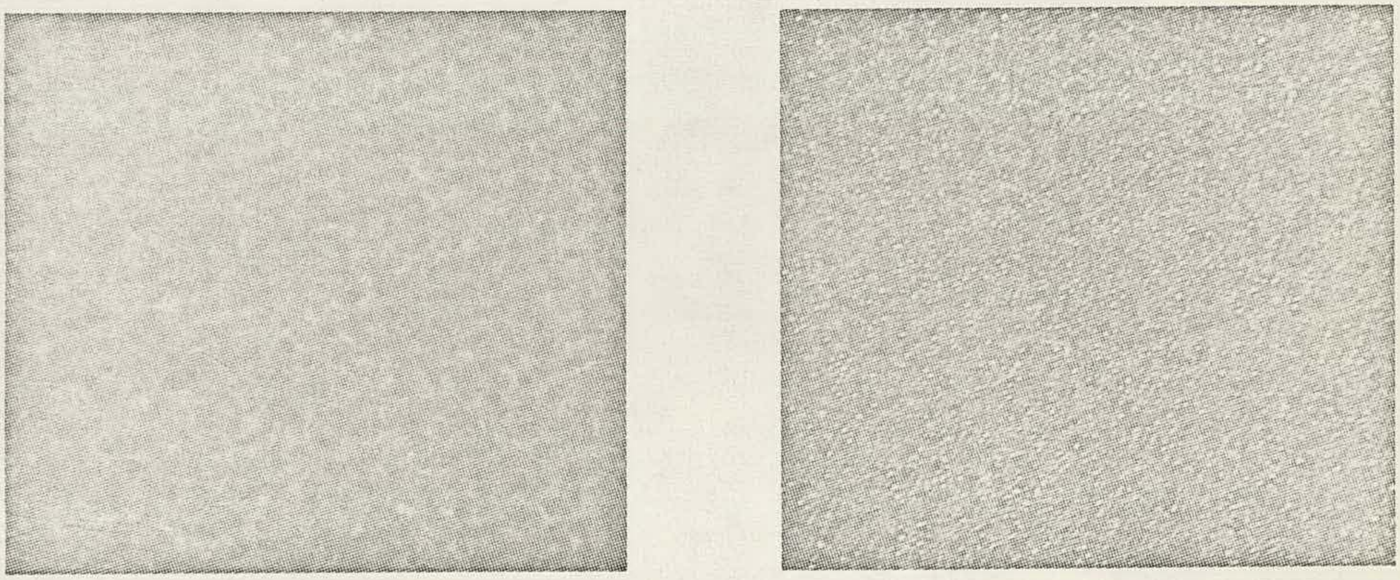
<u>Processing</u>: ID19 in processor, Q2, 2.5 min; I-0, 3.0 min; 103a-0, 3.2 min; SC.5, 1.9 min or * sulfite-free developer 2 min or ** 12 min sulfite-free, stagnant.

<u>Fixing</u>: Q2, 2.5 min; I and 103a, 8 min; SC.5, 1.5 min.



Type Q2 No S4488
(transmitted light)

Type Q2 No S4409
(transmitted light)

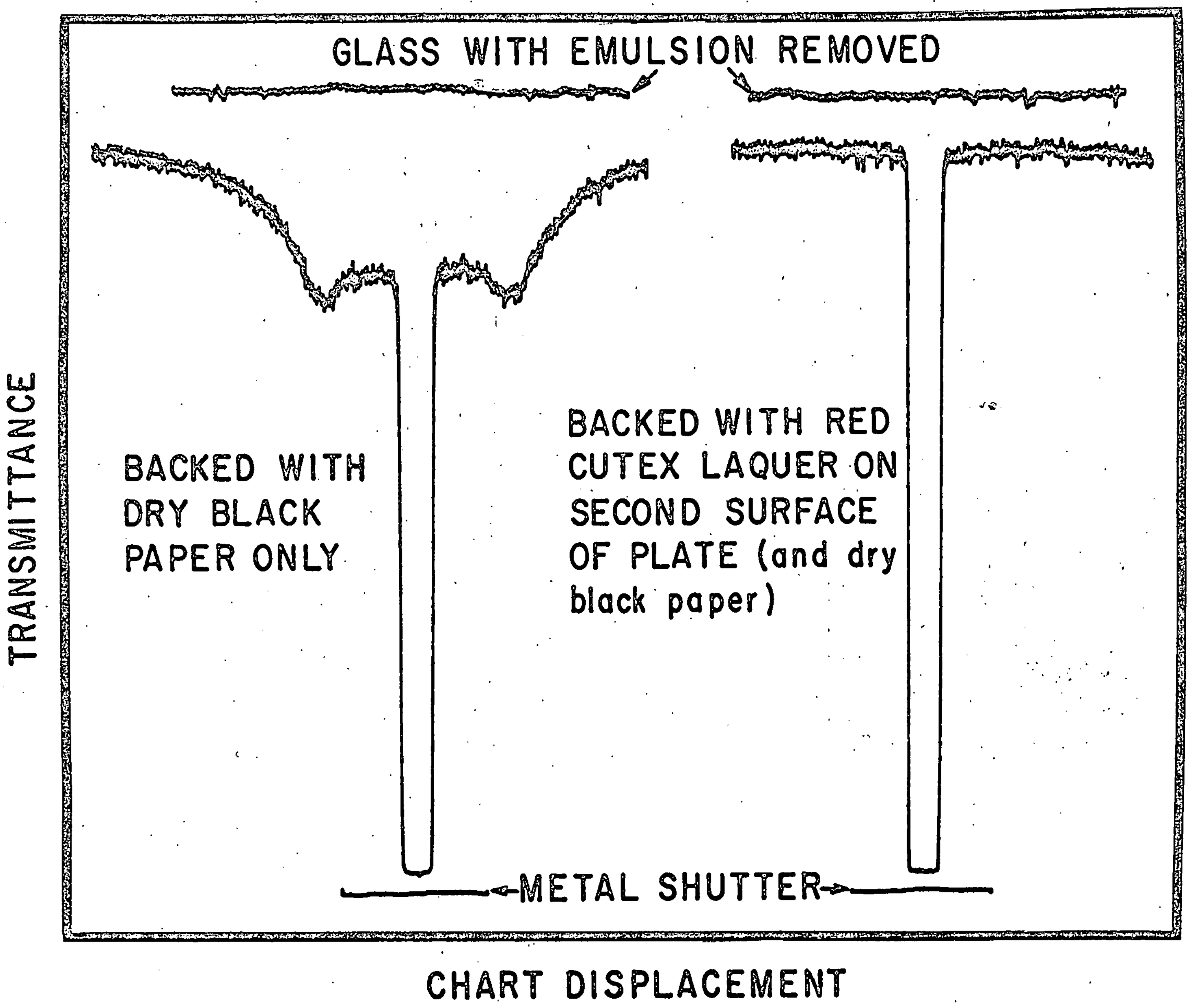


Type I-0 No 10C1L4
(reflected light)

Type SC.5 Film No 101264
(transmitted light)

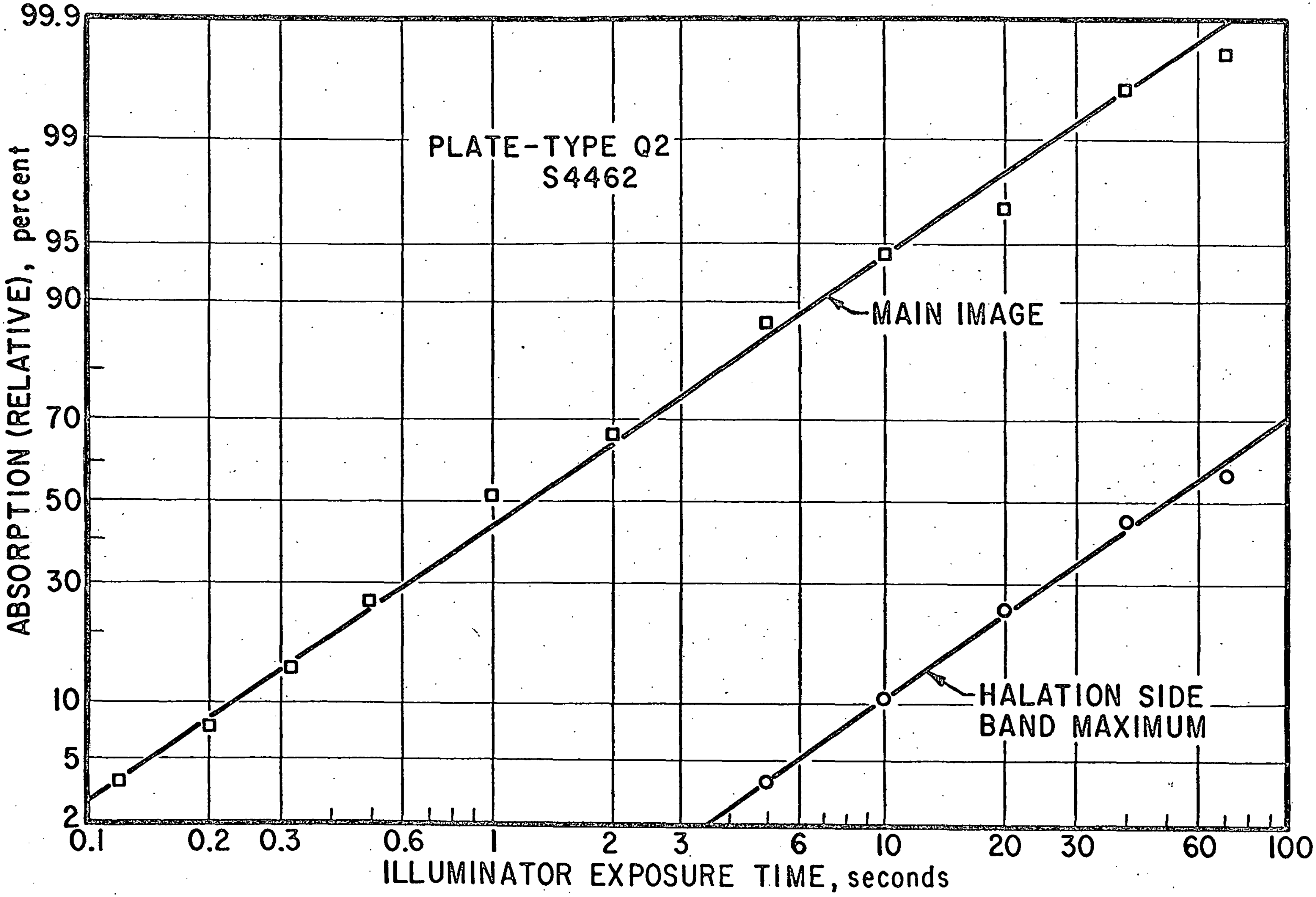
Photomicrographs of Undeveloped Emulsion Surfaces,
Magnification X500

Figure 1



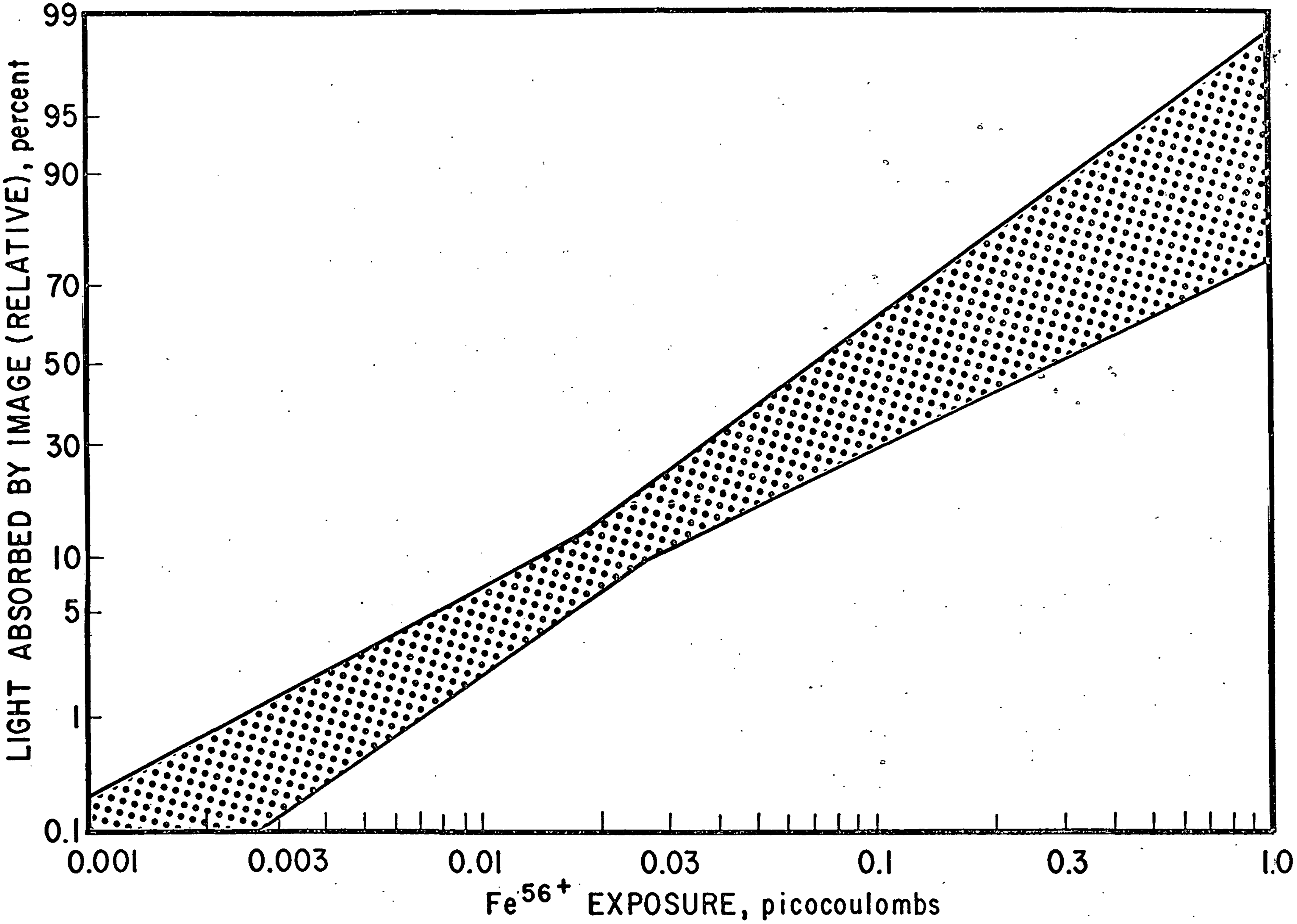
EFFECT OF LOCAL ANTIHALATION BACKING, Q2 PLATE

FIGURE NO. 2



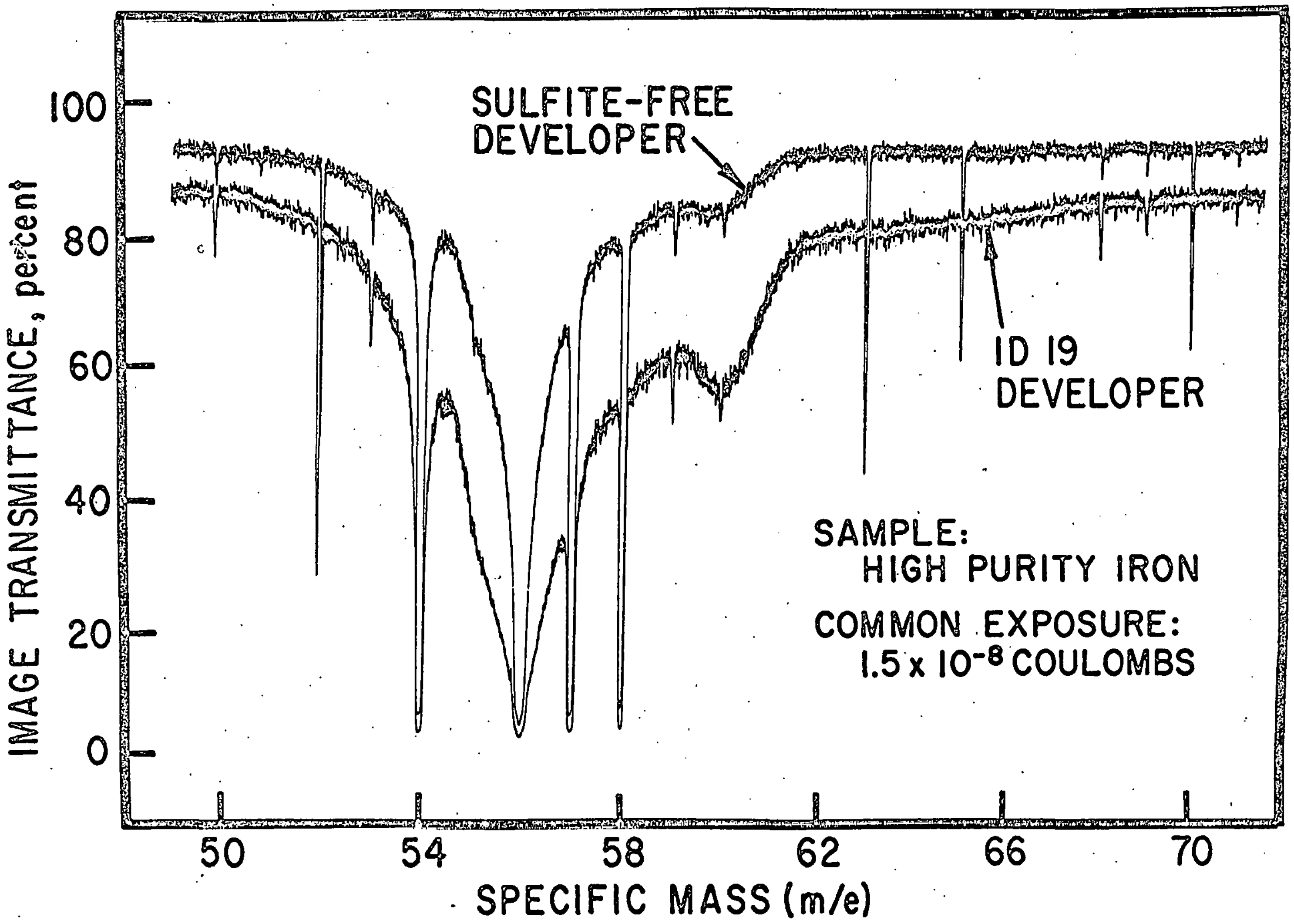
PHOTON RESPONSE OF MAIN IMAGE AND HALATION SIDE BAND

FIGURE NO. 3



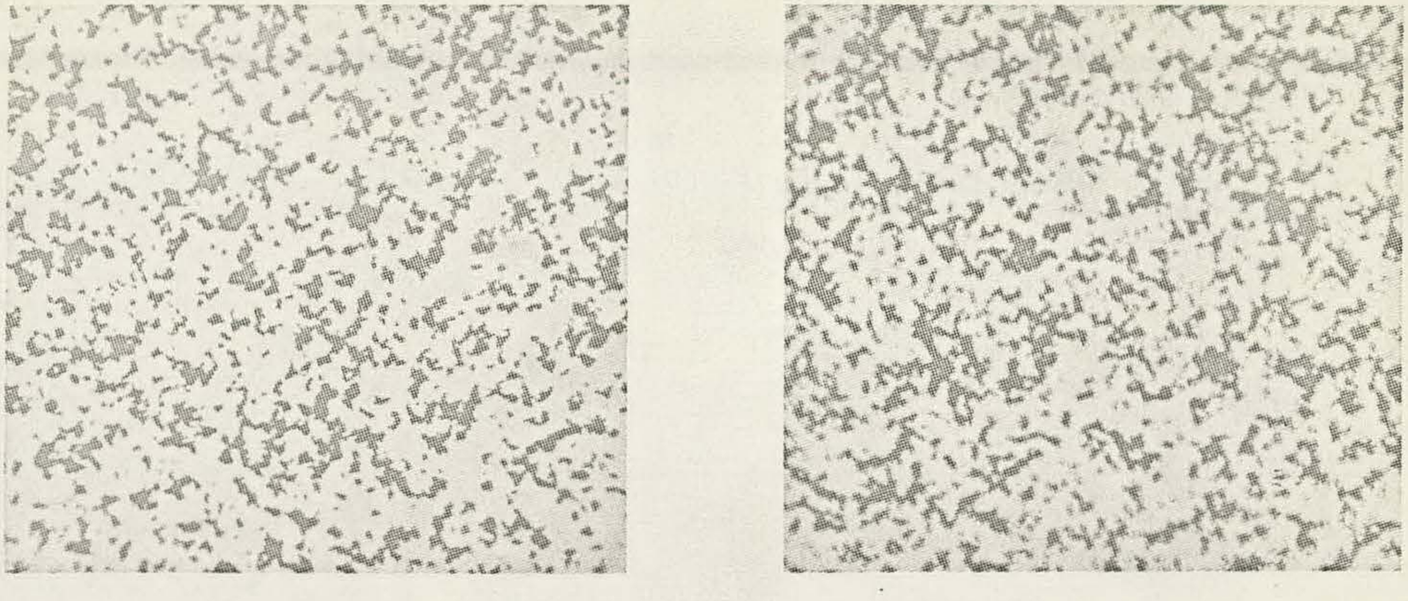
BAND CONTAINING RESPONSE LINES FOR POST-S4358 Q2 PLATES

FIGURE NO. 4



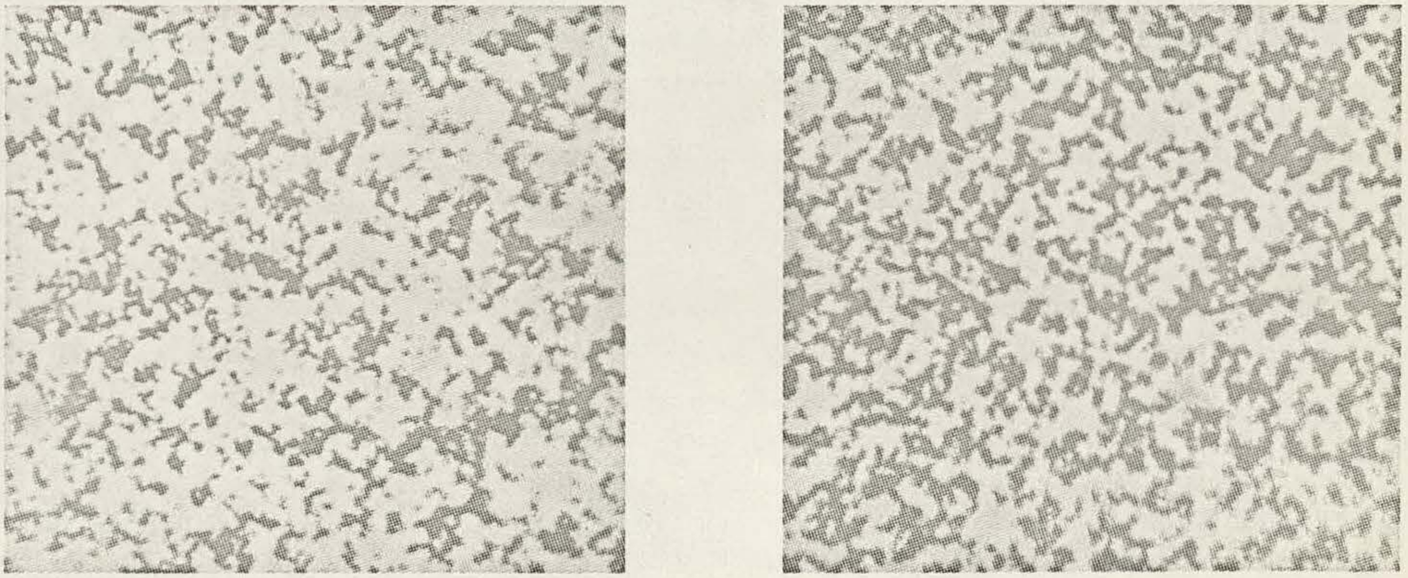
EFFECT OF DEVELOPER ON Q2 PLATE TRANSMITTANCE

FIGURE NO. 5



S4460, developed 2.5 min in ID19
(24% relative image transmittance)

S4495, developed 2.5 min in ID19
(36% relative image transmittance)

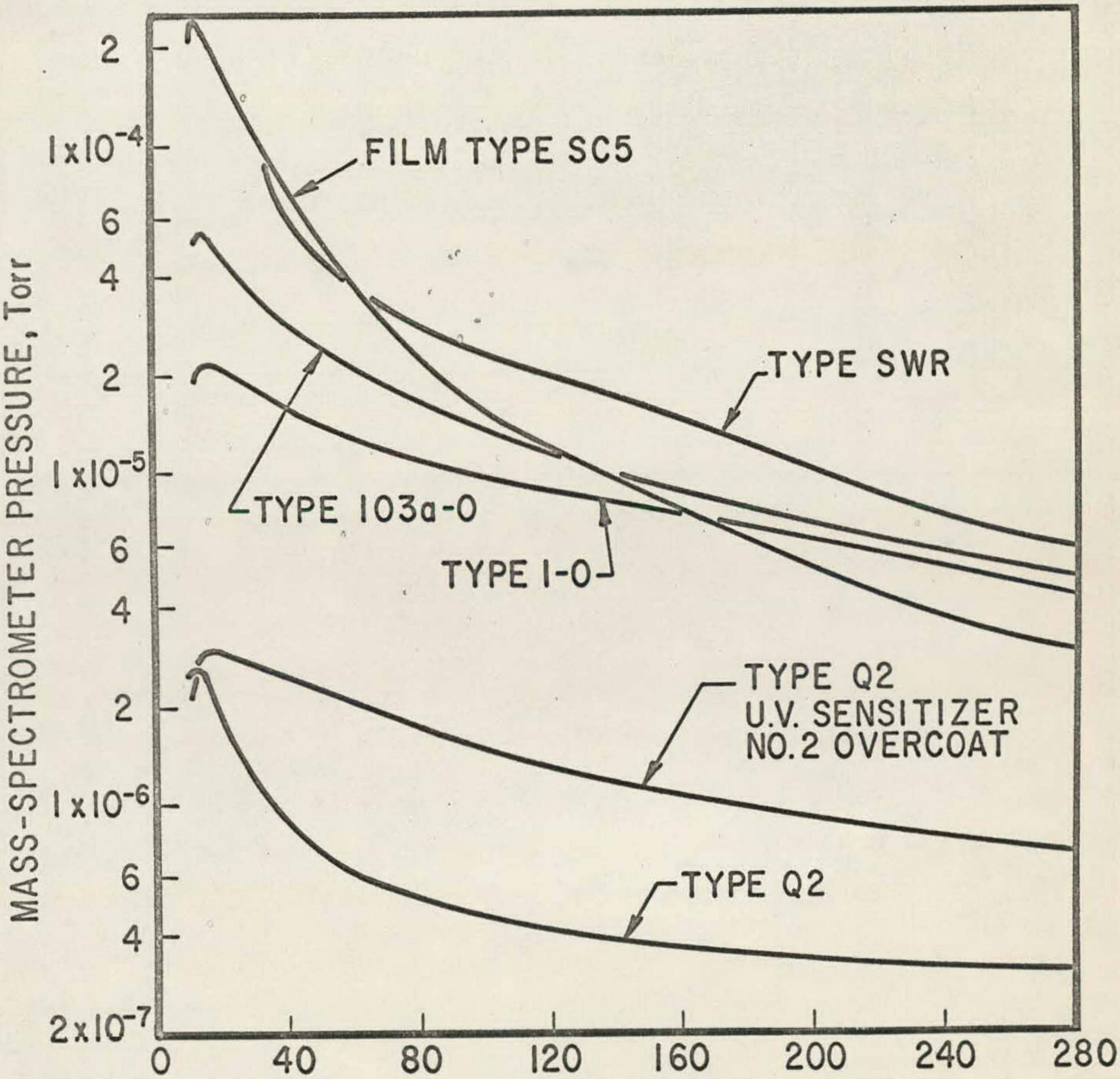


S4495, developed 4.5 min in ID19
(24% relative image transmittance)

S4409, developed 12 min in
Microdol X
(34% relative image transmittance)

Photomicrographs of Exposed and Developed Emulsion
Surfaces of Q2 Plates, Magnification X500

Figure 6



PRESSURE VERSUS PUMPING TIME FOR 2-BY 15-IN. PLATES

FIGURE NO. 7