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PROJECT 325B

SUMMARY REPORT

PERIOD: 1 February 1972 to 29 February 1972

Submitted By: [redacted]
Project Manager

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PROJECT 325B

SUMMARY REPORT

PERIOD: 1 February 1972 to 29 February 1972

Submitted By: [redacted]

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Project Manager

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SUMMARY

The project work for February continued; bearing on shelf life/speed decay. The air sampling program was completed. A complete analysis of the mass of data has not shown a correlation with either "blotch" film samples or low photographic speed samples. However, the experiments showing comparisons between the Chemical R and D control labs and the engineering labs show correlation between these effects and some airborne contaminant. The pure air purging of the engineering room produced good film samples during a three hour run. The new isolated engineering darkroom is forecast for completion by 15 March. If good films are produced here, the engineering work on reactivation and overcoating will be carried out during the last two weeks of March. This effort has slipped one month.

The six HID-2 red light development units were completed. One unit was shipped to Perkin Elmer. The last two film shipments to Perkin Elmer have been delayed in transit for periods up to five days, in both cases the dry ice was depleted and the films useless for red light development. It may be advisable to ship direct to Danbury by air charter.

Considerable work was done on qualifying the Quantametric automatic plotting densitometer as a project standard. Cross over comparisons on the present densitometer filters and the status "A" type on this device are being carried out. Some problems with using the small project film samples has been experienced. This unit is forecast to become the project standard by the end of March.

CHEMICAL R and D

Overview

The problem of environmental contamination, and the resultant failure to obtain satisfactory films, continues. The contaminant has been shown to be airborne, but it is not known whether it is gaseous or particulate. This problem has restricted film evaluation work to two men and one darkroom, the one which has been environmentally controlled. The adjacent dark room has been sealed and shown capable of decontamination. As soon as additional Barnebey Cheney air purifiers arrive, this room should become available for work. This is expected in the second week in March.

Air sampling analyses last month showed a positive correlation between the contamination and chemical oxidants as a class. This month's analyses have failed to support this initial correlation, or provide any new correlation. The air sampling program has therefore been discontinued. The current apporach is to secure uncontaminated work areas so that all work can proceed.

The contamination problem has hindered Chemical R and D's film evaluation work, but not seriously. On the other hand, delays in hiring the two urgently needed organic chemists has affected nearly all aspects of the materials programs to greater or lesser extent. [redacted] 25X1 (M.S.) have just accepted the positions and begin work in early March. Chemical R and D will, therefore, be in full progress by the latter part of March.

Because of the manpower shortage several work areas have fallen behind schedule or have been postponed:

1.1 Leuco Dye Program: The model studies, particularly the purification and testing of leuco malachite green analogues has fallen behind schedule. The situation is not particularly serious, however, and work is again expected to proceed on schedule by the latter part of March.

1.2 CBr_4 - Activator Program: The program to study the deterioration of solid CBr_4 is underway and proceeding on schedule.

1.3 N-Oxide Program: Phase I has been completed. No further work is scheduled until the results have been thoroughly evaluated.

1.4 Binder Studies: No work was scheduled for February. The effort in March will be increased to compensate for delays in the Environmental Studies scheduled to begin in March.

1.5 Environmental Studies: It now appears that the plan to begin purifying all materials, and to formulate and coat film in purified atmospheres will be delayed from March to April. This situation has arisen from delays

in acquiring the necessary LabConco dry boxes. Supply has been limited, and two recently acquired boxes were defective and had to be returned. Current estimates for securing the necessary equipment and for setup, project the new starting date to the beginning of April. The time originally alloted to March will be used to increase our effort on binder studies ahead of schedule and to catch up in other areas; such as the leuco dye program and film analyses studies.

1.6 Other Film Systems: This work was scheduled to begin in early March. It will be delayed to the latter half of March when another darkroom is expected to be usable.

1.7 Film Analyses of Decay Products: Shortage of manpower has delayed this work. Our latest research results (Section 1.1.1.3) necessitates our proving or disproving that 4DMAP is formed during decay. Film analysis work will, therefore, begin during March and as soon as the new hires begin work.

Month's Results

Large batches of TMB-free D260 will begin arriving from Chemical Samples Company in early March.

The alternate synthesis of D260 is underway but has been hindered due to manpower shortage.

D260 has been demonstrated to be the major culprit in rapid speed decay. It is not known, however, if it is the sole cause or if it is due to specific interaction(s) with one or more of the other ingredients.

4-Dimethylamino phenol (4DMAP) has been shown to be extremely deleterious to photoresponse. It is a possible degradation product of D260 hydroperoxide which is suspected of being formed during decay.

The CBr_4 deterioration studies are underway.

New supply of CBr_4 (Berk Ltd.) has been found. It is manufactured in France and appears quite pure. Surprisingly, it has afforded excellent films without any purification.

The N-oxide program: Phase I has been completed. Results are being evaluated.

1.0 CHEMICAL R and D

1.1 Leuco Dye Program

1.1.1 D260

Chemical Samples Company has promised to deliver about 700 g of D260 in early March. This material will be free of tetramethylbenzidine (TMB) by TLC, whose detection limit is about 50 ppm. Large scale purification of D260 will then be undertaken. Our supply of photograde D260 remains adequate for our on-going film studies.

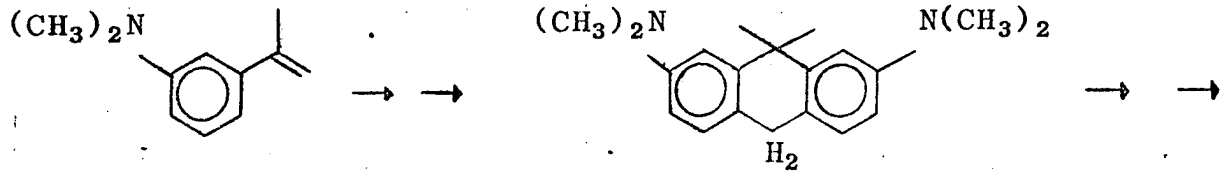
A number of samples of D260 were sent to Gollob Analytical Services for mass spectral analyses. Results of these analyses are not yet known.

During February a fluorimeter was obtained on a trial basis, and was used to study the purity of D260 with respect to TMB. The results indicate that apparently as little as 200 ppb (parts per billion) TMB can be detected by this method. This value is only approximate, however, since the level of TMB in the control sample of D260 was known only to be less than 50 ppm, the current limit of sensitivity by TLC. D260 which is totally free of TMB will be available from the new synthetic route and will therefore serve as an absolute control standard for the TMB determination.

Arapahoe Chemical (Boulder, Colorado) has agreed to prepare 5 kg of D260 by the procedure currently being used by Chemical Samples Company, and eight thousand (8,000) dollars has been provided for this. However, the order for this additional D260 will be postponed until the new synthetic route can be evaluated. Should this new route prove feasible and the D260 of superior quality (because of different or fewer impurities as discussed in last month's report), then money will be available for securing an adequate supply. This new route also affords the means of preparing analogues of D260 by way of the intermediate anthrone.

1.1.1.1 Alternate Synthesis of D260 - The Anthrone Route

The details of this alternate route to D260 were shown in last month's report. For this month's discussion, only selected intermediates will be pictured:

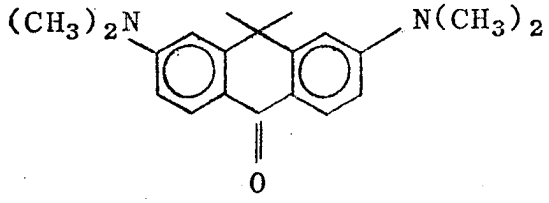


m-isopropenyl-N,N-dimethylaniline

(Step 2)

D263

(Step 4)



anthrone

(Step 6)

D260

(Step 9)

The olefin, m-isopropenyl-N,N-dimethylaniline, is a common intermediate to both the current route and the anthrone route. The first attempt at the anthrone route[1], in January and February, resulted in crude D263 in 50% yield from the olefin. This was reduced to 13% yield by two unfortunate recrystallizations from ethanol. Further work led to 1.9 g of anthrone, mp 205°C, a 2% overall yield from olefin.

The low yield has necessitated a rerun which is currently underway. Alternatives to the ethanol recrystallization and the chloranil oxidation will be investigated.

Optimum yields of D260 by this alternate route have been estimated and are compared with the standard route:

Intermediate	Anthrone Route Optimum Moles	Standard Route Moles
m-isopropenyldimethylaniline	1.00	1.00
2-isopropenyl-4,4'-bis(dimethylamino)diphenylmethane	0.76	—

(continued)

Intermediate	Anthrone Route Optimum Moles	Standard Route Moles
D263	0.44	-
anthrone	0.32	-
D260 carbinol	0.28	-
D392 (D260 dye)	0.25	-
crude D260	0.22	0.48
rex'd D260	0.20	0.24

The anthrone route is expected to yield less D260 than the current method. At this time, however, this fact is not critical especially if it settles questions concerning the effects of TMB and other impurities. The anthrone route is being followed according to the procedure of Aaron and Barker[1]. Many improvements of their procedure can be readily envisioned, should it become necessary to ultimately prepare D260 by this route.

The last step in the Anthrone route to D260 is the reduction of the dye to its leuco form. To confirm the feasibility of this step, a small sample of D391 (the nitrate dye of D260) was reduced with zinc and acetic acid. Authentic D260 was obtained. This was a matter of concern since it was discovered that catalytic hydrogenation (one possible means of reduction) destroys D260. The chemical reduction, on the other hand, appears satisfactory.

1.1.1.2 Role of D260 in Speed Decay - Aging of Coating Solution

Independent series of experiments with coating solutions of Formulas 1, 3 and 5 over the past several years show that bulk coating solutions undergo speed decay (determined by film evaluation in the usual manner), and at a rate which increases with increasing concentration of leuco compound[2]. These observations provide a strong indictment against the leuco compound as the principal culprit in rapid speed decay.

To test this indictment an aging-comparison was made between 5/D7 coating solution and an identical solution without D260.

Both solutions were stored in the dark at ambient temperature. Aliquots were periodically removed and photographically evaluated. In the one case the appropriate amount of D260 was added just prior to coating.

The standard coating solution was found to deteriorate significantly within a matter of hours as can be seen in Table 1. In general, densities and development time decrease and fog increases with age.

Table 2 shows the results of 52 days aging of the solution which contained no D260. The contrast with the standard solution is evident and striking. Within the normally experienced range of variations, the densities and development time remained unchanged. It is not correct, however, to say that the solution has not changed, or aged. An examination of speeds indicates a possible trend toward lower speeds during the latter 30 days. This may be real and due to some form of aging or it could be due to the switch in samples of D260. A physical change in the solution was noted during this latter period. The original bright yellow solution became deep orange-brown. This observation is certainly indicative of some chemical change possibly the formation of bromine (see Section 1.2.2.1).

In any event results of this comparison leave no doubt that the D260 is the principal culprit in rapid speed decay. Whether the real cause is D260 itself or is an interaction of D260 with one or more of the other ingredients is not known. Additional solution experiments are planned in order to evaluate a variety of possible interactions.

1.1.1.3 D260 Decay Products - Film Evaluation

Last month's report (Section 1.1.1.5) described the synthesis of 4-dimethylamino phenol (4DMAP) and referenced[3] the suspicion that the material, a possible degradation product of the D260 hydroperoxide, might be responsible for the observable properties of speed decay. 4DMAP and related compounds were therefore evaluated for their effect on the principal film system--5/D7.

The maximum amount of 4DMAP that can be added to the 5/D7 system without affecting its photosensitivity is about 1 μg. Addition of 5 μg resulted in lower densities with longer development times. Ten micrograms gave only a very slight image (maximum density .23) after 360 seconds development. Printout exposures gave parallel results but to less extent.

In contrast to 4DMAP, its 3-isomer (3DMAP) had considerably less effect. One hundred micrograms had no apparent effect whatsoever, and 500 μg was necessary to observe lower densities and longer development time.

The addition of 3 mg phenol which is about one-thousand times the weight of 4DMAP had no effect on either the development or printout mode. Significantly lower densities and longer development time were observed with 10 mg. The addition of 12 mg N,N-dimethylaniline (DMA) also had no significant effect.

Phenol and DMA each contain one of the functional groups present in 4DMAP and yet both are on the order of a thousand times less effective. Thus, both the hydroxyl and dimethyl-amino groups must be part of the same molecule and preferably para (4DMAP) for maximum effectiveness. In support of this conclusion is the effect of 4-methylamino-phenol (4MAP = Elon) an analogue of 4DMAP in which one methyl group has been simply replaced with a hydrogen. It gave results essentially identical to 4DMAP.

It is noteworthy that both 4DMAP and 4MAP are reducing agents as evidenced by their effectiveness as developers for silver halide films. Phenol and DMA are not. No data on 3DMAP has been found but it is probably not an effective reducing agent, and hence more like phenol and DMA. This can be supported by the work of Tani[4] who has related molecular orbital calculations of certain substituted benzenes, including amino phenols, to their effectiveness as silver halide developers, in other words, as reducing agents. His calculations show that 4DMAP and 4MAP should be effective developers whereas 3DMAP should not be.

It thus appears that easily oxidized compounds are particularly detrimental to the leuco system. This conclusion is further substantiated by earlier work in which antioxidants (compounds which are themselves easily oxidized) such as Plastinox 425 and 2,6-di-tert-butylcresol were found to also be detrimental.

In contrast to those antioxidants, 4DMAP can theoretically be formed in situ from D260 via the hydroperoxide[3]. If the D260 hydroperoxide does form in the film, then it may well be the initial cause of rapid speed decay in which case prevention of its formation could provide the cure. The same argument holds for the 7/D7 system in which LCV replaces D260, since 4DMAP would also be the result of hydroperoxide decomposition.

An alternate approach would be a leuco compound whose hydroperoxide would not yield 4DMAP but rather a less detrimental artifact. An example would be leuco malachite green or its D260 analogue[5], the hydroperoxides of which should give phenol. Phenol is about a thousand times less detrimental than 4DMAP. If all other aspects of the mechanism were the same, a thousand fold increase in shelf-life is conceivable. This would mean extension of the current useful life of 30 minutes to a life of around 21 days.

This argument is of course highly over simplified. It assumes one cause and one mechanism for the rapid decay, and also identical rates for hydroperoxide formation and subsequent decomposition regardless of the leuco compound's structure. The argument does, however, serve to illustrate one approach to solving the speed decay problem, and it also serves as a guide to planning our studies. For example, hydroperoxide formation in the film is not an established fact and direct identification is viewed as extremely difficult. However, identification of 4DMAP and a correlation of its increasing concentration with increasing loss of photoresponse would be next to positive proof. A search for 4DMAP in aging film has been planned but the current shortage of manpower has delayed this effort. A parallel effort in synthesizing other leuco compounds is underway but has been hindered for the same reason.

1.1.2 Other Leuco Compounds

Last month's report (Section 1.1.2) mentioned some preliminary studies with other leuco compounds, particularly leuco malachite green (LMG) analogues. Little progress was made this month due to the shortage of manpower; a situation which will have been corrected by the middle of March. Bearing any unforeseen complications in other work areas, there should be results to report next month.

1.1.2.1 2-Methyl Leuco Crystal Violet (D416)

The title compound has been synthesized in 70% yield by the condensation of N,N-dimethyl-m-toluidine with Michler's hydrol in dilute hydrochloric acid. It has not yet been evaluated.

1.2 Carbon Tetrabromide (CBr_4)

A 500 g sample of CBr_4 was received from Berk Limited's Chemicals Trading Division (England). This material was manufactured in France and was nearly pure white as received. It

gave excellent film results as received and sublimation did not alter the film results. A quote on 50 and 100 kg of this material has been requested. A request has also been made for any available analytical specifications.

1.2.1 Deterioration Studies

The purification and aging studies with BDH and Freeman CBr_4 are underway. Chart I outlines the program. The plan is to comparatively evaluate the variously purified and stored samples over an indefinite period of time. In this way it is hoped that the cause of deterioration can be identified as atmospheric contamination, method of purification, or the crude material itself. Comparative mass spectra are also planned to supplement the film evaluation.

1.2.2 Film Additions of Suspected CBr_4 Decomposition Products

1.2.2.1 Bromine

It was reported last month (Section 1.2.1.4) that 10 µg of bromine (17 ppm of CBr_4 on a molar basis) had no effect on photographic properties. This evaluation was extended to include 100 µg, 300 µg, 500 µg, and 1 mg. The results in Table 3 show that 100 µg is detrimental. The 1 mg level resulted in immediate reaction with the coating solution resulting in formation of a brown color and the precipitation of some unknown material. Interestingly, the behavior parallels that of aging coating solution described in Section 1.1.1.2, with the exception of the precipitation.

1.2.2.2 Tetrabromoethylene

The decomposition of CBr_4 to give bromine can be accompanied by the formation of tetrabromoethylene (C_2Br_4). The addition of 50 mg to 5/D7 has no effect on photographic properties and no effect on speed decay. Total replacement of CBr_4 with 623 mg of C_2Br_4 (maximum solubility) resulted in no photosensitivity in either printout or development mode.

1.2.2.3 Hexabromoethane (C_2Br_6)

Last month's report describes the comparison of 311 mg of C_2Br_6, hexabromoethane, which is near the limit of

its solubility, with the standard 1.2 g of CBr_4. The results showed that C_2Br_6 is a very poor activator with essentially no development capability. The comparison was misleading, however, because the relative effects of CBr_4 at the same low level were unknown. Of particular interest was the blue printout image afforded by C_2Br_6, and whether CBr_4 would also give a blue image rather than the usual deep magenta, at the lower concentration.

Both printout and development comparisons were made between CBr_4 and C_2Br_6 at the 311 mg level. The results are shown below:

Development Mode

	Dmax	Dmin	ΔT_r	AEI	γ
CBr_4:	1.22	0.22	109 sec.	0.70	0.9
C_2Br_6:	-	-	960 sec.	-	-

Printout Mode

	Dmax	Dmin	AEI	γ
CBr_4:	2.24	0.06	2.8×10^{-4}	1.4
C_2Br_6:	0.98	0.07	7.0×10^{-5}	0.8

At this level CBr_4 still gives a deep magenta image whose maximum density is read with the Wratten 94, blue filter. The blue image with C_2Br_6 gives maximum density with the Wratten 93, green filter. Visible spectra of the two images confirm the fact that little if any D7-dye is formed in the image with C_2Br_6 while considerable D7-dye is formed with CBr_4.

This fact establishes a difference in mechanisms for these two compounds. It is not known whether D7-dye is formed by direct reaction between D7 and hydrogen bromide or whether another mode is operative, such as extraction of hydrogen by D7 radical cation. Whatever the mechanism, CBr_4 is capable of producing D7-dye and apparently C_2Br_6 is not. This fact should be valuable in further elucidating the mechanisms of image formation and optical development.

1.3 N-Oxide Program

1.3.1 4-Picoline-N-Oxide (4PO)

The in-house synthesis of 4PO has been completed. The material was purified and dried in the standard manner. It was found to give film results identical to those obtained with purchased and purified 4PO. This completes the standardization study of 4PO. The ability to synthesize acceptable material provides insurance against future supply problems and provides additional assurance that presence or absence of unique impurities is no problem.

1.3.2 Other N-Oxides

The last two N-oxides (N-36 and N-37) of the current study have been evaluated. The results of the Phase I study are being evaluated and will be reported in the future.

1.4 Binder Studies

Nothing to report.

1.5 Environmental Studies

1.5.1 Air Sampling

With more data, the argument incriminating oxidants, which was presented in last month's report, becomes less persuasive (Table 4). In the past week we have "good" film, samples 51PQ and 53PQR, with values of 0.069 and 0.100 mg O_3/M^3, and "bad" film, samples 48CH and 48RS, with values of 0.022 and 0.019 mg/M^3. This leaves us with no good correlation at present. Preliminary data for H_2Se and H_2Te show no correlation either. A few additional samplings will be made, using both "good" and "bad" film areas. There is a time lag. We have taken quite a few samples for which the data have not yet been reported. We are currently still sampling for oxidants, HCl, HBr, H_2Se, and H_2Te. Costs for the sampling from December 17, 1971 to date are still under the original estimate for three weeks of intensive sampling.

1.5.2 Environmental Contamination: "Good" and "Bad" Films

Previous reports including the proposal for 1972 have described the phenomenon of environmental contamination. The preceding section and last month's report have presented the results of our air analysis program and have attempted to draw a correlation between particular contaminant levels and the corresponding film results. Film results have been generally described as "good" or "bad." Figure 1 shows some examples of what is meant by "good" (nos. 5 and 6) and "bad" (nos. 1, 2, 3 and 4) films. It can be seen that "bad" is a matter of degree. Although nos. 3 and 4 appear comparable to nos. 5 and 6 and would therefore appear "good." Their D-log E curves show otherwise as can be seen by comparing Figures 2 and 3 with 4 and 5.

It is not known at this time whether the contamination is gaseous or particulate, but there is no doubt that it is airborne. Figure 1 shows the films results conducted in the darkroom which is adjacent to the environmentally controlled room. The controlled room has continually given "good" films as evidenced by nos. 5 and 6. Film 1 shows the results from the adjacent uncontrolled room. After the installation of a plastic, airtight ceiling the results was film 2 in which a slight improvement can be imagined. The floors were then cleaned and waxed and a Barnebey Cheney air purifier was installed. Two days later the result was film 3. A significant improvement is obvious but contamination is still prevalent as evidenced by the D-Log E curve of Figure 2. After another two days the result was film 4 which is essentially identical to film 3 (see Figure 4). Door jambs were sealed with felt padding and air purification continued. Another three days afforded film 5 which is "good" as seen by the D-Log E curve of Figure 4. Four days later the films were still "good" as seen in film 6 and Figure 5. At a later date the air-purifier was removed and within 24 hours films were again like nos. 3 and 4.

1.6 Other Film Systems

Nothing to report.

1.7 Film Analysis of Decay Products

Nothing to report.

1.8 Dye Identification

Nothing to report.

1.9 Systems Nonuniformities

Nothing to report.

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TABLE 1

AGING STUDY OF STANDARD 5/D7 COATING SOLUTION

Exp. No. 1769	γ	AEI	Dmax B	Dmin B	Dnet B	ΔT_r sec.	Curve Smooth	Curve Shape	ΔT_a
48-2	1.30	1.10	2.14	.22	1.92	120	2	2	2min.
43-9	1.80	.40	2.06	.26	1.80	120	1	1	5min.
43-10	2.60	.35	2.07	.28	1.79	130	1	1	10min.
43-11	1.60	.40	1.76	.22	1.54	120	1	1	15min.
44-2	1.80	.55	2.06	.33	1.73	120	1	1	20min.
44-3	3.00	.50	2.44	.37	2.07	120	1	1	30min.
44-4	1.00	1.20	1.52	.38	1.14	110	1	0	40min.
44-5	-	-	1.70	.33	1.37	110	-	-	50min.
44-6	1.5	.50	1.50	.33	1.17	100	0	1	1 hr.
44-7	2.40	..13	1.90	.40	1.50	90	1	1	2 hr.
44-8	-	.1	1.42	.37	1.05	90	-	-	3 hrs.
44-10	1.30	.15	1.30	.36	.94	90	1	0	4 hrs.

ΔT_a = Time lapse after addition of activator(s) to coating solution.

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TABLE 2

AGING STUDY OF 5/D7 COATING SOLUTION MINUS D260

Exp. No. 1769-	Gamma	AEI	Dmax B	Dmin B	Dnet B	△Tr sec.	Age Days	Comm
45-1	1.4	1.1	2.30	.22	2.08	110	0	-
45-2	1.2	2.0	1.95	.24	1.71	130	.16	-
46-2	1.2	1.5	2.30	.25	2.05	120	2	-
47-1	2.0	.67	2.20	.28	1.92	120	3	-
47-4	1.4	8.5	2.60	.35	2.25	140	4	Some Blotch
48-1	1.4	1.6	2.36	.24	2.12	130	8	-
49-4	1.6	.32	1.50	.24	1.26	140	15	-
49-5	1.8	6.3	2.40	.37	2.03	130	15	Some Blotch
1824-1-2	1.5	1.2	2.31	.21	2.10	210	23	Used Another D260
5-1	1.9	.61	2.24	.20	2.04	190	30	-
9-1	1.5	.35	1.94	.24	1.70	210	37	-
13-2	2.0	.25	1.50	.16	1.34	130	44	Used Another D260
18-5	2.8	.38	2.40	.34	2.06	130	52	Some Blotch

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TABLE 3

Addition of Bromine to 5/D7
(Ref. 1833-33)

Br_2	Dmax	Dnet	ΔTr (sec.)	Comments
-	1.88	1.67	36	Control
100µg	1.70	1.50	25	
300µg	1.34	1.00	18	
500µg	1.42	1.09	24	
1 mg	-	-	-	Precipitate

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...ides results reported to us through Feb. 28, 1972.
...corresponding to bad film are marked with an asterisk.

Sample Number	NO_2, ppm	Oxidants, mg/M³	I_2, mg/M³	HCl mg Cl^-/M³	HBr mg Br^-/M³	Arsenic, mg/M³	H_2Se mg/M³	H_2Te mg/M³
1	0.011		0.027	0.011	0.007	0.0004		
2	15			08	08	< 007		
3	19		04	90	08	04		
4	10		07	1.167	10	< 007		
5	16		08	08	07	< 007		
6	07		29	11	08	02		
7	05		21	11	08	11		
8	11			1.228	11	< 007		
9	17		19	28	07	< 007		
10	15		03	39	09			
11	19					05		
12	25	0.026	25	0.640	07			
13	06							
14	17	23	12	09	07	< 007		
15	05	03	13	12	07			
16	14	26	44	0.106	08	< 007		
17	10	23	13	13	08	< 007		
18	07	46	15	0.139	07	04		
19	12	17	14	0.184	09	< 007		
20	07	10	18	25	06	< 007		
21	07	13	22	21	08	< 007		
22	12							
* 23	10	21	20	13	09	< 007		
24	12	49	08	0.360	09	< 007		
25						< 007		
* 26	18	31	09	0.128	13	< 007		
* 27	12	65	11	0.161	10	< 007		
* 28	12	74	14	94	09	< 007		
* 29	08	95	12	24	08	< 007		
* 30		52	10	51	09	04		
* 31	12	77	11	0.119	09	< 007		
* 32		74		0.120	08	01		
* 33		27		0.140	12			
* 34	15	18						
* 35		84	14	20	07	< 007		
* 36	08	58	19	63	11	< 007		
* 37	02	12	19	0.125	10	< 007		
* 38	07	23	16	49	08	01		
* 39		0.186	32	84	0.110	< 004		
** 40	22	0.099	09	0.108	0.117	< 01		
* 41	14	0.154	10	0.122	0.131	< 01	0.035	0.080
* 42	25	21	06	0.101	0.101	01	0.129	0.207
* 44		< 05	21					
45		37	30					
* 46		20						
47		06						
* 48CH		22					0.148	0.024
48PQ		37					0.139	0.136
* 48RS		19					0.099	0.113
* 49CH		43						
49PQ		55						
* 51CH		0.114						
51PQ		69						
(*) 52CH		45						
52PQ		43						
(*) 53CH		82						
53PQ		0.100		1.152	0.058			

25X1

CHART I

CBr_4 - Deterioration Study

I. Controlled Atmosphere (80% N_2 and 20% O_2 purified)

A. Samples Currently Underway

1. BDH sublimed

a) First sample sublimed yellow
b) Second sample sublimed colorless

2. Crude BDH

3. Newly acquired Berk Limited material

B. Samples Not Yet Underway

1. Freeman (twice recrystallized)

2. Freeman (twice recrystallized and sublimed)

3. Freeman (twice recrystallized and dried over P_2O_5)

II. Laboratory Atmosphere

A. Samples Currently Underway

1. BDH sublimed
a) First sample sublimed yellow
b) Second sample sublimed colorless

2. Crude BDH

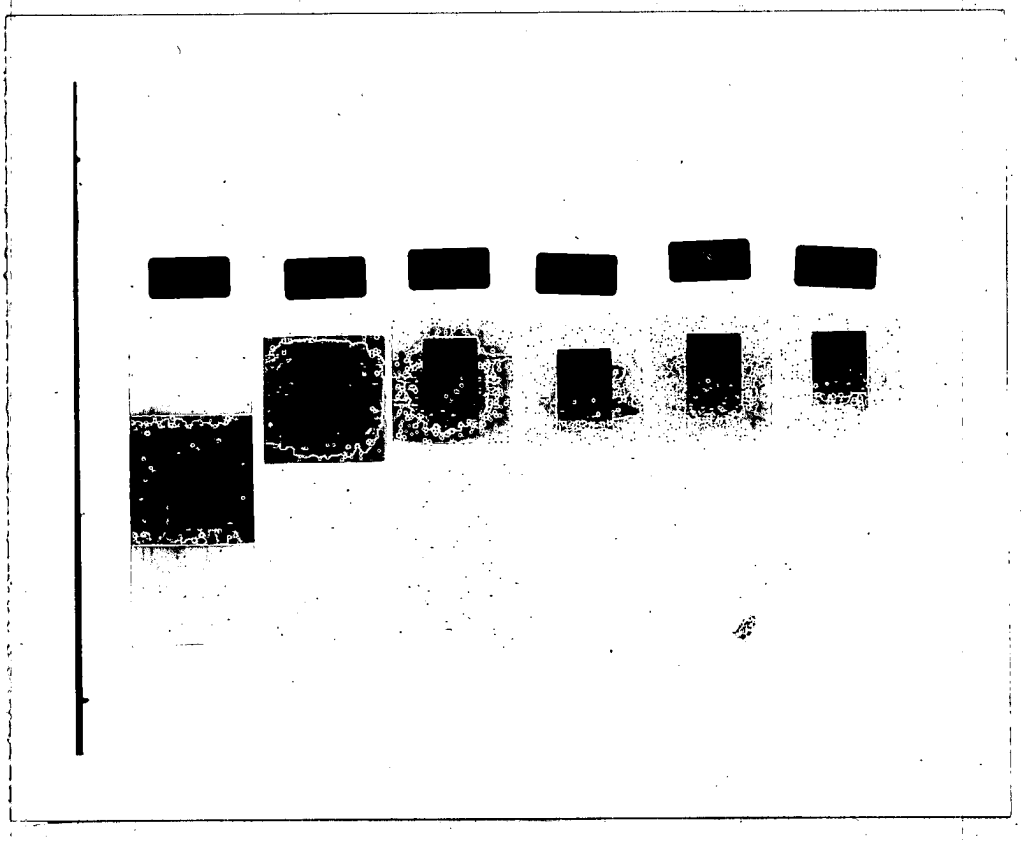
B. Samples Not Yet Underway

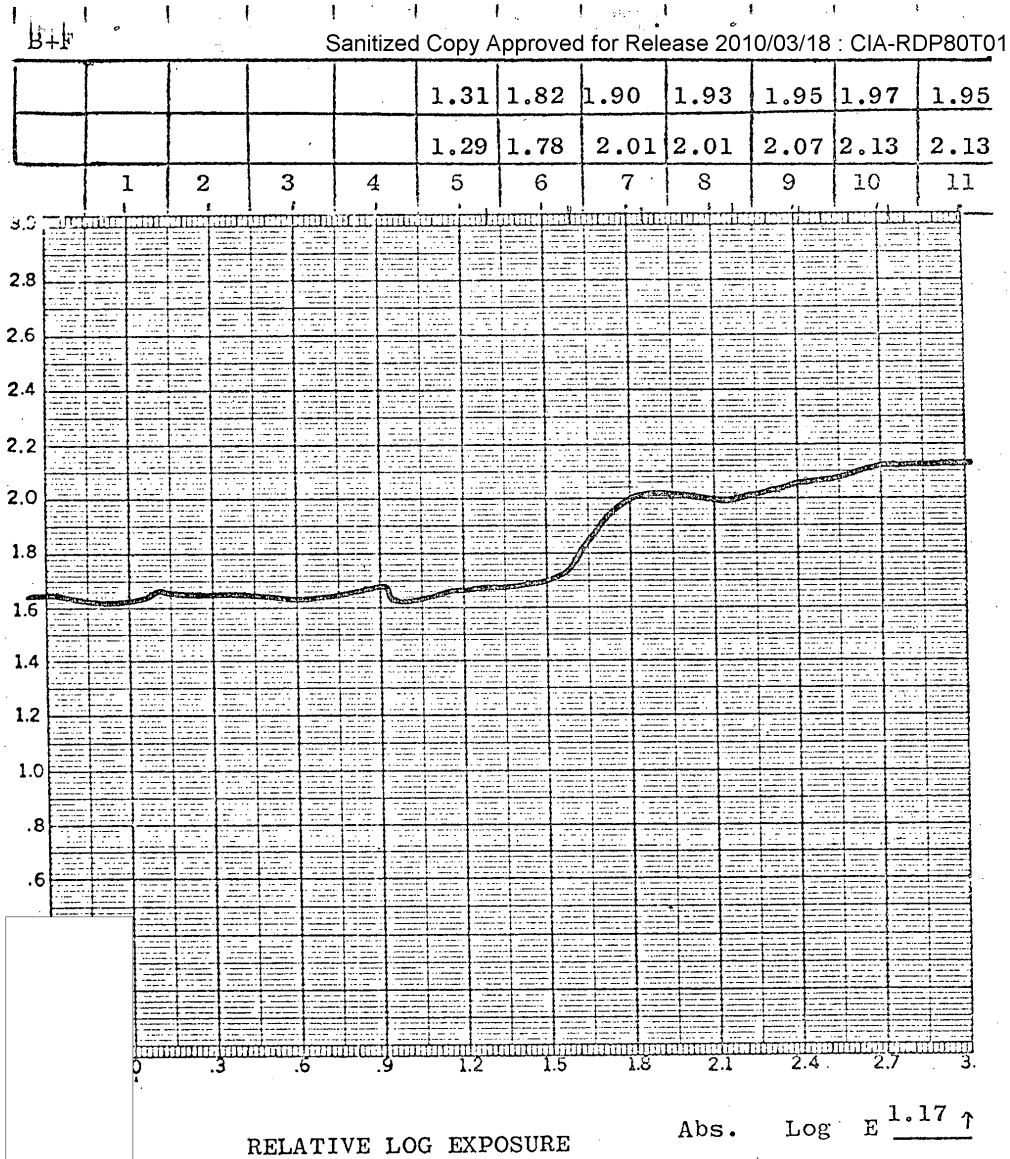
1. Freeman (twice recrystallized)

2. Freeman (twice recrystallized and sublimed)

FIGURE 1

ILLUSTRATION OF "GOOD" AND "BAD" FILMS





25X1

FIGURE 2

FILM 3 of FIGURE 1

EXPOSURE STATION: No. 2
INTENSITY: 166 mc
EXPOSURE TIME: .09 sec.
E EXPOSURE: 14.9 mc sec.
Log EXPOSURE: 1.17

RED LITE STATION: HID2 LAMP:
STATIONARY PLATEN: X OSCILLATING:
RED LITE DEV. TIME: 40 sec.
TIME/PASS:
FILTERS USED: 2030, RG654 N.D.:
RADIATED POWER:

ROOM TEMP: 70°
HUMIDITY: 41%
PLATEN TEMP:

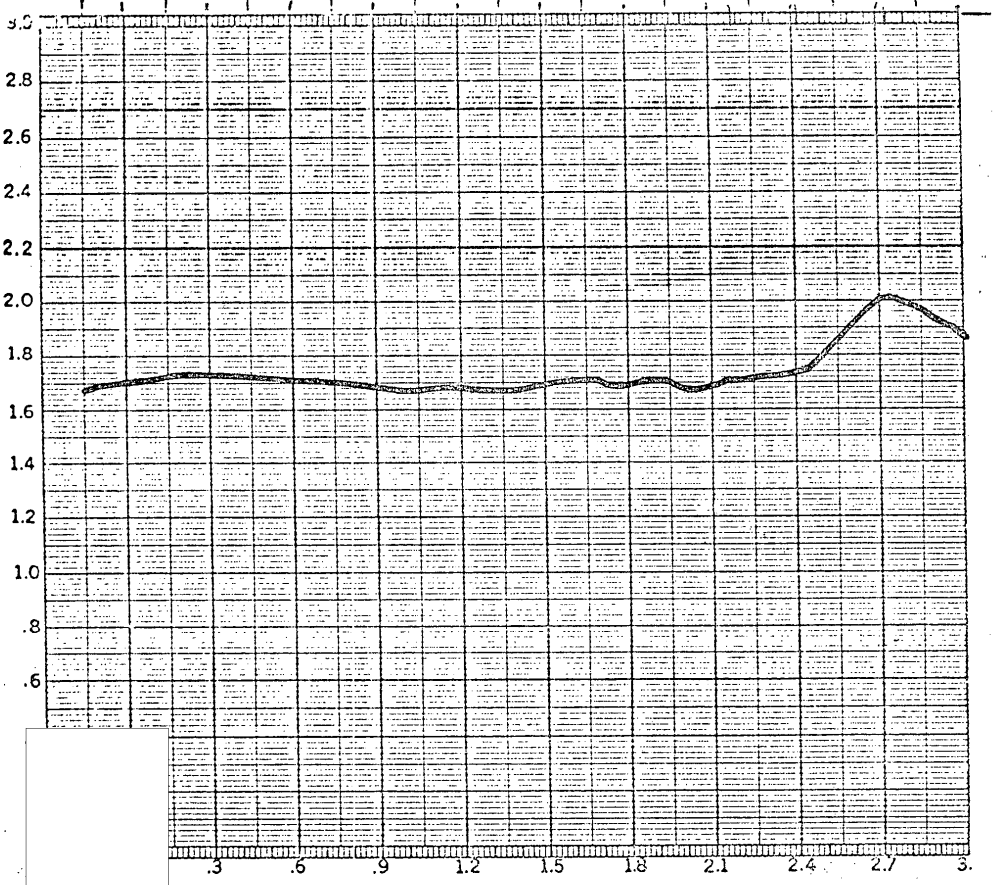
25X1

γ = -

AEI: -

B+F

G								1.83	1.84	1.93	1.89
B								1.71	1.75	2.02	1.88
	1	2	3	4	5	6	7	8	9	10	11



RELATIVE LOG EXPOSURE

Abs. Log E 1.17

25X1

FIGURE 3

FILM 4 of FIGURE

EXPOSURE STATION: No. 2
INTENSITY: 166 mc
EXPOSURE TIME: .09 sec.
E EXPOSURE: 14.9 mc sec.
Log EXPOSURE: 1.17

RED LITE STATION: HID2 LAMP:
STATIONARY PLATEN: X OSCILLATING:
RED LITE DEV. TIME: 34 sec.
TIME/PASS:
FILTERS USED: 2030, RG654 N.D.:
RADIATED POWER:

ROOM TEMP: 70°
HUMIDITY: 41%
PLATEN TEMP:

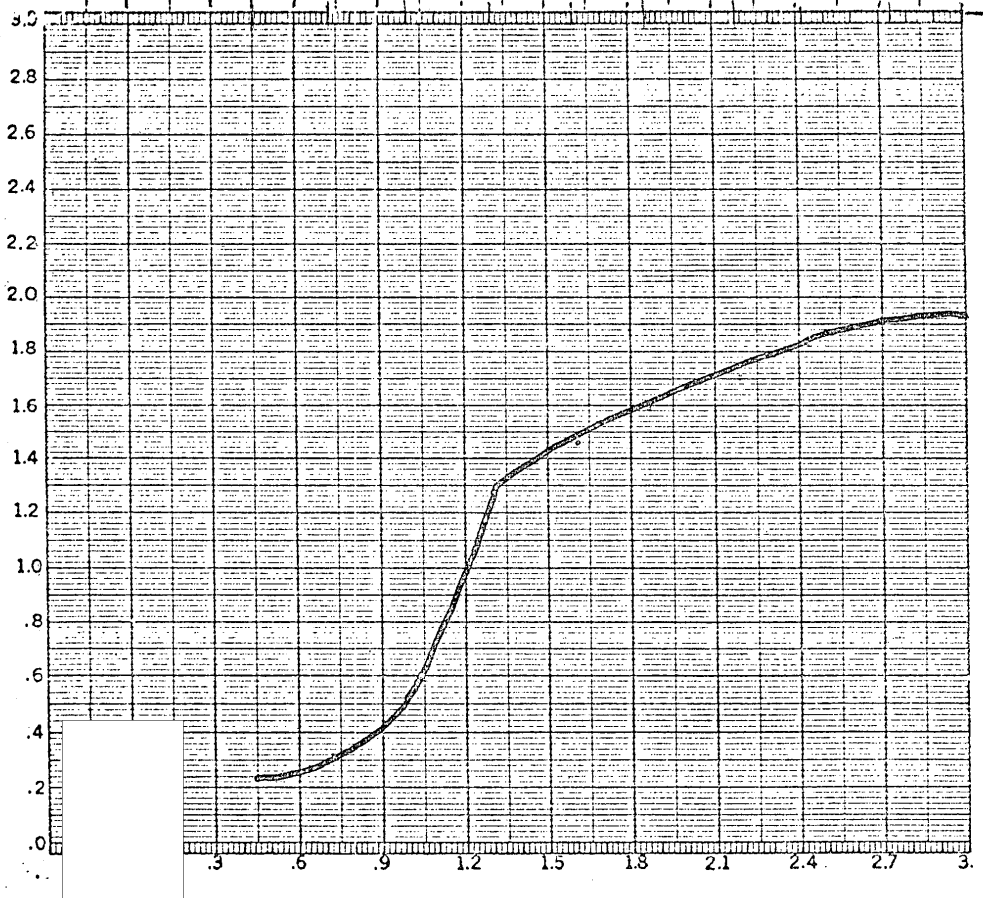
25X1

γ = -

AEI: -

B+F

		.24	.35	.56	1.42	1.62	1.75	1.78	1.86	1.89	1.88
		.24	.32	.55	1.31	1.46	1.59	1.71	1.83	1.91	1.93
	1	2	3	4	5	6	7	8	9	10	11



RELATIVE LOG EXPOSURE

Abs. Log E 1.17↑

FIGURE 4

25X1

FILM 5 of FIGURE

EXPOSURE STATION: No. 2
INTENSITY: 166 mc
EXPOSURE TIME: .09 sec.
E EXPOSURE: 14.9 mc sec.
Log EXPOSURE: 1.17

RED LITE STATION: HID2 LAMP:
STATIONARY PLATEN: X OSCILLATING:
RED LITE DEV. TIME: 45 sec.
TIME/PASS:
FILTERS USED: 2030, RG654 N.D.:
RADIATED POWER:

ROOM TEMP: 70°
HUMIDITY: 41%
PLATEN TEMP:

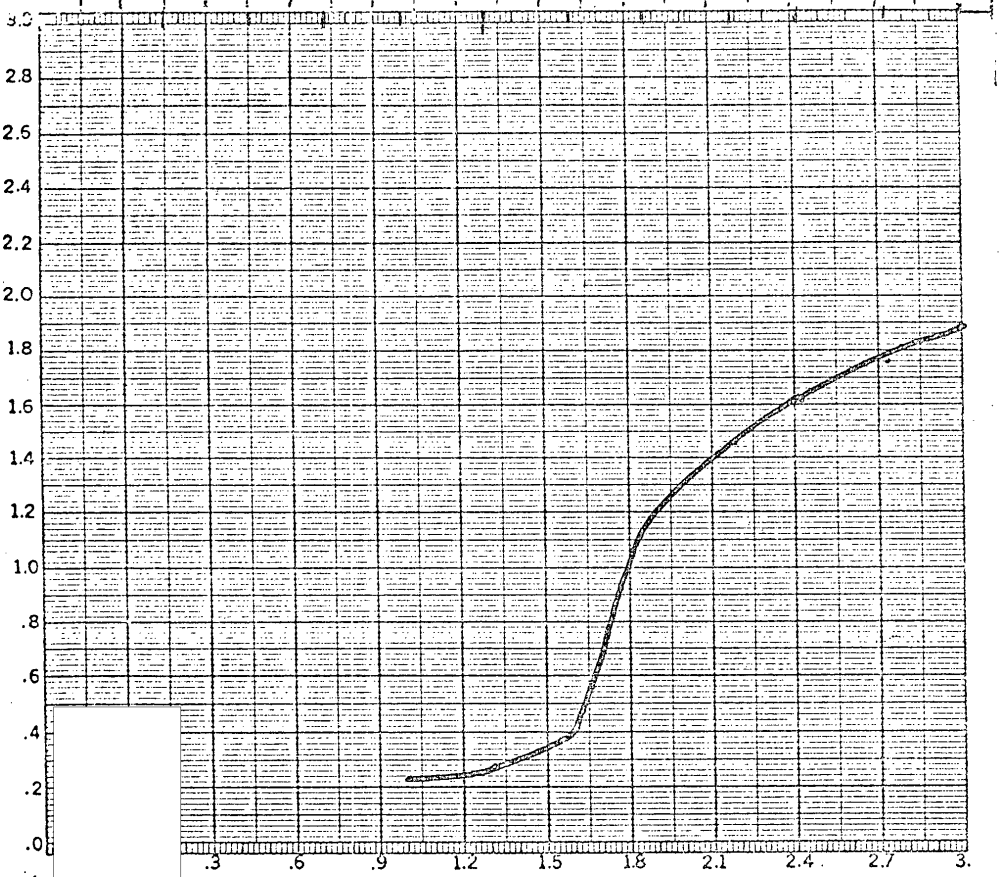
25X1

γ = 2.5

AEI: 5.5

B+F

G					.23	.26	.40	1.28	1.61	1.71	1.78	1.83
B					.23	.28	.41	1.13	1.46	1.62	1.76	1.89
	1	2	3	4	5	6	7	8	9	10	11	



RELATIVE LOG EXPOSURE

Abs. Log E 1.17 ↑

FIGURE 5

FILM 6 of FIGURE 1

25X1

EXPOSURE STATION: No. 2
INTENSITY: 166 mc
EXPOSURE TIME: .09 sec.
E EXPOSURE: 14.9 mc sec.
Log EXPOSURE: 1.17

RED LITE STATION: HID2 LAMP:
STATIONARY PLATEN: X OSCILLATING:
RED LITE DEV. TIME: 47 sec.
TIME/PASS:
FILTERS USED:2030, RG 654 N.D.:
RADIATED POWER:

ROOM TEMP: 70°
HUMIDITY: 41%
PLATEN TEMP:

25X1

γ = 3.1

AEI: .89

REFERENCES

1) Aaron and Barker, J. Chem. Soc. 1963, 2661.

2) Proposal: January 1, 1972 through December 31, 1972; Section 2.6, p.40.

3) Ibid, Section 3.3.1.1, p.48.

4) Tadaaki Tani, Photograph Sci. Eng., 1971, 15(5), 384.

5) Ref. 2, Section 3.3.1.2, p. 50.

6) Ref. 2, Section 3.3.6.

2.0 ENGINEERING

2.1 Calibration and Equipment Maintenance

Calibration of all 325B equipment has continued on a routine basis. All of the HID-2 red light developers recently completed have been calibrated and tested; and for the first time multiple red light development units have been built which are identical in processing characteristics within the experimental error of the present testing equipment.

New designs for portable, exposure and coating stations are being reviewed for portable coating and processing of the film in remote areas in support of the environmental studies program.

2.2 Mechanical

The HID-2 units have been completed, tested and evaluated. The units have performed well and are presently used for all processing of the 325B material. Work will continue evaluating these units and improving red light technology in general.

2.3 Shelf Life

No work has been done on shelf life because of the contamination found throughout the [redacted] building with the exception of the environmental room presently being used by the chemistry group. 25X1

2.4 Red Light Research and Development

Work is continuing on the HID-1 and HID-2 processing units to determine the proper filtration for optimum performance of the 325B film. A series of interference filters has been received which have nearly identical cut-on characteristics. These are presently under test to determine if in fact all filters are exactly the same when placed in all of the HID-2 units. These filters have a 50% cut-on point of 665 nm and 655 nm. Evaluation is not yet complete on the repeatability of these particular filters.

25X1

2.5 Environmental Conditioning

This is a new section having been added because of the extreme environmental contamination problem encountered within the [redacted] building. As we reported in the last 25X1
report, some form of contaminant has essentially stopped 325B processing outside of the one environmentally conditioned room that the chemistry group is presently using. Tests have been run in other parts of the building which show this contamination problem. A large effort is underway to (1) determine the cause of the contaminant and (2) determine methods for countering the contaminant. Barnebey Cheney activated charcoal units have been placed in several laboratories with the hope they will remove the contaminant, thus cleaning up the room and allowing experimentation to continue. The engineering darkroom located in the front portion of the building has had a special double door, plastic sealing of the room ceiling, and a Barnebey Cheney unit placed within the room area. This proved inaffectual; only very slight improvement in film response was observed. An experiment using 275 cubic foot of 20% oxygen, 80% nitrogen (pure air) was introduced into the room and this made a significant improvement in the film samples. This pure air mixture was dumped directly into the hood where the coating and red lighting was done. A marked improvement in the samples was observed. However, upon the room standing idle for 24 hours, the film produced showed severe blotch. This showed that the contaminant is airborne and is not contained in a pure air mixture; however, this method is unsuitable for this type of room operation and another method must be found which is more efficient and practical. An alternate darkroom is under construction and consists mainly of a small environmental room 14' x 8' x 7' constructed in the rear section of the [redacted] 25X1
building. It will be completely sealed from the surrounding contaminated air. A Barnebey Cheney unit will be installed along with a room air-conditioner to maintain temperature and a double trapped door installed to reduce the airflow of contaminated air when entering the room. The room will be pressurized with an activated charcoal makeup air system, which will cycle through the Barnebey Cheney unit in a double filtration system. An exterior mounted vacuum system will be used for daily cleaning of the area. An air sampling hose will be introduced so that air samples within the compartment can be made without contaminating the room with sample vapors. It is anticipated the room can be completed and tested prior to 1 April 1972. Information received from testing within this unit should assist in determining the contaminant, thereby making cleaning of the air much simpler than the present gross removal system used. If successful this should lead the way to inexpensive, multiple, contaminant-free test rooms available to the entire project.

25X1

3.0 PERKIN ELMER INTERFACE, QC/QA

3.1 Supply of Film and Equipment

3.1.1 Delivery of 325B Film

Shipment No.	Date Coated	Date Received	Time Lapse
1	14 Jan.	17 Jan.	2 Days 17 Hrs.
2	31 Jan.	3 Feb.	2 Days 17 Hrs.
3	14 Feb.	16 Feb.	1 Day 17 Hrs.
4	1 Mar.	7 Mar.	5 Days 17 Hrs.

Shipment 1 was delivered by station wagon and was received in good order. It was successful and is described in the first monthly report. It consisted of sixty 1-1/2" wide strips of film with sufficient coating for exposure with a Kodak No. 3, 21 step tablet.

The second shipment was sent by Airborne Freight Corp. The freight company sent it to the wrong airport causing a two day delay in reaching its destination. The ten 7" x 7-1/2" sheets were fogged upon arrival. A possible cause of the fog is displacement of the air in these individual boxes by CO_2 from the dry ice and/or an increase in temperature, even though the box was still "cool" when opened (see Section 3.3).

Shipment 3 was delivered or produced in three parts: one by Airborne Freight Corp., one by station wagon and the third part by hand coating in the Perkin Elmer laboratory. The success of delivery is judged primarily on the good printout results. The lack of a good developed film sample prevented the test of the film from being conclusive. Because the problem could not be identified with certainty, all of the equipment plus two sheets of the film were returned. Upon return a subsequent additional control of the HID-2-004 red light development unit was found to be operating within tolerances and the film materials used for hand coating were found to be "probably" good. The quality control on the film and ingredients brought back yielded only probable conclusions because in both cases the effects of aging were already apparent. A possible cause of the problem at the P.E. facility might be air contamination, although at this point it is not at all conclusive. Air samples were taken in their lab each of the three days and will be analyzed for oxidants, hydrogen-halides, tellurium and selenium Although the hand

coatings made for this purpose appear inconclusive, the analyses of the air samples will still be useful for establishing a baseline at their facility and for comparison with the pollution profile found at our facility.

Shipment 4 was made for the sole purpose of testing and solving storage and shipping problems. The shipment spent an undue amount of time in transit because Airborne Freight Corp. did not off-load it the first time it was at Bradley International Airport, and because the receiver could not pick it up on the weekend when the freight company finally got it to Bradley. The use of this film was not totally lost, apparently because some dry ice remained and none of the film had fogged. Fogging is the primary indication of problems, which are to be tested this time. The number of sheets made for Shipment 4 was only 1/3 of the normal size shipment. Of this only 2-1/2 sheets, cut up for sample size, were sent. An equivalent shipment was retained for comparison purposes. The sixth coating was cut up for coating control purposes.

Perkin Elmer was made aware of the work load placed on the 325B project. The expected need will be approximately 15 sheets of the film twice a month as originally planned. All understood that a lost shipment for whatever reason could not be made up. The estimated cost of each shipment including time and materials but not overhead is about $1,000. The date of the next shipment depends on solving the current storage shipment problem. Up to now the shipments are still on schedule.

3.1.2 Delivery of Solvent Rinse

One and half gallons of solvent rinse was delivered 16 February with Shipment 3. They now have a total of two gallons which should hold them until the scheduled shipment of solvent rinse in mid April.

3.1.3 Delivery of Red Light Development Units

The HID-2-004 unit was sent air freight and received in good order 7 March. It had originally been shipped on schedule the second week of February by station wagon. It was not left at Perkin Elmer because the problems with the developed samples could not be identified at the time. The unit was returned with film for more controls and testing. The subsequent controls show that the problems encountered were not traceable to the development unit. The earlier model, 4B, was also returned.

25X1

3.2 Cross Calibrations

The comparison of three TD-102 densitometers, one at Perkin Elmer and two at [redacted], show that a major discrepancy results when using 325B film. The ordinary black calibration tablet supplied with each densitometer does not reveal any discrepancy, with the visible or the color filters. The mixed color portion of an Ektacolor internegative tablet supplied by P. E. used as a second check, shows a discrepancy but not of the same magnitude as revealed by the 325B tablet. With the Ektacolor tablet, the P. E. blue filter density is only 0.01 density units above the reading when using the 325B filter at a density of 1.43 (Figure 5). With the 325B printout sample processed at P.E.'s lab, the density discrepancy at the 1.5 range is 0.11 density units. The Dmax read with P.E.'s densitometer is 3.73 (Figure 1); with the same type instrument used in another department, 3.20 (Figure 2); with the 325B densitometer, 2.70 (Figure 3). One of the defects in making this comparison is the fact that the 325B tablets are not uniform enough to rely on exact positioning of the film in all three of the densitometers. The data plotted to this point, therefore, is only indicative and cannot be used quantitatively.

25X1

Color ratios which could be compared quantitatively between the three units, were plotted. The color ratio in the case of interest is the ratio of the density through the blue filter over the density read through the green filter of the TD-102 densitometer. They are read by placing the reading head of the densitometer down on one spot, and then changing the filters after the unit has been calibrated for each filter using the standard silver tablet. In this way the color ratio can be derived without worrying about replacing the densitometer on the exact spot. The plots comparing the ratios of the three densitometers on the color tablet (Figure 6) as well as the 325B tablet (Figure 4), reveal unequivocally that there is a major discrepancy in the densitometers when reading 325B material. They also show that 325B color might vary with density. At the higher density range, the blue/green ratio of the 325B densitometer is the highest; the same type instrument from another department is the lowest; and P. E.'s densitometer falls in between. This comparison gets mixed below 1.0 densities. Furthermore, there results in a much sharper rise and drop in the color ratio of P. E.'s densitometer going from the eighth to the eleventh step. This explains why P. E.'s plots of the 325B material have such a sharp shoulder at the ninth step when reading through the blue filter.

25X1

A plot of ratios also points out that the Ektacolor tablet will not be a suitable standard for calibrating the densitometers for 325B film. The comparison of the three densitometers on a ratio plot shows them to be very close and to follow each other across the range of the color tablet. One must note that the color ratios of the densitometers do drop as the densities become higher than the fog levels and then rise again. This apparently is a characteristic of the manufacturer's design of the densitometers. This apparently is the reason why the 325B color "appears" to change with density in Figure 4. The plot of the 325B film could be corrected by normalizing against the plot of the Ektacolor ratios.

The probable reason for the densitometer variabilities is the very sharp color profile (Figure 7) of 325B film in the blue and red regions. The convolution of the 325B color with the color curve of the densitometer filter, lamp color, and phototube color detectability is very sensitive to any non-comparability of any of the densitometer components. MacBeth Corporation does not attempt to make color densitometry duplicatable between TD-102 instruments. They recommend making a nomograph of correction values which would mean replotting of all curves before comparing D-Log E curves generated at different facilities. The densitometers, therefore, must be calibrated with sharp filter cut-ons in the critical 325B regions.

It is advantageous to choose that filter which gives us the highest density readings of 325B film. The implications of the 325B color have been presented in three previous studies: identifying maximum density wavebands with a radiometer, checking out a suitable light table color, and maximizing the printing efficiency of 325B negative onto the PH 2000 duplicating film.

Based on the plot of maximum density vs. wavelength (Figure 7), narrow band interference filters at 480 nm and 540 nm instead of the standard Wratten filters at 455 and 540 nm will yield the highest density and will provide the necessary color ratio for the comparisons which have been a valuable tool for judging chemistry work and quality control. A 10 nm 1/2 peak bandpass interference filter is a practical compromise between cost, availability in quantity, maximum transmission (50%), and the narrowness needed to minimize the deviations of source and detector color.

This report does not identify completely the reason why the samples processed at their facility yield such high densities.

25X1

In spite of the difference in densitometers, the samples processed at P. E. had a significant increase in density above what is normally attained at [redacted] The real density 25X1
increase appears to be caused ty the fact that their printouts are done with an EK-101 sensitometer with a 2850°K color temperature. Compared to the daylight exposure of 5500°K, their exposures of 200 seconds to 1700 meter candles allows significant development as well as production of latent image. Without the Pitt 2043 heat absorbing glass, the exposure and development occurs above room temperature. Temperature significantly affects printout results (reported in June, 1970, 325 Progress Report). By removing the color correction CG 5900 and Pitt 2043 filters from one of the [redacted] sensitometers, the 25X1
same increase in densities was produced at [redacted] Because 25X1
of the end use of the film, the printouts as well as the exposures for the developed film, must be done with the correction filters in place to produce 5500°K color temperature as well as a minimum amount of IR. The daylight correction, therefore, must extend out into the infrared region, also. The two filters were taken along with Shipment 3 and the above effect was demonstrated at P.E. Their exposures will now be done with those filters.

3.3 Evaluation of Production, Shipping and Contamination

Two problems have come up within the last month; the air carrier has performed very poorly two out of three times. The first time he sent it to the wrong airport; the last time it went from Boston to Chicago before it was correctly offloaded at Bradley International Airport. Future mistakes will be circumvented by sending the film either by charter plane to Danbury Airport or by using American Airlines priority parcel service to LaGuardia Airport. The reliability of shipment is especially important because of the costs of the film shipment, which is approximately $1000, and of the delay in project performance at P. E. Although the time involved in hand coating limits the amount of film that can be shipped, the primary cost is still the synthesis, purification and quality control of the materials.

The other problem that has appeared in February is the fogging of film in storage. Ten percent of Shipment 1, all of Shipment 2 and the 30% of Shipment 3 that was left at P. E. fogged to about 1.0 density. On the other hand the last samples used from Shipment 1 after four weeks of storage were not fogged and still were up to speed. Shipment 4 was set out to test this and find a

25X1

secure way of sending the film. Unsealed shallow boxes were used for the first two shipments; shallow boxes are necessary because the film is still wet when it is placed in storage and then frozen within two minutes of coating. The third shipment included putting the boxes in unsealed polyethylene bags, which in turn were placed inside unsealed black light-tight bags. A fourth shipment included packaging that duplicated the first three shipments, plus an additional sample sealed in an air-tight metal tube. The displacement of air as a cause of fogging has been observed at [redacted] when argon or CO_2 was purposely used to displace the air. Time and temperature were also observed to bear upon the problem. One sheet that was retained was allowed to have its surrounding air displaced by CO_2 and then warmed to approximately 40°F around the edges only. Because the outside edge was fogged and the center was still useful for sensitometric evaluation, the test indicated that the lack of the oxygen inhibitor plus a "warm" temperature was needed to cause fogging. Shipment 5 will be sent under the conditions demonstrated by the results of Shipment 4.

That the air at the P.E. facility may be contaminating our film is not being overlooked. The three sets of air samples taken the 16, 17, and 18 of February will be used to hopefully shed some light on the subject. The problem involved in evaluating this data is formable (see Sections 3.7 and 1.5).

3.4 Monitoring of Results and Anticipation of Subsequent Plans

The first shelf life study at P. E. was begun with Shipment 1. The data and conclusions will be reported in the monthly report from P. E. It was in regard to this that the Corning Glass 5900 and Pitt 2043 filters were supplied with Shipment 3. Because of the demonstrated difference that the source color has on the film, the shelf life study up to now will have to be repeated using the 5500°K source. The results of 2850°K and the future results of 5500°K will be coordinated with the shelf life study going on concurrently at [redacted] In particularly, one point to be evaluated is the importance of establishing a starting point of the decay curve since the film P.E. uses might already be aged.

The possibility of using their environmental chambers for test and evaluation of storage and sensitometry of our film under a variety of conditions was brought up. In the laboratory adjoining P.E.'s 325B lab is a vacuum chamber that has been instrumented for photographic evaluation of film under camera vehicle environments. A test plan based on the use of this

equipment is being established. Past and concurrent work at [redacted] using glove boxes will be coordinated with their test plan. P. E.'s vacuum chamber is equipped to go to deep vacuums as well as duplicate actual camera environments. It has the advantage of being fully instrumented with controls and monitoring probes. Throughputs are available for working on the film in a photographical and mechanical way under the variety of environmental conditions. To start with, it will be useful for evaluation of storage problems. The program can then advance to environmental conditions during exposure and development. Previous offgassing data was generated with this equipment.

3.7 QC/QA

The supply of chemicals and materials for standard formula 5/D7 has been keeping comfortably ahead of usage rate, in spite of one accidental loss of a quantity of D260. The quality of materials found acceptable by the coaters has decreased somewhat. The materials that yielded a Dmax of only 2.0 and a γ above 3, has been accepted for chemistry work. This has been acceptable for much of the screening work in the chemistry section. The reason for accepting these materials is the same as the reason for not filtering each coated sample and for not adding silicone in each case; i.e., the "cosmetic" defects do not hinder the reading of the necessary data. The supply of materials from ChemSampCo has been increasing, especially since the identification of TMB as an impurity. The new D260 synthesis route being worked on at [redacted] is expected to yield higher and more consistently high quality D260.

Sensitometry within our facility has remained consistent over the last several years and is being maintained at a high quality. Sensitometers and the development units are routinely checked. Comparisons of our data with data that might be generated outside the facility runs into the problem explained in Section 3.2. The use of our MacBeth TD-102 densitometer, Gamma meter, and averaging technique has been such as to maintain consistency within the 325B project.

A major problem for maintaining the engineering R and D schedule has been environmental contamination. Additional Barnebey Cheney charcoal filtering units for air filtration have been ordered. Three of the four cabinet units have been delivered. Difficulties in securing delivery from Barnebey Cheney has delayed upgrading additional coating rooms. Each unit contains six panels with a total of 42 lbs. of activated charcoal, type CH, and delivers 400 cfm of filtered air. That has been sufficient for the one room which is approximately

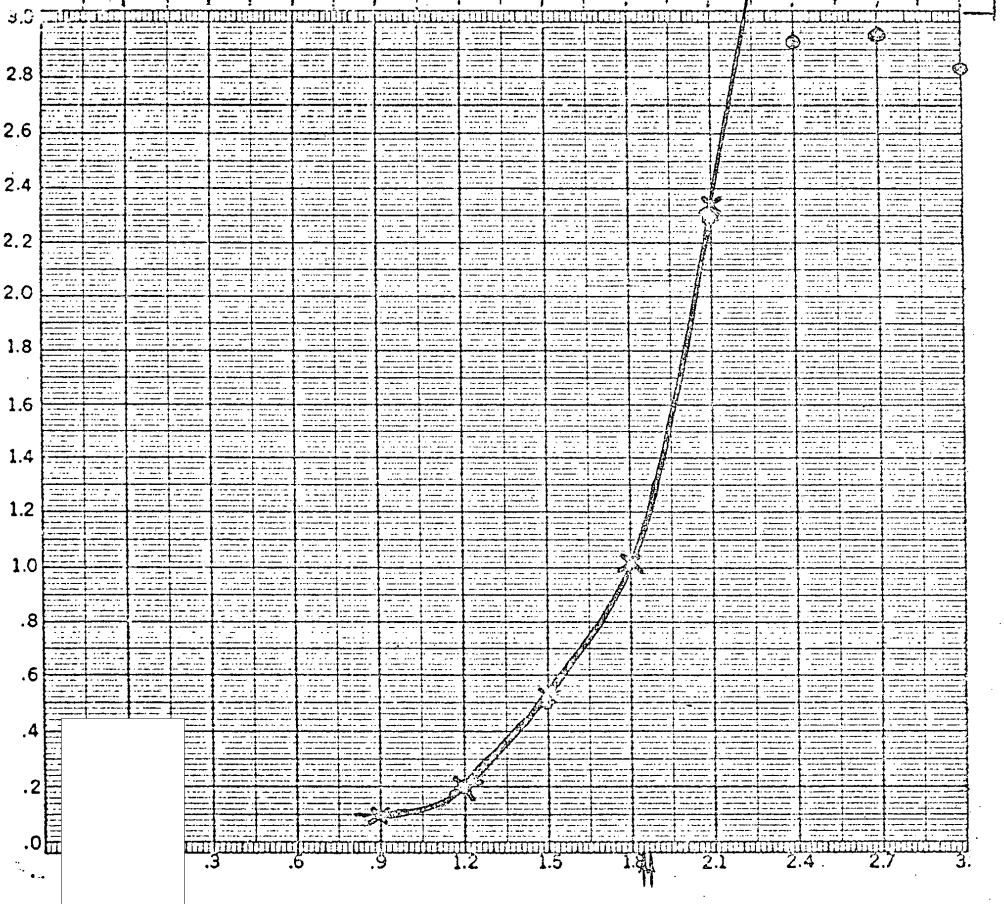
2200 cubic feet in volume. A small adjoining lab of approximately 2000 cubic feet should be ready for daily use the second week of March. Difficulty has been experienced with the engineering darkroom of 2600 cubic feet. A fourth Barnebey Cheney cabinet unit should be delivered by the end of March. At that time at least three, possibly four, rooms should be in full operation.

Experience has shown that the 42 lbs. of charcoal is good for at least one month of continuous operation. Apparently larger amounts of charcoal/cfm will be necessary for the larger labs with particularly severe contamination problems. Type CH charcoal, according to the Barnebey Cheney Company, is particularly suitable for filtering out sulphur dioxide, hydrogen chloride, chlorine gas and other acid gases. As with any activated charcoal the effect of filtration is very broad with respect to the type of contaminants removed. The impregnant only optimizes the efficiency with regard to certain contaminants. Ozone for instance continues to be converted to 0_2 in spite of the impregnant. Although a fiber glass filter is provided ahead of the activated charcoal, it is not effective against particulates to the extent desired. The RFQ that was put together for clean chemistry rooms and submitted to a list of suppliers revealed that "economical" off-the-shelf units all had precise control with regard to temperature and relative humidity. Our primary need is filtration and purification of the air. The temperature and relative humidity need to be maintained at a reasonable 72 $\pm$2°F and 40 to 50% relative humidity. As the rooms are upgraded, the use of "hats and booties" and other procedures typical of a machine coating facility will be instituted.

The identification of the air contaminant producing film problems from the extensive gas samples has not proved fruitful. The most recent results received from the analytical lab seem to go against the earlier observation that oxidants are the possible sources of "bad" film (see Section 1.5). Because of the large number of samples taken, the evaluation of the data lends itself to frequency diagrams (Figures 8 through 12). The data is presented in Table 4, Section 1.5. Although there is considerable overlap of the data, the frequency diagram will distinguish two separate signals if there is a correlation between "good" and "bad" film samples. On this basis a higher level of oxidants show a correlation with bad samples, which was somewhat obvious from the run down of the data on Table 4. In addition the frequency diagram shows a positive correlation with a slightly higher concentration of HBr, which was not so obvious on Table 4. The levels of the contaminants found are low enough that general air pollution in the Cleveland area cannot be overlooked!

B+F

.10	BLUE	X		.10	.21	.52	1.02	2.34	3.73	3.56	3.22
.10	GREEN	⊙		.10	.21	.50	1.01	2.30	2.93	2.96	2.84
		1	2	3	4	5	6	7	8	9	10	11



RELATIVE LOG EXPOSURE

Abs. Log E 4.53↑

FIGURE 1

P. E.'s TD 102 DENSITOMETRY

FORMULA 5/D7

Coated: [redacted] 25X1an

Exp. and Fix: P. E. 18 Jan

Densitometry: P. E. 18 Jan

25X1

Shipment 1
Box 23
Sample No. 4

EXPOSURE STATION: P.E.'s EK 1
INTENSITY: 1700 No Filters mc
EXPOSURE TIME: 200 sec.
E EXPOSURE: mc sec.
Log EXPOSURE: 4.53

RED LITE STATION: No RLD
STATIONARY PLATEN: OSCILLATING:
RED LITE DEV. TIME: sec.
TIME/PASS:
FILTERS USED: N.D.:
RADIATED POWER:

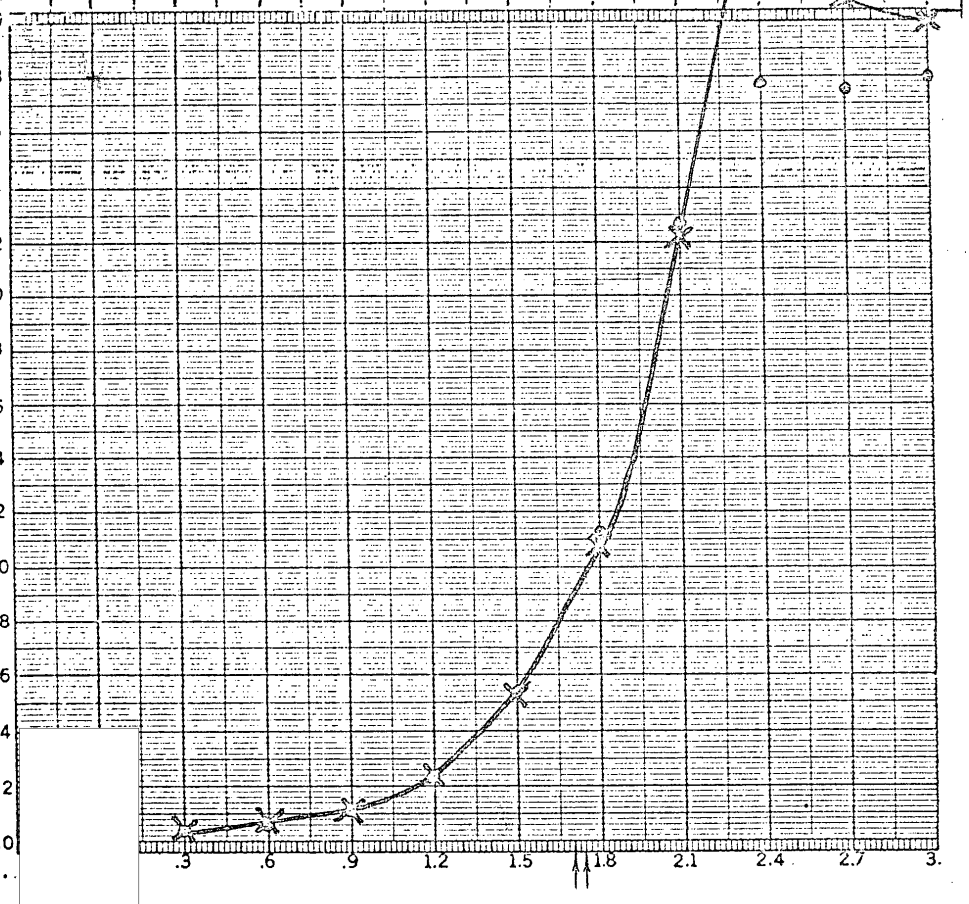
ROOM TEMP:
HUMIDITY:
PLATEN TEMP: 25X1

	Blue	Green
γ =	5.2	4.6
AEI:	2.0×10^{-4}	2.1×10^{-4}

[redacted]

B+F

B		.05	.08	.12	.25	.53	1.08	2.22	3.20	3.09	3.00
G		.04	.06	.12	.24	.54	1.13	2.27	2.78	2.76	2.80
	1	2	3	4	5	6	7	8	9	10	11



RELATIVE LOG EXPOSURE

Abs. Log E 4.53↑

FIGURE 2

TD 102 Ddensitometr[illegible] 25X1

FORMULA 5/D7

Coated: [redacted] 14 25X1
Exp. and Fix: P. E. 18 Jan.
Densitometry: [redacted] 25X1

Shipment 1
Box 23
Sample 4

EXPOSURE STATION: EK 101 (
INTENSITY: 1700 mc No Fi
EXPOSURE TIME: 200 sec.
E EXPOSURE: mc sec.
Log EXPOSURE: 4.53

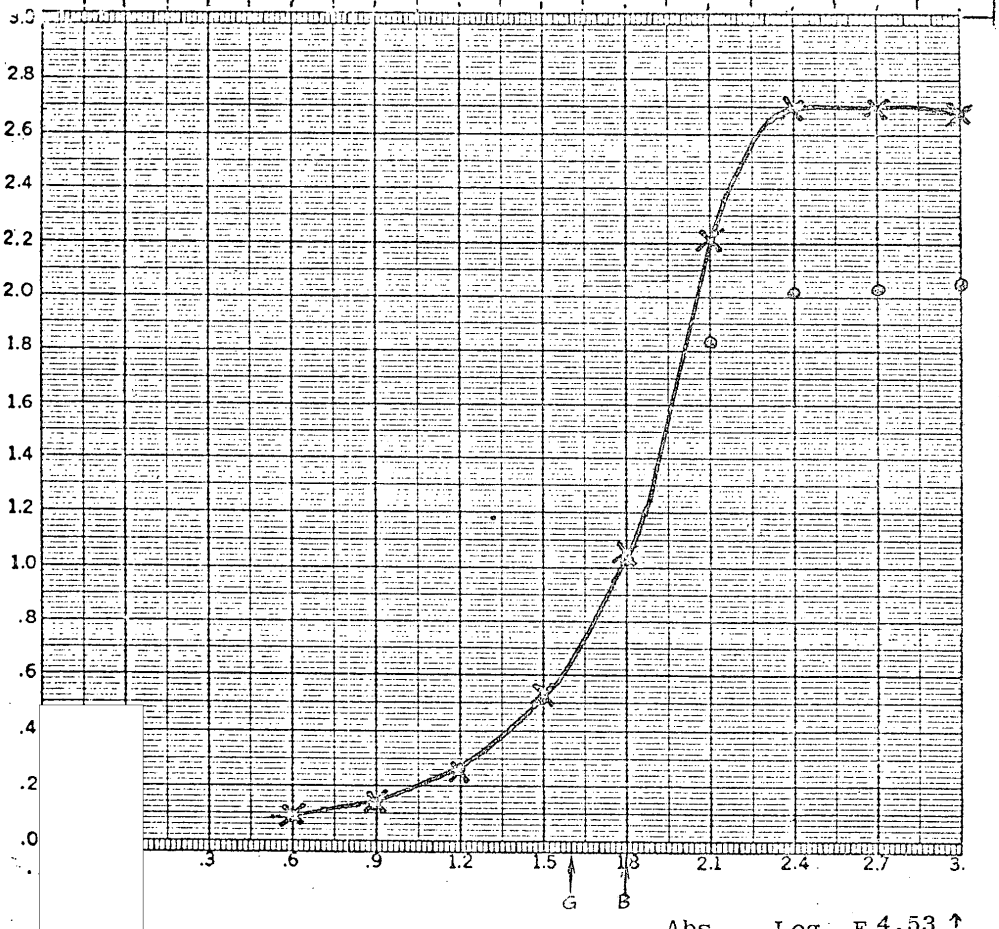
RED LITE STATION: No RLD.
STATIONARY PLATEN: OSCILLATING:
RED LITE DEV. TIME: sec.
TIME/PASS:
FILTERS USED: N.D.:
RADIATED POWER:

ROOM TEMP:
HUMIDITY:
PLATEN TEMP: 25X1

Blue	Green
γ = 4.0	3.9
AEI: 2.7×10^{-4}	2.9×10^{-4}

B+F

.10	BLUE	X	.10	.15	.26	.54	1.07	2.22	2.70	2.70	2.68
.10	GREEN	⊙	.10	.14	.27	.52	1.04	1.84	2.02	2.04	2.06
		1	2	3	4	5	6	7	8	9	10	11



Abs. Log E 4.53 ↑

FIGURE 3

325B's TD 102 DENSITOMETRY

FORMULA 5/D7

Coated: [redacted] 14 25X1
Exp. and Fix: P. E. 18 Jan.
Densitometry: [redacted] 325X1, 25X1

Shipment 1
Box 23
Sample No. 4

EXPOSURE STATION: P.E. EK 1[illegible] No F[illegible]
INTENSITY: 1700 mc
EXPOSURE TIME: 200 sec.
E EXPOSURE: mc sec.
Log EXPOSURE: 4.53

RED LITE STATION: No RLD LAMP:
STATIONARY PLATEN: OSCILLATING:
RED LITE DEV. TIME: sec.
TIME/PASS:
FILTERS USED: N.D.:
RADIATED POWER:

ROOM TEMP:
HUMIDITY: 25X1
PLATEN TEMP:

	Blue	Green
γ =	4.60	2.64
AEI:	2.3×10^{-4}	3.7×10^{-4}

25X1

FIGURE 4

COMPARISON OF 3 TD 102 DENSITOMETERS

RATIO OF $\frac{\text{BLUE FILTER DENSITIES}}{\text{GREEN FILTER DENSITIES}}$

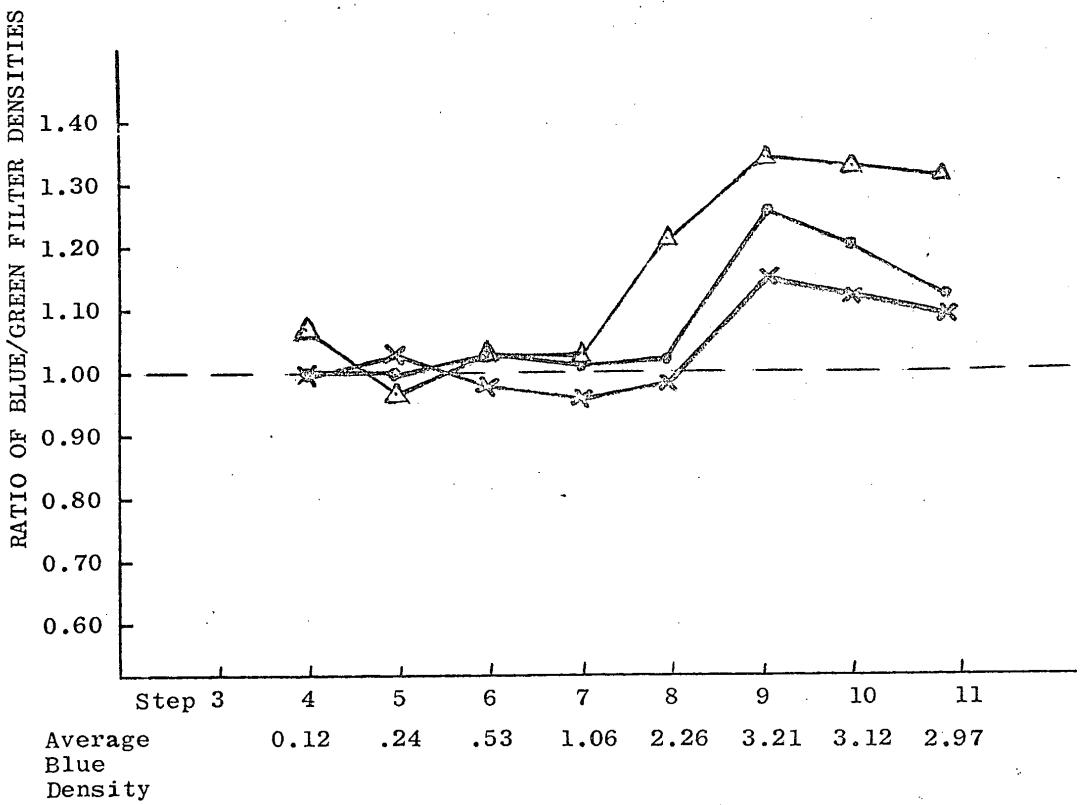
P325B 2850°K, PRINTOUT AT P. E.

⊗ Perkin Elmer's TD-102

x [redacted] TD-102

△ Project 325B's TD-102

25X1



25X1

1)	B+F	.15	.16	.17	.21	.26	.36	.50	.71	.98	1.25	1.44	o
2)		.15	.16	.18	.22	.28	.36	.51	.72	1.00	1.26	1.43	x
3)		.13	.14	.16	.20	.25	.33	.47	.68	.94	1.17	1.32	Δ
		1	2	3	4	5	6	7	8	9	10	11	

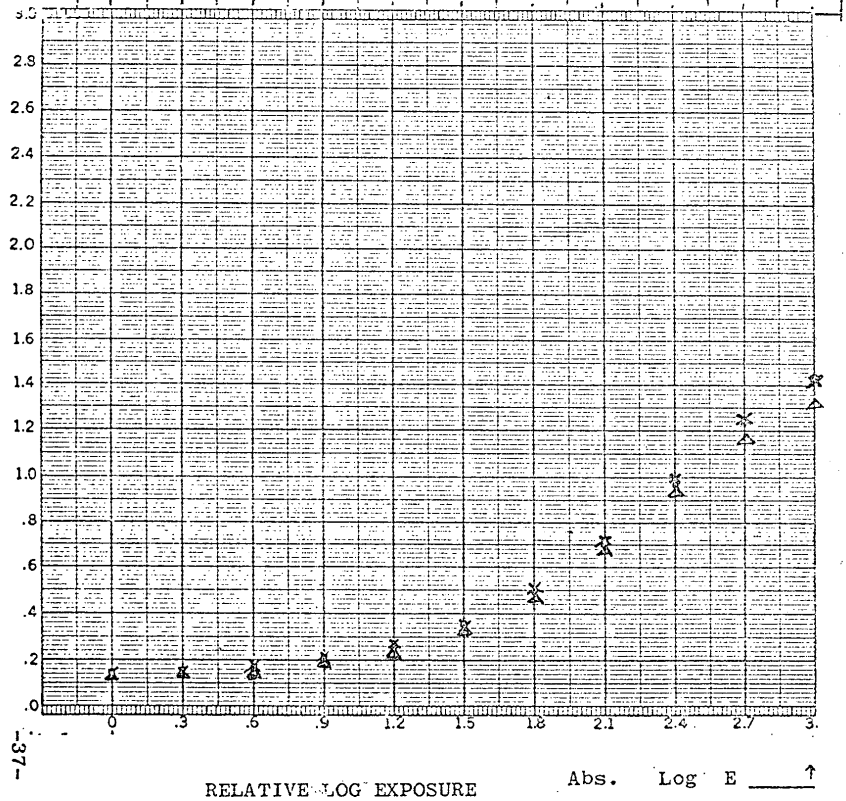
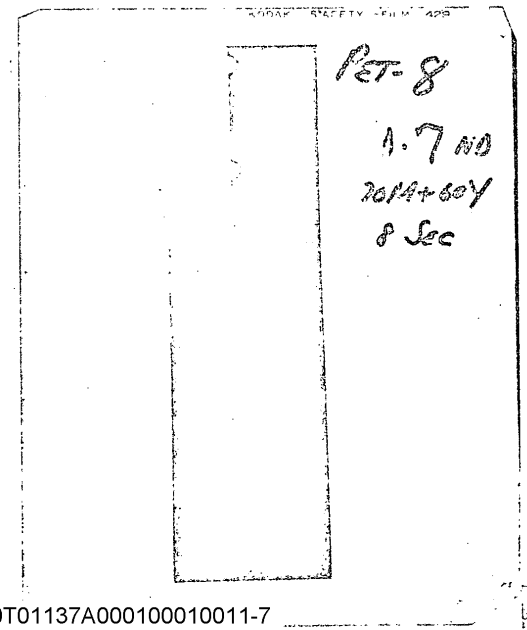


FIGURE 5

COMPARISON OF 3 DENSITOMETERS
With Blue Filter,
Ektacolor Internegative Tablet

25X1



25X1

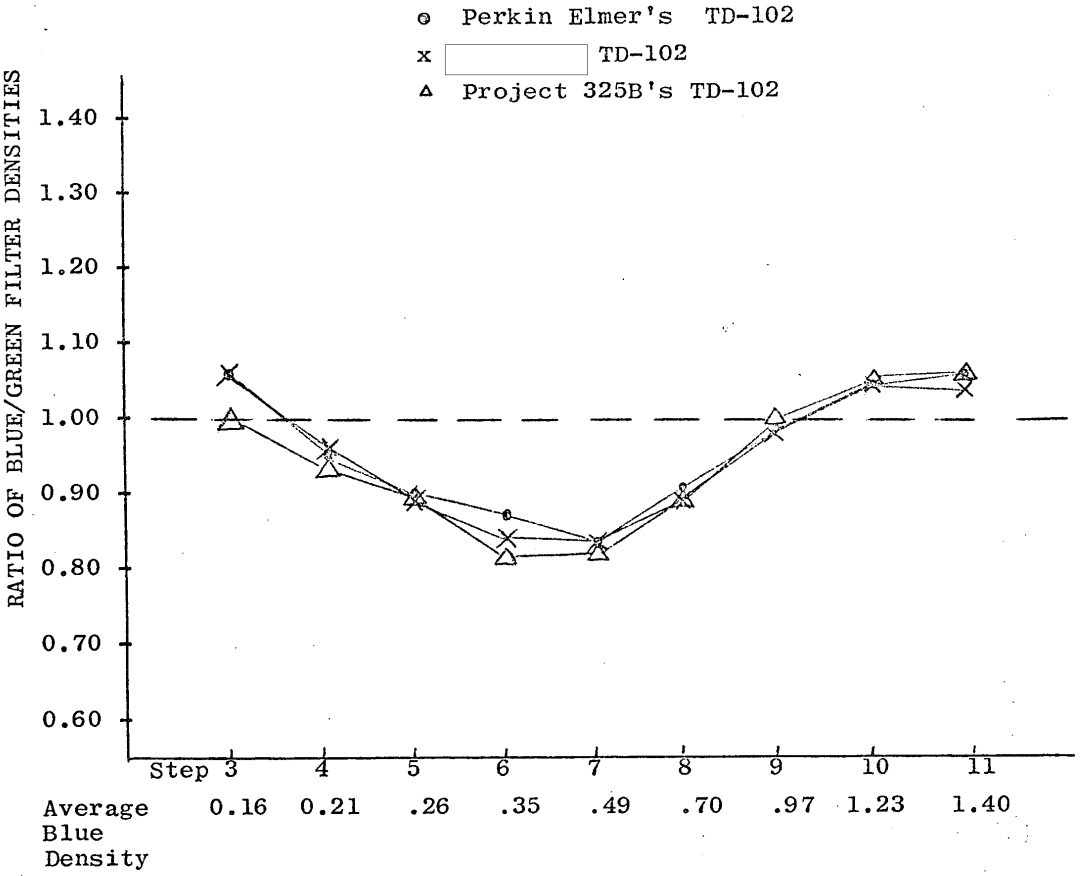
1) P.E.'s Densitometer
2) [redacted] Densitometer
3) Project 325

25X1

25X1

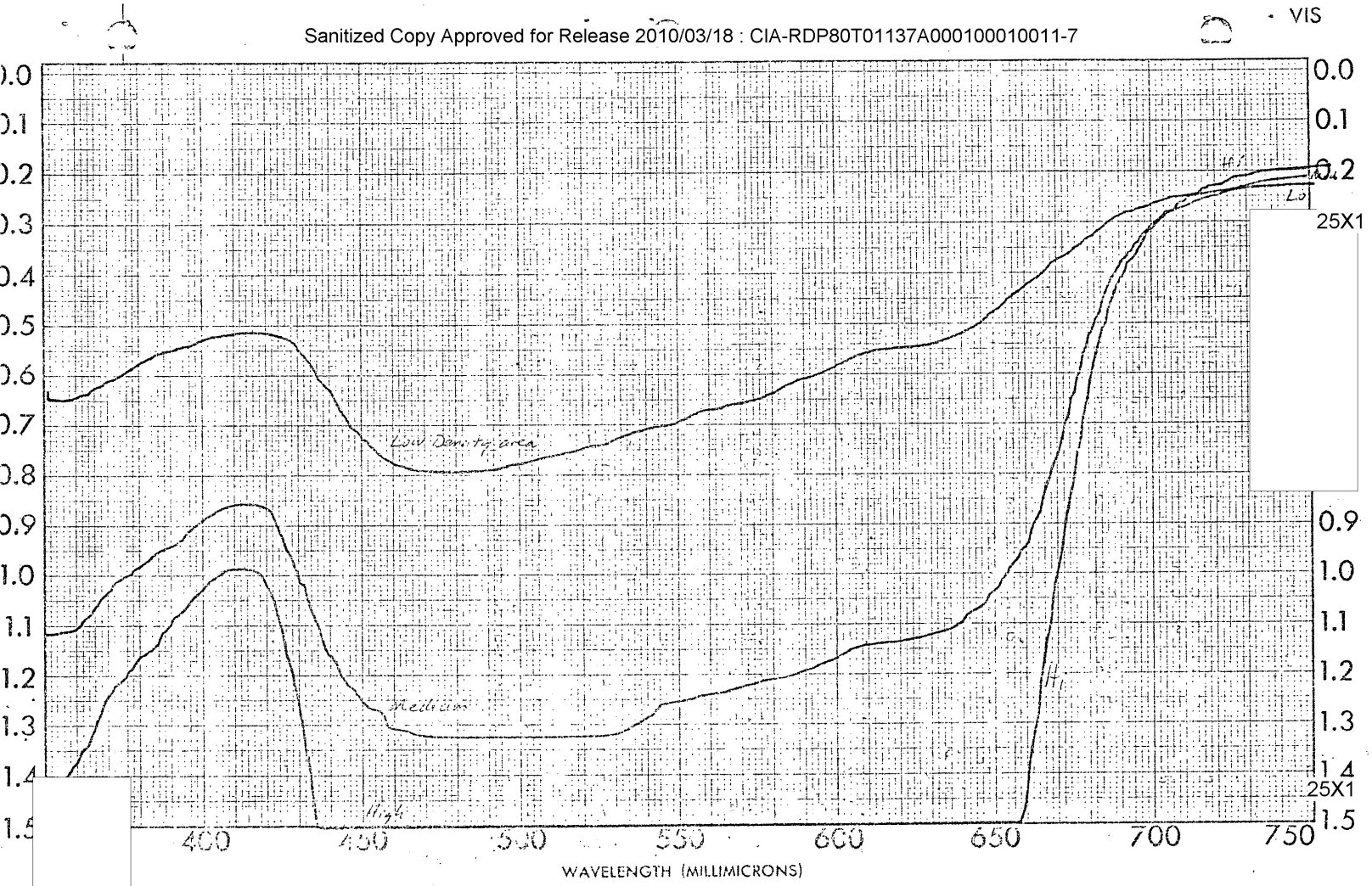
FIGURE 6

COMPARISON OF 3 TD-102 DENSITOMETERS
RATIO OF BLUE/GREEN FILTER DENSITIES
EKTACOLOR TABLET



25X1

25X1



No. 1674-24-19 325A 5/D7

edium, Low Density Segments
let.

FIGURE 7

COLOR PROFILE OF 325B FILM
at
HIGH, MEDIUM, and LOW DENSITIES

SCAN SPEED FAST
SLIT 25
OPERATOR F.W.S.
DATE 21 Apr. 1971
REMARKS

25X1

FIGURE 8

FREQUENCY OF GOOD AND BAD FILM DAYS

VS.

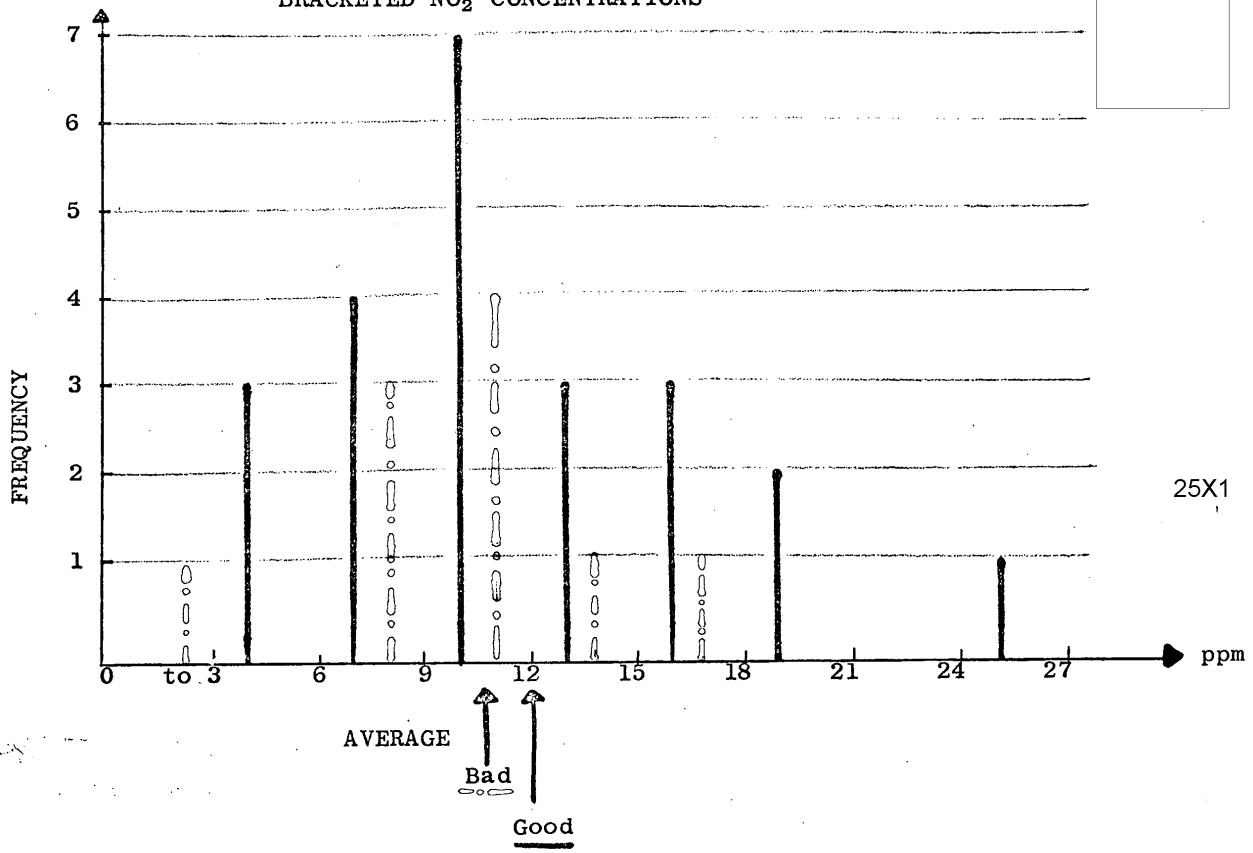
BRACKETED NO_2 CONCENTRATIONS

NO_2

Total Population

23 + 10 = 33

Ave. 12.1 10.4



25X1

25X1

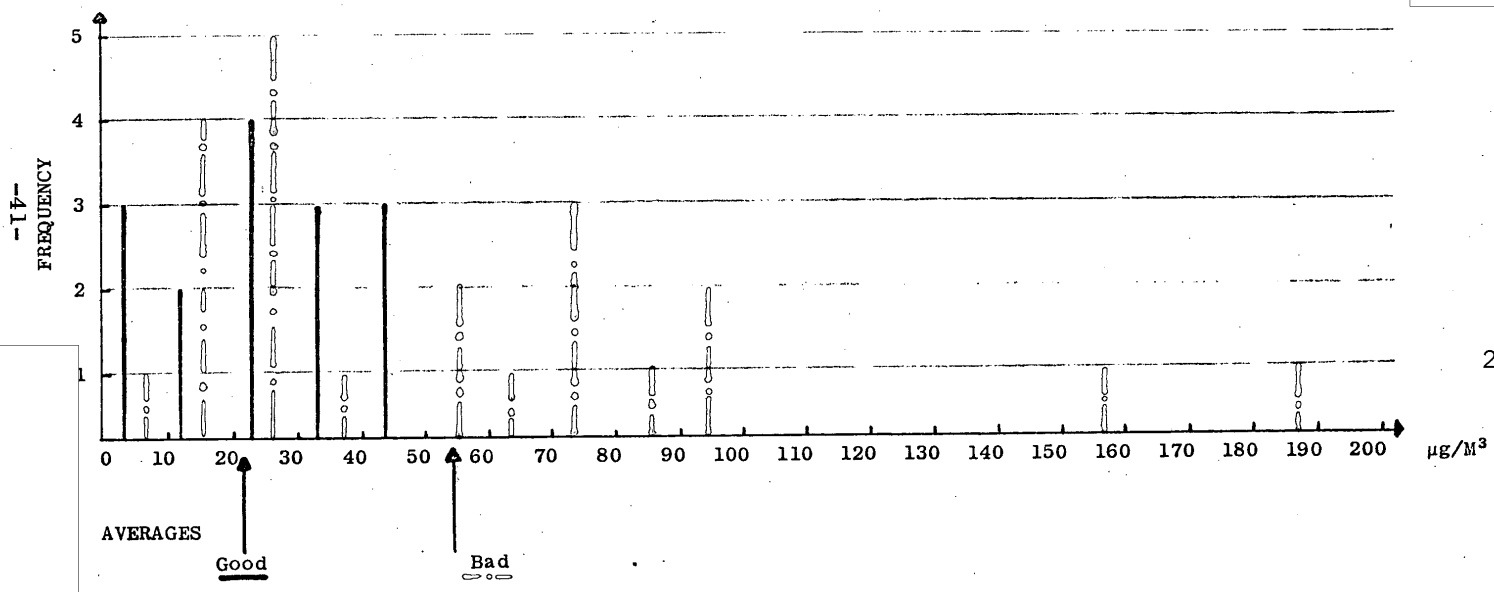
OXIDANTS

Total Population

13 + 22 = 35

AVE. 24.3 56.2

FIGURE 9
FREQUENCY OF GOOD AND BAD FILM DAYS
VS.
BRACKETED OXIDANTS CONCENTRATIONS



25X1

25X1

X_2

Total Population

19 + 16 = 35

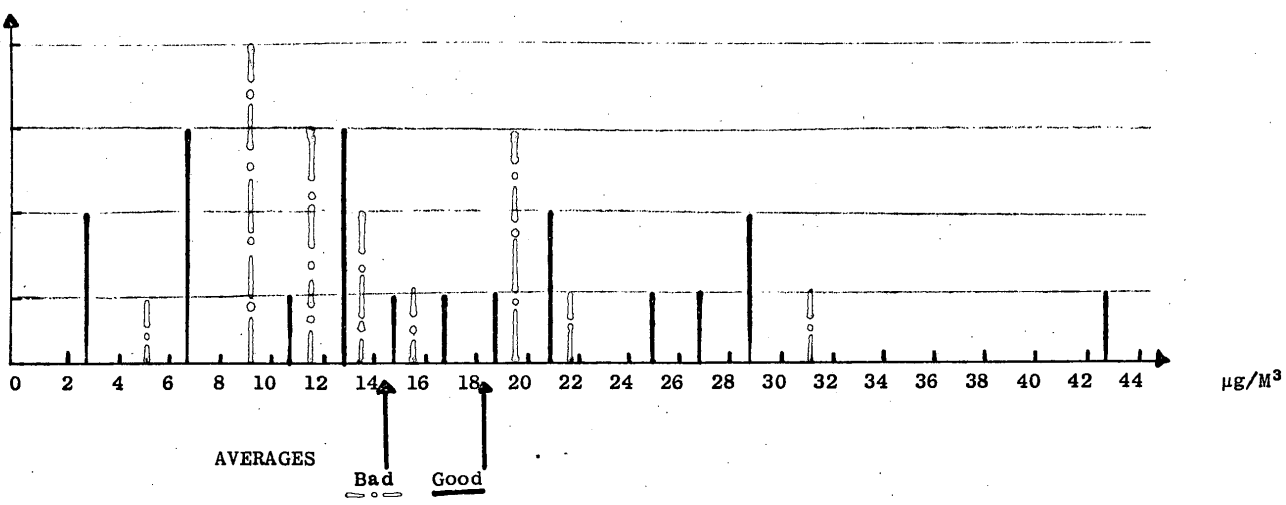
AVE. 17.5 14.6

FIGURE 10

FREQUENCY OF GOOD AND BAD FILM DAYS

VS.

BRACKETED X_2 CONCENTRATIONS



25X1

25X1

HCl

Total Population

20 + 13 = 33

AVE. 206 87.5

HCl

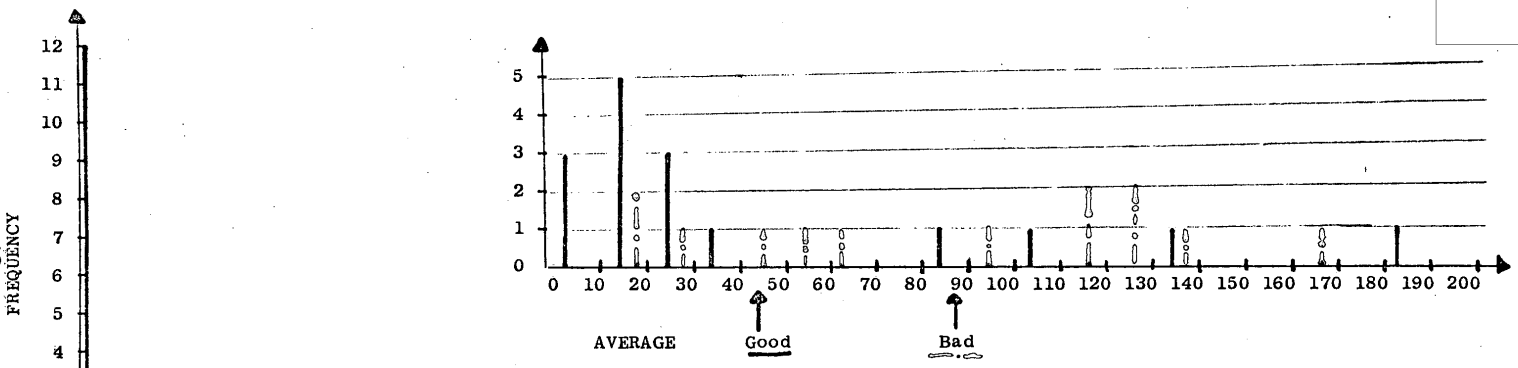
Total Population

16 + 13 = 29

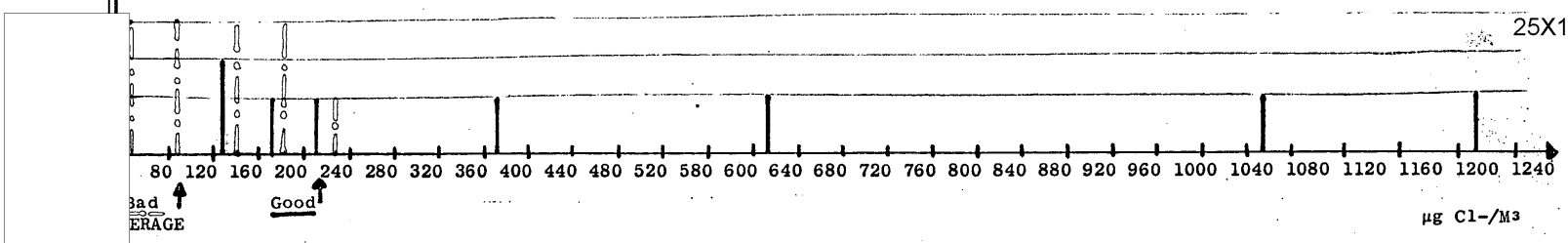
AVE. 44.7 87.5

FIGURE 11

FREQUENCY OF GOOD AND BAD FILM DAYS
VS.
BRACKETED HCl CONCENTRATIONS



25X1



25X1

HB_r

Total Population

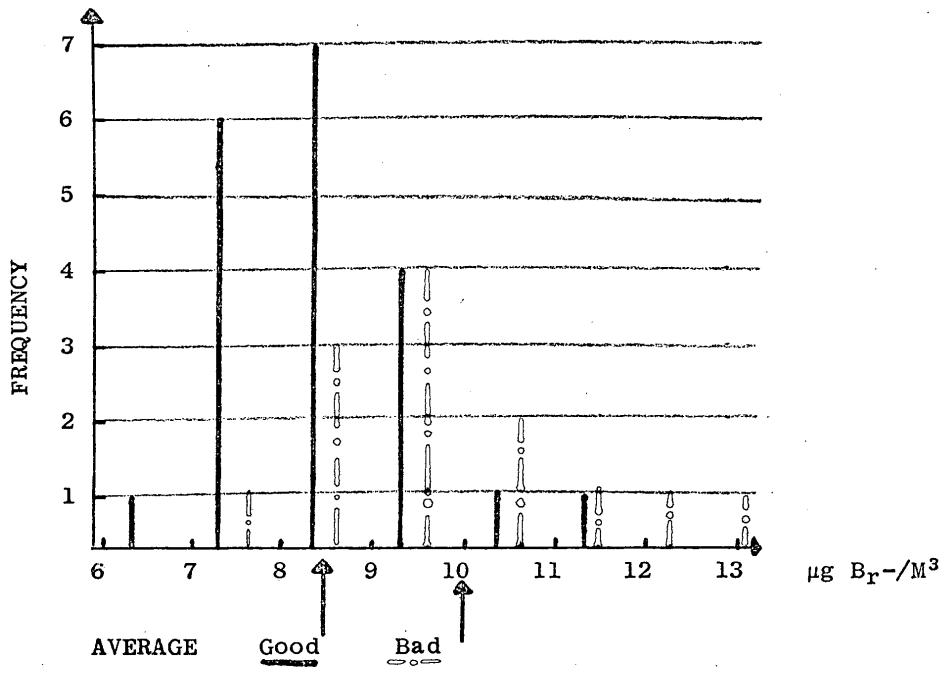
20	+	13	= 33
AVE. 8.1		9.5	

FIGURE 12

FREQUENCY OF GOOD AND BAD FILM DAYS

VS.

BRACKETED HB_r CONCENTRATIONS



25X1

PROBLEMS

1) As of February 28, written authorization to proceed not received.

2) Determining the exact air contaminants that produce film blotch.

3) Delivery of laboratory chambers for baseline speed decay measurements. Two of three chambers received were defective.

4) Shipment of film sample to Perkin Elmer.

5) Slippage of schedule on engineering experiments on reactivation and overcoating.

PLANS FOR NEXT REPORTING PERIOD

1) Press to secure three usable laboratory chambers.

2) Attempt engineering reactivation and overcoating work.

3) Continue chemical R and D work according to schedule possible with one laboratory chamber.

FINANCIAL

Project is within labor and materials forecasts.

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