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APPARATUS FOR THE STUDY OF THE PHOTOCHEMISTRY OF SHEET MATERIALS

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ABSTRACT

An apparatus is described which was developed for studies of the photochemistry of organic materials in sheet form. The problems involved in such studies are discussed, and data are presented to show the extent to which various important requirements have been met in the design of the apparatus.

One of the most important problems involved in such studies is the elimination of concurrent temperature effects. Temperatures of thin sheet materials—such as paper, cellulose acetate, Cellophane, rubber, felt cloth, and a paint film—under various conditions of irradiation with the Pyrex-enclosed electric arc, were measured with thermocouples. Temperatures as high as 280° C were found in white paper exposed to a 2,080-watt arc, at a distance of 16 cm. Under less severe conditions, some of which were similar to those of the artificial "weathering" and "light" stability tests, in use at the National Bureau of Standards and elsewhere, temperatures between 80° and 150° C were found in the various sheet materials. Since most of the materials named are known to undergo marked thermal decomposition at these higher temperatures, the control of temperature is important.

An apparatus for controlling the temperatures of sheet materials during irradiation, through intimate contact of the sheet with a thermostated aluminum backing, was found to be effective. Contact was secured by reducing the pressure between the sheet and backing. Temperature rises were thereby kept smaller; in the extreme case cited, the temperature rise was only 10° C above the temperature of the thermostated backing, which was maintained at 30° C.

Temperature rises were further minimized by removal of most of the infrared radiant energy by means of a flowing cupric chloride filter. The removal of the infrared alone was found to be insufficient for adequate temperature control, owing to the heat effects produced by the visible and near ultraviolet, freely transmitted by the filter. When the filter was used in conjunction with the thermostated backing, the temperature rises due to irradiation were only 2° to 3° C above 30° C. Because of the large amount of infrared energy to be removed, as much as 19,000 calories per minute for the arc lamp, and because of the corrosive nature of cupric chloride, special equipment was required for this filter.

An arrangement for controlling the moisture and oxygen content of the atmosphere around the samples, from normal down to very low values, is also necessary for a comprehensive study. For porous materials, such as paper, in which gas absorption on the extensive fiber surfaces of the interior of the sheet may be important, direct circulation of the gases through the sheet is desirable.

CONTENTS

I. PROBLEMS INVOLVED IN STUDIES OF THE PHOTOCHEMISTRY OF SOLID MATERIALS

Studies of the photochemistry of "solid" materials present problems which are peculiar to and at the same time common to practically all solid organic materials. In the present publication an apparatus is described which was designed to meet various important common requirements of studies of the photochemistry of organic materials in sheet form.

One of the most important problems involved in the photochemical study of paper, plastics, rubber, etc., is the control of the temperature of the exposed materials. Temperature control is necessary to avoid not only the chemical and physical changes due to heat, but also to avoid secondary effects of high temperature upon the content of moisture and other volatile components, which may result in changes not directly photochemical or in conditions which affect the normal course of the photochemical reaction.

Inasmuch as solid materials of the type named are relatively stable to ordinary intensities, sources of high intensity are necessary to produce measurable photochemical changes in reasonably short periods of time. Such sources, however, cause heating of the sample under irradiation. Part of this heating may be avoided by removing the undesired portion of the spectrum, for example the infrared, by filters before it reaches the sample; but the heating accompanying absorption of the desired portion must be lessened by removing heat from the sample as rapidly as it appears, in order to secure control of the temperature of the sheet material.

The choice of a light source is governed by the absorption characteristics of the particular material studied. All practical light sources, however, radiate large amounts of infrared. For example, over 64 percent of the radiant energy from the solid carbon, Pyrex-enclosed arc lamp lies in the infrared region beyond 750 mμ. A filter capable of absorbing such large quantities of infrared, approximately 19,000 cal/min. for an arc operating at 2,080 watts, without seriously diminishing the intensity of the desired visible and ultraviolet regions, must be free from physical and chemical changes due to light, and must permit the dissipation of the absorbed energy.

Aside from temperature control, another important problem in exact photochemical studies is the necessity of measuring the incident radiant energy. This is relatively simple when the source is constant in intensity, but such a useful source as the carbon arc varies, in its minute to minute average, from the mean intensity by as much as 15 percent. Apparatus for the continuous measurement and recording of high radiant intensities is, therefore, desirable in order to obtain a curve or function which may be integrated to obtain mean intensity. From the incident radiant energy, the absorbed radiant energy may

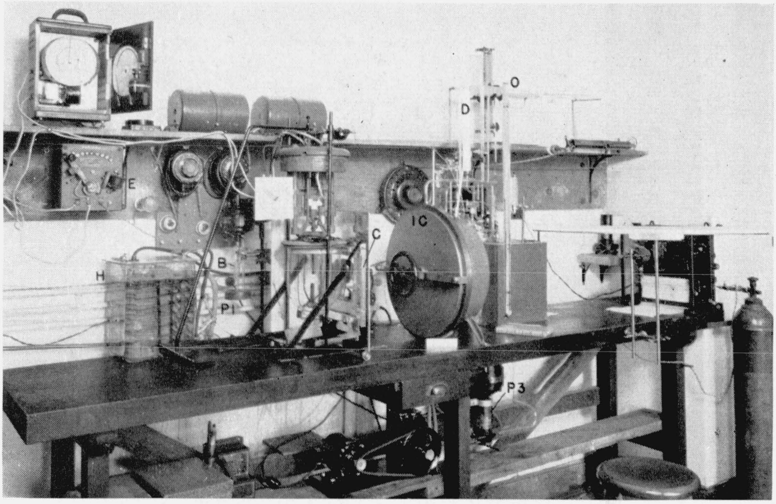

FIGURE 1.—*The photochemical apparatus.*

(For significance of letters see legends under fig. 2.)

be calculated for any material for which the reflection and transmission factors are known. In this respect, each sheet material presents a special problem.

Another problem is the control of moisture and oxygen content of the samples, if such factors are found to have an effect upon the photochemical action. In any case, the fact must first be established; thus, a system is necessary in which the samples during exposure are in contact only with oxygen-free or moisture-free gases, as the case may be. To accomplish this the gases must be continually maintained in the desired condition by circulation through a conditioning apparatus, not only to remove a given substance to determine the effect of its absence—water vapor, for example, which continues to diffuse to the surface after the surrounding gases have been dried—but also to remove water vapor concurrently formed as a product of the photochemical reaction. In this connection, it would appear highly desirable to secure a circulation of the purified gases directly through the material being irradiated, at least through fibrous materials, in order to remove moisture and oxygen adsorbed on the extensive fiber surfaces within the sheet.

The various factors which have just been discussed were taken into account in the design of the apparatus to be described, and data are presented which show the extent to which the requirements have been met.

II. LIGHT SOURCE AND MEASUREMENT OF RADIANT ENERGY

The light source, figure 1 (in this case a carbon arc lamp), together with filters and reflectors, was mounted rigidly on a steel carriage which could be moved back out of the way to permit changing of samples and which was replaceable at the same distance, ±0.1 percent, from the sample being irradiated.

Under normal operation, the arc was 15.8 cm from the middle of the sheet under irradiation. A diffusing reflector of etched aluminum, not shown in figure 1, enclosed the back and sides of the glass globe of the lamp and extended forward to the cupric chloride absorption cell, *C*, described later.

For the continuous recording of the incident energy, a Leeds and Northrup recorder, equipped with a 100-ohm galvanometer, was used directly in conjunction with a thermopile. The continuous measurement over long periods of time of direct and diffusely reflected incident radiant energy of high intensity required a thermopile of special design. An instrument capable of intercepting the energy incident from a wide solid angle was essential. Further requirements were negligible zero shift, ruggedness, minimum area, and minimum thickness. Such a thermopile was described by the writer elsewhere [1].[1]

For control purposes, the thermopile was mounted as shown in figure 4. For the purpose of determining the average incident intensity over the irradiated surface (400 cm^2), the thermopile was movable to any position on the surface.

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[1] Figures in brackets indicate literature references at the end of this paper.

III. ABSORPTION OF INFRARED RADIANT ENERGY

Cupric chloride solution has been found by Coblentz [2, 3] and others to be effective in absorbing the infrared beyond 750 μ without undergoing change in transmissive properties over a long period of time. Some absorption of ultraviolet (and red) also occurs, but this can be regulated by suitable choice of the concentration of the solution. An 11-mm layer of cupric chloride solution, 2 percent in $CuCl_2 \cdot 2H_2O$ (0.117 molar) and 0.015 molar in HCl, was found to be satisfactory, and its transmission in a cell with windows of Corex-*D* glass is given in table 1. Table 1 also shows the change in spectral quality of a carbon arc effected by the filter.

TABLE 1.—*Transmission of an 11-mm layer of 2 percent cupric chloride in a Corex-D cell, and the effect upon the spectral distribution of radiation from the Pyrex-enclosed carbon arc*

Wavelength interval [a]	Transmission of cupric chloride solution in Corex-*D* glass cell	Spectral distribution of arc lamp [b]	Spectral distribution of transmitted radiant energy
Millimicrons	*Percent* [c]	*Percent*	*Percent*
290 to 320	1.9	4.6	0.4
320 to 750	63.0	32.3	96.5
750 to 1,400	5.3	12.1	3.1
1,400 to 12,000	0.0	52.0	0.0

[a] The transmissions in the ultraviolet and visible regions were found by R. Stair to be as follows, for wavelengths in millimicrons and transmissions in percent. 302:0.5; 313:3.5; 334:40; 365:78; 405:88; 436:90; 546:86; 579:80. The transmissions for the infrared region were calculated from the data of Coblentz [2, 3]. The averaged transmissions in column 2 were obtained by integrating the curve plotted from the data so obtained.

[b] The spectral distribution of radiant energy emitted by the carbon arc lamp was taken from data furnished by the manufacturer, and the percentages are based upon the total emission in the region 290 to 12,000 mμ.

[c] Based upon each wavelength interval. For this reason, these percentages do not equal 100.

By circulating the cupric chloride through a heat exchanger, *H* (figs. 1 and 2), which consisted of 50 feet of glass tubing immersed in a water bath maintained at a constant temperature, the heat arising from the absorption in cell *C* (fig. 1), was dissipated.

Since cupric chloride reacts rapidly with most metals, all parts with which it came in contact were of glass or rubber. The absorption cell, *C* (fig. 1), consisted of two sheets of Corex-*D* glass, 2 mm thick, pressed against a rubber gasket, giving a closed cell, except for the inlet and outlet tubes. Corex-*D* glass was selected primarily because of its ability to withstand extreme heat-shock, and because of the small possibility of a decrease in transmission in the region between 320 and 400 mμ through solarization [4].

The pump, *P1*, shown in figure 3, consisted of a ⊥-tube, rotated about the vertical axis, so that the crosspiece produced circular motion of the liquid in a shallow, cylindrical case, causing the liquid to enter at the center hole and to leave the case through the side tube, *M*, which also served as support for the case. The case, *R*, was machined from hard rubber and during operation was immersed in a beaker (not shown) containing the cupric chloride solution. The evaporation which occurred at this point, 50 ml during a 17-hr period, was compensated by a device which allowed water to drip at this rate into the beaker.

The pump, driven by the 1/30-hp fan motor shown, was capable of pumping a 7-ft head of water. Running at "low" speed, the pump circulated the solution through the system, including heat exchanger, at the rate of 700 ml/min. At this, the usual rate, the average temperature of the solution in the cell *C* was 35° C. A device, *B*, for breaking the arc-lamp circuit, if for any reason the flow of cupric chloride should cease, is also shown in figure 3.

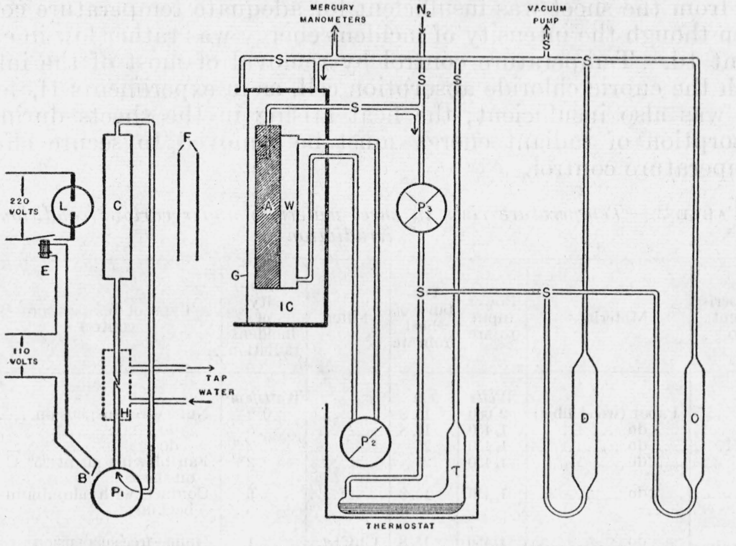

FIGURE 2.—*Schematic drawing of the photochemical apparatus.*

L=light source.
E=electromagnetic circuit breaker.
B=control mechanism for *E*.
C=cupric chloride absorption cell for infrared.
H=heat exchanger.
P1=cupric chloride circulating pump.
F=glass light filters.
G=glass (Corex-*D*) window—4-mm thickness.
AW=thermostated aluminum backing for sheet materials.
IC=irradiation chamber.
P2=circulating pump for water in jacket of aluminum backing.
P3=mercury-sealed circulating pump for gases in *IC*.
T=tube containing saturated NaBr, for constant moisture content.
D=dehydrating tube containing anhydrous magnesium perchlorate.
O=oxygen remover containing copper metal at 500° C.
S=stopcocks.

IV. CONTROL OF THE TEMPERATURE OF SHEET MATERIALS

1. NECESSITY OF TEMPERATURE CONTROL

(a) TEMPERATURE RISE UNDER TYPICAL CONDITIONS OF IRRADIATION

The necessity of removing heat from materials during irradiation with high-intensity light sources is shown in the following experiments. Typical "sheet" materials, including paper, cellulose acetate, Cellophane, rubber, wool felt, and a paint film, were exposed to high-intensity illumination under various conditions. Some of these conditions were similar to those prevailing during arc-lamp "weathering" and artificial "light"-aging tests used at the National Bureau of Standards and elsewhere. Temperature rises above 30° C due to irradiation were measured by means of thermojunctions embedded in the sheet materials.

The temperature rise during irradiation is shown in table 2 and is seen to be large when no effort was made to control the temperature of the sheets, as in experiments 1a, 1b, 1c, 5b, 5c, 7b, 7c, 8, and 9. Even a rapid current of cool air from a large electric fan located 30 cm from the sheet was insufficient for adequate temperature control, even though the intensity of incident energy was rather low in experiment 1d. Temperature control by removal of most of the infrared with the cupric chloride absorption cell, as in experiments 1f, 4c, and 6c, was also insufficient; the heat arising in the sheets during the absorption of radiant energy must be removed to secure effective temperature control.

TABLE 2.—*Temperature rises in sheet materials under various conditions of irradiation* [a]

Experiment No.[b]	Material [c]	Power input to arc	Distance of sheet from arc	Filter	Intensity of incident radiation	Type of temperature control	Temperature rise above 30° C
		Watts	*cm*		*Watts/cm²*		*° C*
1a	Paper (wood fiber)	2,080	15.8		0.9	None—free suspension	[e]250
1b	do	1,450	15.8		.5	do	172
1c	do	1,450	25		.2	do	[f]79
1d	do	1,450	25		.2	Fan blowing air at 25° C on sheet.	43
1e	do	1,450	15.8		.5	Contact with aluminum backing.	10
1f	do	1,450	15.8	$CuCl_2$[g]	.1	None—free suspension	49
1g	do	1,450	15.8	do	.1	Contact with aluminum backing.	1.3
1h	do	2,080	15.8	do	.17	do	2.3
2a	Paper[d] (cotton fiber).	2,080	15.8	do	.17	do	2.0
3	do	2,080	15.8	do	.17	do	2.2
4a	Cellulose acetate	2,080	15.8	do	.17	do	2.4
4b	do	2,080	31.6	do	.04	do	0.5
4c	do	2,080	15.8	do	.17	None—free suspension	15
5a	do	2,080	15.8	do	.17	Contact with aluminum backing.	3.7
5b	do	2,080	25		.4	None—free suspension	[f]71
5c	do	2,080	38		.2	do	[f]32
6a	Rubber (raw)	2,080	15.8	$CuCl_2$	.17	Contact with aluminum backing.	3.0
6b	do	2,080	38	do	.03	do	0.7
6c	do	2,080	38	do	.03	None—free suspension	11
7a	Lacquer (dark gray).	2,080	38	do	.03	Contact with aluminum backing.	1.1
7b	do	2,080	25		.4	None—free suspension	[f]80
7c	do	2,080	38		.2	do	[f]52
7d	do	2,080	38	$CuCl_2$	.03	do	13
8	Cellophane	1,450	25		.2	do	[f]81
9	Felt (black)	1,450	25		.2	do	[f]120
2b	Paper (cotton fiber).	Direct sunlight[h]			.075	do	8

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[a] A Pyrex-enclosed arc lamp with ½-in. solid-carbon electrodes was used in all experiments except 2b.

[b] Experiments with the same number were carried out with the same specimen; differing letters indicate different conditions.

[c] The average thicknesses of the sheets, the thickness of material in front of, and the thickness of material in back of the thermojunctions, respectively, were as follows, in millimeters: No. 1—0.10, 0.03, 0.03; No. 2—0.11, 0.03, 0.03; 3—0.14, 0.03, 0.04; No. 4—0.05, 0.025, 0.025; No. 5—0.21, 0.05, 0.05; No. 6—0.62, 0.31, 0.18; No. 7—0.35, 0.11, 0.11; No. 8—0.15, 0.075, 0.075; No. 9—1.5, 0.75, 0.75.

[d] The papers made from cotton fibers were whiter than the one made from wood fibers.

[e] Paper started to char after a few minutes.

[f] The conditions prevailing in these experiments are representative of the routine "weathering" tests in use at this Bureau and elsewhere. Higher intensities are often used.

[g] An 11-mm layer of 2 percent $CuCl_2$ (see text) was used.

[h] August 22, 1939; 11:30 to 11:50 a. m. Air temperature 26° C. Bright sun. Mild breeze.

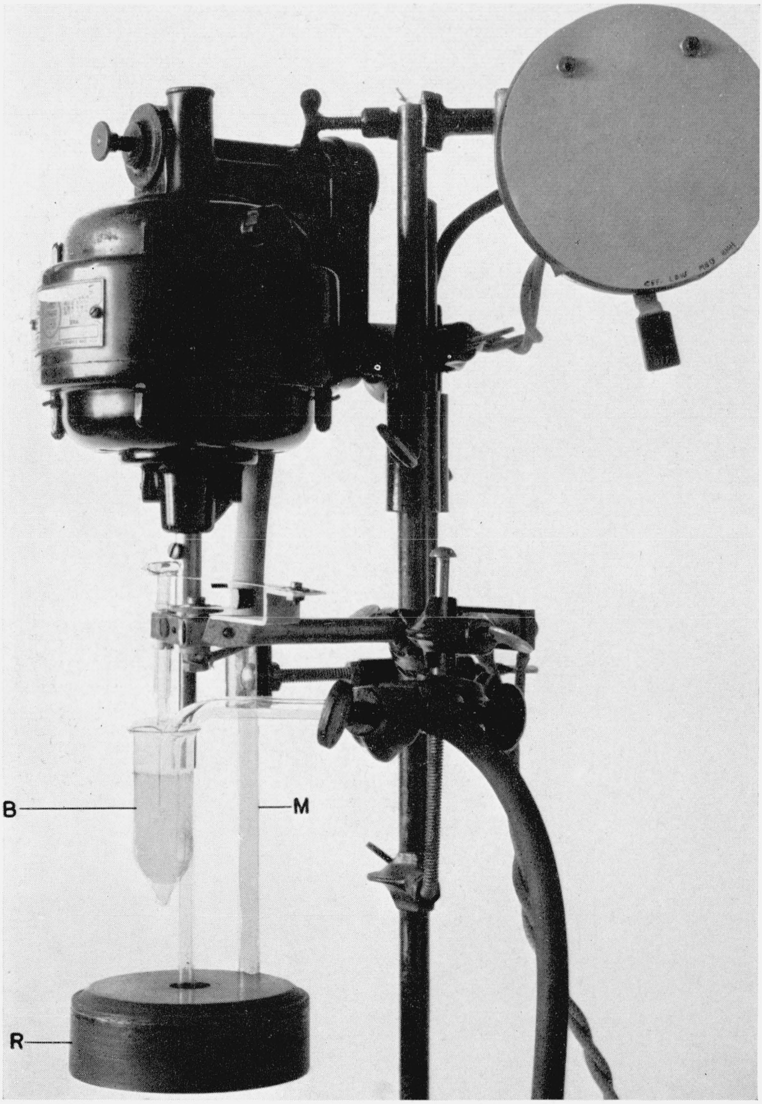

FIGURE 3.—*Hard rubber-glass circulating pump for corrosive liquids, and control mechanism for circuit breaker.*

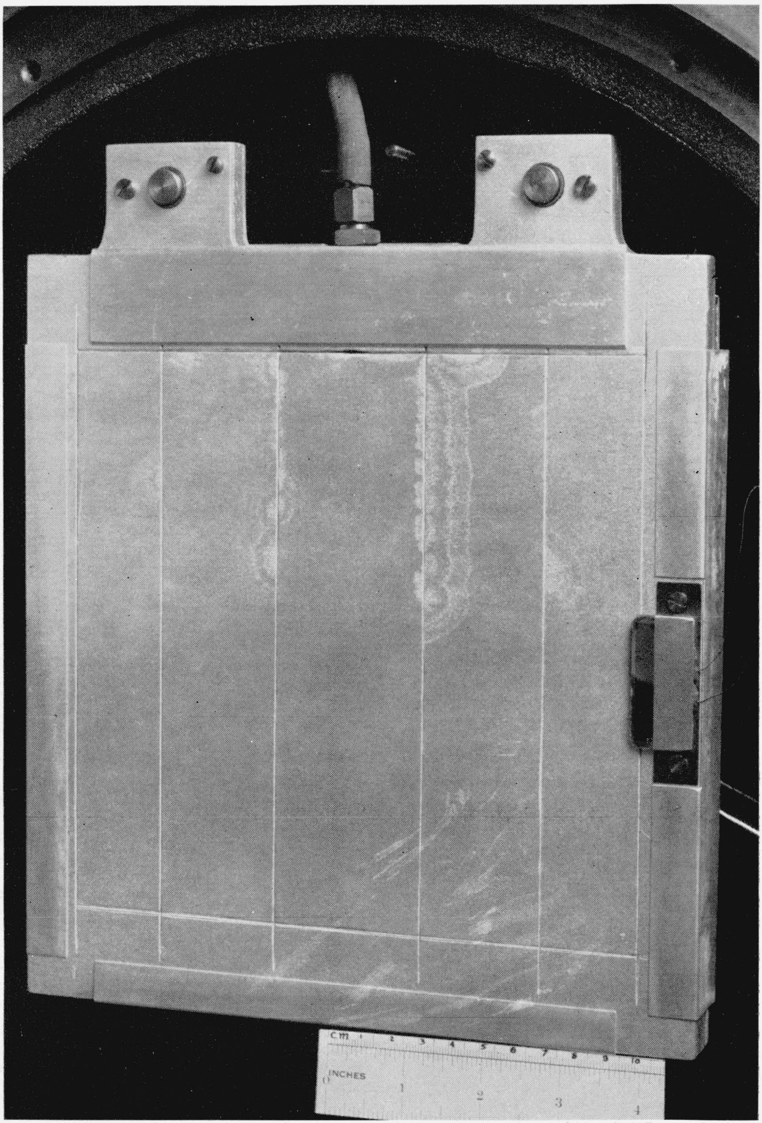

FIGURE 4.—*Thermostated aluminum backing and thermopile.*

(b) TECHNIQUE OF MEASURING TEMPERATURES OF THE INTERIORS OF THE SHEETS

The temperatures of the interiors of the sheet materials listed in table 2 were measured by means of constantan-Chromel *P* (AWG gage No. 36 wires, 0.127 mm) thermocouples. One junction was kept in a thermostated bath at 29.92° ±0.07° C, and the other embedded in the sheet. The electric circuit was completed by a galvanometer and a series resistance of 9,900 ohms. Deflections were read from a scale at 1-m distance, which was calibrated for various temperatures. The temperature-deflection curve was practically a straight line from 20° to 300° C. Temperature readings were reproducible to at least 0.3° C. This is not necessarily the reproducibility of the values in table 2, since the measured temperatures changed with fluctuations in the intensity.

The thermojunctions were incorporated in the sheets during their making, with the exception of the felt, into which the junction was threaded with a sewing needle. The film of lacquer was applied to 0.4-mm aluminum sheeting.

2. APPARATUS FOR CONTROL OF TEMPERATURE OF SHEETS

The thermostated aluminum backing is shown in figure 4, and the backing is schematically represented by *AW* in figure 2. The temperature of the 13-mm aluminum slab, *A*, was controlled by pumping water from a thermostat through the water-jacket, *W*, provided with baffles extending inward from the edges, so that the thermostat water had to follow a devious path over practically the entire rear surface of slab *A*. Circulation of the thermostat water, at the rate of 1 liter/min, was maintained by pump *P2*, of the same type as that used to circulate the cupric chloride solution, figure 3, but constructed of copper instead of hard rubber. The temperature of the water returning to the thermostat differed by 0.1° C from the temperature of the thermostat (29.92°±0.07° C) when the air surrounding the backing was 26° C. Under experimental conditions the temperature of the air in the irradiation chamber, *IC*, was usually 30° C.

Intimate contact between the sheet and the metal backing was obtained by reducing the air pressure between the two. A network of grooves, 1 mm wide, milled in the aluminum surface to a depth of 1 mm, communicated with a hole drilled in the top of the slab, which was tapped and connected with brass fittings to the rubber tube, which in turn was connected to a glass tube leading out of the irradiation chamber. This tube led to pump *P3*, figures 1 and 2, which was a small commercial water-cooled air pump. Pump *P3* not only served to reduce the pressure in the grooves of the aluminum backing when a sample for irradiation was in place, but also circulated the gases through one of a variety of conditioning systems before the gases were returned to the space surrounding the sample. The arrows in figure 2 indicate direction of flow. These conditioning systems will be described in a later section.

The aluminum surface was etched with a solution of sodium hydroxide–sodium fluoride, which produced a surface of high and constant diffuse reflectance for the visible and ultraviolet regions [5]. Aside from the optical advantages, the etching process produced an additional network of minute crevasses which facilitated the passage of air to the grooves. Thus, air pressure was exerted over the entire surface of the sheet.

3. EFFECTIVENESS OF THE APPARATUS FOR CONTROLLING THE TEMPERATURE

When the sheet materials were in intimate contact with the flat surface of the aluminum backing, *A*, it was found that heat was removed satisfactorily and adequate temperature control during irradiation was secured, as in experiments 1g, 1h, 2a, 3, 4a, 4b, 5a, 6a, 6b, and 7a, table 2. Even when a large amount of long-wavelength infrared, which is strongly absorbed by paper, was incident upon the sample, as in experiment 1e, the contact with the aluminum backing sufficed to remove most of the heat from the sheet and thus to secure fair temperature control. The conditions prevailing in experiments 1g, 1h, 2a, 3, 4a, 5a, 6a, and 6b are the usual experimental conditions and indicate the effectiveness of the arrangement for controlling temperatures of sheet materials.

Adequate temperature control was obtained whether the material allowed the passage of air through it or not. In the case of papers, air was found to pass through the pores of the sheet at the rate of 4.8 to 5.9 liters per minute (measured at 1 atmosphere and 26° C) for papers having an air permeability of 32 to 105 $\frac{\text{cm}^3/\text{sec}}{\text{m}^2(\text{g/cm}^2)}$, as measured in the instrument developed by Carson [6]. The edges of the sheets were lightly clamped to prevent curling. The upper limit of air permeability, beyond which insufficient pressure differential for adequate contact is secured in the present apparatus, appears to lie in the region between 5,000 to 10,000 $\frac{\text{cm}^3/\text{sec}}{\text{m}^2(\text{g/cm}^2)}$, that of coarse filter papers. The temperature of porous textile materials might be controlled by clamping them against the metal backing with a sheet of suitable glass.

4. SOURCES OF ERROR IN THE TEMPERATURE MEASUREMENTS

The conduction of heat to or from the thermojunction along the wires, discussed by Roeser and Mueller [7] along with similar problems arising in the use of thermocouples, was minimized by embedding the wires to a distance of approximately 10 cm on each side of the junction in the sheet, all of which was irradiated. Thus, temperature gradients in the embedded wires were probably negligible. The wires, furthermore, had low thermal conductivities (Chromel *P* 0.04, and constantan 0.05 $\frac{\text{cal/sec cm}^2}{^\circ\ \text{C/cm}}$).

If a thermojunction absorbs more radiant energy than the sheet material, which may be the case for highly transmissive sheets, the temperature of the junction may be higher than that of the sheet, but the difference will be negligible if the thermal conductivity from the junction to the surrounding material is great enough to offset the greater absorption by the junction. The fact that the temperatures of all the sheets, irrespective of their transmissive properties, approach the temperature of the aluminum backing when the latter is brought into action is an indication that absorption by the thermojunction is not an important factor and that the temperatures in table 2 are sufficiently correct to warrant the conclusions based upon them.

The temperatures recorded in table 2 refer to the interiors of the sheets. Calculations based upon thermal conductivity data show, however, that the temperature of the foremost surface, the hottest part, cannot be essentially different from that of the interior. From the coefficients of thermal conductivity—0.0003 for paper, 0.0006 for cellulose acetate [8], 0.0003 for raw rubber [9], in $\frac{\text{cal/sec cm}^2}{{}^\circ\text{C/cm}}$,—from the thickness measurements given in table 2, footnote c, and from the incident intensities, multiplied by the absorption values, it can be calculated that the temperature differences between the front and rear surfaces of the sheets in experiments 2a, 4a, and 6a were probably no greater than 0.4°, 0.02°, and 5.9° C, respectively. These values were computed by assuming all heat to be applied at the front surface, whereas, actually, the radiant energy was distributed throughout the thickness in accordance with Lambert's law. Thus, the actual differences were smaller than those calculated. In any case, the interior temperatures were not essentially different from those of the exposed surfaces, with the possible exception of that of the rubber sheet, which was made especially thick because of mechanical difficulties encountered in the incorporation of the thermojunction. In photochemical experiments, the temperature of rubber sheets of 0.1-mm thickness, or thereabouts, could be controlled readily by the method described.

V. IRRADIATION CHAMBER AND COMPOSITION OF THE ATMOSPHERE SURROUNDING THE SAMPLES DURING IRRADIATION

The irradiation chamber is shown in figure 1. It consisted of a welded steel case screwed into a cast-iron flange, the front surface of which was machined to provide three concentric ledges. The window, Corex-*D* glass, was cemented to a large steel ring with litharge-glycerine putty. When the steel ring was screwed against the center metal ledge, the pressure of the glass against a closed-pore sponge-rubber gasket, cemented to the inside ledge, provided an airtight seal, as shown by the data of the analyses of the gases.

The outside ledge, machined in the flange, provided a seat for a steel cover with rubber gasket. As shown in figure 1, the steel cover could be clamped tightly against the chamber by means of a crossbar and screw jack. With the cover in place, it was possible to evacuate the irradiation chamber, in spite of the fragility of the window (thickness=4 mm), by concurrently evacuating the space between the metal cover and the outside of the window through a hole drilled into the top of the rim. This hole was connected through a large stopcock to the glass tube which led to the chamber. The arrangement is indicated in figure 2. Although all experiments were carried out at atmospheric pressure, variations in the partial pressures of moisture or oxygen were obtained by evacuation to approximately 1 mm and refilling with the desired gas.

Without the metal cover, the window would probably have to be 25 to 30 mm thick in order to withstand a pressure differential of 1 atmosphere. In such thickness, Corex-*D* glass would show pronounced absorption in the near ultraviolet.

Two mercury manometers indicated the pressures: One, a small closed-end U-manometer indicated the pressures near 1 mm Hg; the other, a large open-end U-manometer indicated all pressures concerned, but was used for indication near and at atmospheric pressure.

Pump *P3*, as was mentioned previously, circulated gases through the various conditioning systems at rates varying from 4.8 to 5.9 liters/min, depending upon the porosity of the sample. Considerable leaking occurred along the shaft of this pump, which was, therefore, provided with a mercury seal after mounting the pump vertically.

Air containing moisture corresponding to a partial pressure of water vapor of 18.1 mm Hg, and a relative humidity at 30° C of 57.5 percent, was produced by circulating air through tube *T* (fig. 2), which was a glass tube with an inside diameter of 25 mm, 4 meters long, in the form of a flat, horizontal coil, half filled with saturated sodium bromide solution. This provided a long path in contact with the solution, with negligible resistance for the circulating air; and rapid establishment of equilibrium was thus obtained. Tube *T* was kept in a thermostated bath, the temperature of which was maintained at 29.92°±0.07° C. The temperature of the sodium bromide solution was more constant, however, owing to the lag in temperature changes, and remained at 29.92°±0.02° C.

Dried air was obtained by evacuation of the chamber and refilling through the large glass tube, *D* (fig. 2), packed with fresh anhydrous magnesium perchlorate. After the chamber was refilled, the air was maintained in continued circulation through this tube. Data given in table 3 show the resulting moisture content, along with that obtained using saturated sodium bromide solution, and show also the time necessary for the attainment of equilibrium. The values were obtained by allowing samples of the gases from the irradiation chamber to pass through a P_2O_5 weighing tube into a large bottle of known volume, previously evacuated to a known pressure.

TABLE 3.—*Moisture content of air in irradiation chamber*

Conditioning system	Original moisture content of air [a]	Time of circulation	Water found [c]	Partial pressure of water vapor in chamber	Relative humidity at 30° C
	mm Hg	*min*	*mg*	*mm* Hg	*Percent*
Saturated sodium bromide solution at 29.92° ±0.02° C	19	15	27.6	18.2	57.8
	9	15	27.8	18.2	57.8
	9	30	27.3	17.9	56.8
	4	15	28.1	18.2	57.8
	4	30	27.9	18.0	57.2
	12	120	27.8	18.2	57.8
Average				18.1	57.5
Anhydrous magnesium perchlorate	19	30	1.2	.25	.79
	19	60	.2	.04	.13
	19	90	.0	.00	.00
	19	120	.1	.02	.06
	19	180	.0	.00	.00
	19	240	.0	.00	.00
Average				[b].005	.015

[a] The air temperature was 30° C, with a relative humidity of approximately 60 percent, giving an initial $P_{H_2O}=19$ mm. Lower initial humidities were obtained by evacuation of the chamber to various pressures and refilling with air drawn through magnesium perchlorate.

[b] Average based upon values after circulation periods of 90 min or more, the usual experimental condition.

[c] These values correspond to slightly varying quantities of gases taken for analysis.

An atmosphere of nitrogen was obtained by evacuating the chamber and refilling with purified N_2. Nitrogen from a commercial cylinder was passed lengthwise through a roll of copper screen, 37 cm long and 2.2 cm in diameter, contained in the Pyrex tube *O* (fig. 2), and kept at a temperature of 500° C by an electric furnace. (The copper was regenerated frequently by passing H_2 through tube *O*.) After refilling, continued circulation through tube *O* was maintained. A sample of gas,[2] taken after 90 min of circulation, had a partial pressure of O_2 not greater than 0.23 mm.

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VI. REFERENCES

[1] Herbert F. Launer, *An easily constructed, rugged, sensitive thermopile*, Rev. Sci. Instr. **11**, 98 (1940).
[2] W. W. Coblentz, *Radiometric investigation of water of crystallization, light filters, and standard absorption bands*, Bul. BS **7**, 619 (1911) S168.
[3] W. W. Coblentz, *Selective radiation from various substances*, Bul. BS **9**, 111 (1913) S191.
[4] W. W. Coblentz and R. Stair, *Date on ultraviolet solar radiation and the solarization of window materials*, BS J. Research **3**, 674 (1929) RP113.
[5] A. H. Taylor and Junius D. Edwards, *Ultraviolet and light reflecting properties of aluminum*, J. Opt. Soc. Am. **21**, 683 (1931).
[6] F. T. Carson, *A sensitive instrument for measuring the air permeability of paper and other sheet materials*, BS J. Research **12**, 567 (1934) RP681.
[7] Wm. F. Roeser and E. F. Mueller, *Measurement of surface temperatures*, BS J. Research **5**, 793 (1930) RP231.
[8] Int. Crit. Tables **2**, 312 (1927).
[9] L. A. Wood, Values of the physical constants of rubber. International Proceeding of the Rubber Technology Conference, London, May 1938.

WASHINGTON, March 19, 1940.

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[2] Analyzed by Martin Sheppard of this Bureau.