GOVERNMENT OF INDIA : THE PATENT OFFICE, 214, ACHARYA JAGDISH BOSE ROAD, CALCUTTA-17.

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"IMPROVEMENTS IN OR RELATING TO PREPARATION OF A PLASTIC FILTER TO TRANSMIT" NEAR INFRARED RADIATION."

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, RAFI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860)

This is an invention by Chittari Venkata Suryanarayana, Scientist, Holavanahally Narayana Rao Venkoba Rao, Scientist, Kurumathu Krishna Pillai Janardhanan Pillai, Senior Seientific Assistant, Venkata Subramanya Subramanian, Junior Scientificsis Astant and Kandan Ramakrishnan, Senior Laboratory Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3., India all Indian Nationals.

PROVISIONAL

The following Specification describes the nature of this invention.

This invention relates to improvements in or relating to the preparation of a plastic filter capable of transmitting radiation in the near infrared region.

Filters in the near infrared region are at present imported. Plastic filters have been generally known to have a limited usefulness due to multiplicity of overlapping absorption bands in the infrared. In case a clear window is available in a particular plastic exhibiting relatively good transmitting characteristics over a wide spectral region, they are known to do so only for thin samples. Further known filters like lucite (polymethyl methacrylate also sold under the name plexi glass) which serve as windows in the near infrared region have also considerable transmission in the visible region. Also lucite has the additional difficulty that it has a tendency to crack and coldflow. Even nonplastic filters useful in this range are to be imported from abroad.

To obviate these difficulties, the object of this invention is :

(i) to prepare a stable plastic filter capable of transmitting radiation in the near infrared region, which can be used as unsupported optical element in sufficient thickness to ensure good transmission, particularly without the absorption increasing markedly for thicker samples. We require a filter not only transmitting the near infrared region but having very little transmission in the visible and without a tendency to crack and coldflow.

To these ends, the invention broadly consists in the following:

To methyl methacrylate monomer iodine is added which dissolves readily. To this solution, macro-molecules which can initiate good polymerisation like perspex (polymethyl methacrylate). ethyl cellulose methyl cellulose etc are added. After getting the desired consistency of the solution, it is poured over a plain glass plate and hastened to set by exposure to heat radiation from an infrared source. In case no heat radiation is applied, the setting will take a few hours. The plastic filter thus obtained is uniform in thickness, does not crack and coldflow and it can be used unsupported as a thin sheet. Only mounting is used for purposes of holding. A solution of iodine in trichloroethylene with perspex, ethyl or methyl cellulose etc do give similar films with the desired transmission characteristics. But those films obtained with methyl methacrylate are superior.

The new plastic filter has optical transmission characteristics as shown in the graphs attached. Broadly it transmits from 80-90% from 0.9 to 2.2 microns and has a transmission of about 30-40% at microns with a cut-off around 2.8 microns. The filter material starts deforming at 80°C, softens around 110°C, starts blackening at 180°C and starts decomposition around 210°C. Up to 70-75°C the material is quite stable.

The filter material is resistant to moisture and after immersion in water for a period of 24 hours, the absorption of water ranges from 0.04% to 0.11%. The filter material is freely soluble in methyl methacrylate, trichloroethylene, nitric acid and acetone. It is insoluble in hydrochloric acid, concentrated potassium hydroxide and sodium hydroxide solutions. The filter material has a refractive index around 1.43 to sodium D-line.

The following typical examples are given to illustrate the invention:

Example 1:

A saturated solution of iodine in methyl methacrylate (3.2%) is prepared. In another container, a saturated solution of ethyl cellulose in methyl methacrylate is prepared. Equal volumes of these two solutions are mixed and well stirred and poured over a plain glass and allowed to spread. The ultimate thickness of the filter material can be controlled by adjusting the consistency of ethyl cellulose in methyl methacrylate. The mass spreads to a thin film over the glass plate over a time of a few hours. This can be hastened by irradiating with infrared radiation in which case the setting can be obtained in about an hour. A film thickness of 0.5 to 0.6 millimeters was obtained. By taking more dilute solution of ethyl cellulose in methyl methacrylate a filter film of 0.05 mm was obtained. The optical transmission characteristics of these two filters are given in Figure 1 of the accompanying drawings.

Example 2:

About 0.6% of iodine in methyl methacrylate is prepared. To this saturated solution of perspex in methyl methacrylate (about 10%) is added in equal volumes. After thorough mixing this is poured over a plain glass, and allowed to stay. One sample was heat-treated with infrared radiation and a thin sheet of the filter material was obtained within about one hour. This had a thickness of 0.2 mm. Another sample was allowed to remain over on plain glass plate for about 2-3 hours without the application of infrared radiation. The thin sheet of filter gave a thickness of 0.22 mm. The transmission characteristics of these filters are plotted in Figure 2 of the accompanying drawings.

The following are the main advantages of the invention:

(i) A plastic optical filter having a transmission of 80-90% from 0.9 micron to 2.2 microns and with lower transmission subsequently up to 2.8 microns with a cut off thereafter has been obtained for the first time in our country.

The region of transmission of the filter is suitable for use with the lead sulphide infrared detector whose transmission characteristics are almost in the same region, having a peak

at 2.1 microns and a cut-off at 3 microns. Whereas the normal defect with a plastic filter is reported to be its inability to be used as unsupported optical element in thickness sufficiently small to ensure good transmission, this filter has no such defect.

(ii) Production of this filter avoids import of known, costly, other types of inorganic material filters useful in the near infared region as windows.

(iii) The new filter material does not crack and cold-flow.

COMPLETE

The following Specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This invention relates to improvements in or relating to preparation of a plastic filter to transmit near infrared radiation.

Preparation of a filter transmitting near infrared region useful in opto-electronic devices has been done.

The near infrared region starts from about 0.7 microns. Devices which can detect radiation include the lead sulphide and lead selenide infrared detectors as well as other ones, as for example, indium antimonide. For example, the lead sulphide detector is responsive in the region of 0.7 microns to about 3 microns,. In this region, we do not have a single filter of response of the lead sulphide detector. A few wet and dry filters have been known in the near infrared region.

But their usefulness is limited due to multiplicity of over-lapping absorption bands. Filters incorporated with organic dyes like naphthol green B exist often in gelatin base which is difficult to handle at temperatures warmer than we meet with in the laboratory. Even if a window is available which transmits a wide spectral region, they are known to do so only for thin samples. Known filters like Lucite (polymethyl methacrylate also sold under the name 'Plexi Glass') have a considerable transmission in the visible region in addition to being a window in the near infrared region. Also Lucite has the additional difficulty that it has a tendency to crack and cold-flow. All filters in this near infrared region require to be imported from abroad.

The wet filters are rarely used nowadays due to handling difficulties. For example, organic dye filters prepared in the gelatin base involve complications in their technological preparation as well as handling. For a given dye the method of preparing the filter is involved. The gelatin filters are also prone to moisture and temperature effects and do not keep self-supporting physically. The colour glass-filters do not have a steep absorption edge. Besides, the glass filters are prone to mechanical shocks.

To obviate these difficulties, the object of this invention is:

to prepare a stable, self-supporting plastic filter capable of transmitting radiation in the near infrared region of response of the lead sulphide detector. In addition, another object of this filter is to have less of transmission in the visible region and without a tendency to crack and cold-flow.

The main finding of the invention is that iodine, methyl methacrylate monomer and ethyl cellulose in optimum proportions solidify to give a plastic filter transmitting the infrared radiation in the wavelength of 0.8 to 2.2 microns. The preparation does not involve any sophistication in handling.

The new result flowing from the above mentioned new finding is as follows:

To methyl methacrylate monomer iodine which dissolves readily in it is added. To this solution, macro-molecules like "perspex" (polymethyl methacrylate), ethyl cellulose, methyl cellulose are added. After getting the desired consistency of the solution, it is poured over a plain glass plate and to hasten setting, heat radiation from an infrared source is allowed to fall on it. In case no heat radiation is applied, the setting will take a long time, say, a few hours. The plastic filter thus obtained is uniform in thickness, does not crack and cold-flow and it can be used as a thin sheet without being sandwiched between transmitting support, only using a mount for purposes of keeping it in position. A solution of iodine in trichloroethylene with "perspex", ethyl or methyl cellulose do give similar films with desired transmission characteristics. But we have found that films obtained with methyl methacrylate are superior.

The new plastic filter has optical transmission characteristics as shown in the Figures attached. Broadly it transmits from 80-90% in the region of 0.8 to 2.2 microns and has a transmission of about 30-40% at 2.3 microns with a cut-off around 2.8 microns. The filter material starts deforming at 80°C, softens around 120°C, starts blackening at 180°C and starts decomposition around 210°C. Up to 70-75°C, the material is quite stable. The filter material is resistant to moisture and after immersion in water for a period of 24 hours, the absorption of water ranges from 0.04% to 0.11%. The filter material is freely soluble in methyl methacrylate, trichloroethylene, nitric acid and acetone. It is quite insoluble in hydrochloric acid, concentrated potassium hydroxide and sodium hydroxide solutions. It has a refractive index around 1.43(for sodium D line). The dielectric constant data of material are as follows:

Frequeney:		1.5 x 10^5 c/s	1 x 10^6 c/s	1 x 10^7 c/s
Filler at 30°C	$\varepsilon'\varepsilon_0$	3.1	3.05	2.9
	$\tan\delta$	374.7 x 10^{-4}	411.1 x 10^{-4}	538. x 10^{-4}

where ε_0 refers to dielectric constant at vacuum.

Statement of invention

The present invention consists of a process for the preparation of a plastic filter using ingredients which are capable of transmitting in the infrared region wherein by choosing a new combination of ingredients like iodine, methyl methacrylate and macromolecular substances like "perspex", ethyl cellulose, methyl cellulose and allowing the solution to polymerise either by allowing exposure after spreading on a smooth glass surface or additional heating by infrared radiation to quicken the process of setting, which results in the production of a smooth transparent self-supporting sheet of material of brownish yellow colour which has the characteristics of transmitting the infrared radiation in the region of 0.8 to 2.8 microns with a flat transmission from 1 to 2.2 microns, and being able to stand temperature up to 70-75°C.

The following typical examples are given to illustrate the invention:

EXAMPLE 1

A saturated solution of iodine in methyl methacrylate (3.2%) is prepared. In another container, a saturated solution of ethyl cellulose in methyl methacrylate is prepared. Equal volumes of these two solutions are mixed and well stirred and poured over a plain glass and allowed to spread. The ultimate thickness of the filter material can be controlled by adjusting the consistency of ethyl cellulose in methyl methacrylate. The mass spreads to a thin film over the glass.

plate over a time of four to five hours. This can be hastened by irradiating with infrared radiation in which case, the setting can be obtained in about an hour. A film thickness of 0.5 to 0.6 millimeters was obtained. By taking more dilute solution of ethyl cellulose in methyl methacrylate a filter film of 0.05 mm was obtained. The optical transmission characteristics of these two filters are given in Figure 1 of the accompanying drawings.

EXAMPLE 2

About 0.6% of iodine in methyl methacrylate is prepared. To this a saturated solution of "perspex" in methyl methacrylate (about 10%) is added in equal volumes. After thorough mixing, this is poured over a plain glass and allowed to stay. One sample was heat-treated with infrared radiation and a thin sheet of the filter material was obtained within about one hour. This had a thickness of 0.2 mm. Another sample was allowed to remain over a plain glass plate for about two to three hours without the application of infrared radiation. The thin sheet of filter gave a thickness of 0.22 mm. The transmission characteristics of these filters are plotted in Figure 2, of the accompanying drawings.

The main advantages of the invention are : (i) this avoids importation of equivalent filters; (ii) the filter is self-supporting and can be easily prepared and it can stand temperatures up to 70-75°C; (iii) the filter is also resistant to atmospheric moisture being absorbed; and (iv) the filter proposed herein does not crack and cold-flow and has a satisfactory shock resistance.

In the near infrared radiation transmission filters are usually of some organic dyes or inorganic materials embedded in gelatin or existing in solutions. A filter which is self-supporting, stable up to temperatures in the range of 30-70°C. and resistant to the action of moisture in the atmosphere is not easily available excepting for a few special ones (to be imported from abroad) and we have invented a process by which iodine, methyl methacrylate and macromolecular substances like "perspex", ethyl cellulose, methyl cellulose when mixed in optimum proportions and allowed to set on a smooth surface in the form of a sheet of a uniform thickness results in a filter with a good transmission to the eye having a browish yellow colour. This filter has been found to transmit the near infrared region ranging from 0.8 to 2.8 microns, which is appropriately useful in the region of response of the lead sulphide detector. This is easily made with all the freely available ingredients and can stand up to 70-75°C. Further the filter (3) A filter with these characteristics in a solid plastic base particularly having the additional characteristics of having less transmission in the visible region is not known so far.

We claim:

1. A process for making a filter which has the property of transmitting infrared radiation of 80-90% from 0.8 to 2.2 microns, 30-40% at 2.3 microns and with a cut-off around 2.8 microns which comprises mixing of iodine, methyl methacrylate and a macromolecular material like ethyl cellulose methyl cellulose, polymethyl methacrylate and the resultant mix is poured on a smooth surface, dried and set into a plate of desired thickness (dependent on the consistency).

Dated this 2nd day of July 1971.

Sd.
of Council of Scientific and Industrial Research.
Patents Officer

Adarsh Bharat Printers, Meerut.

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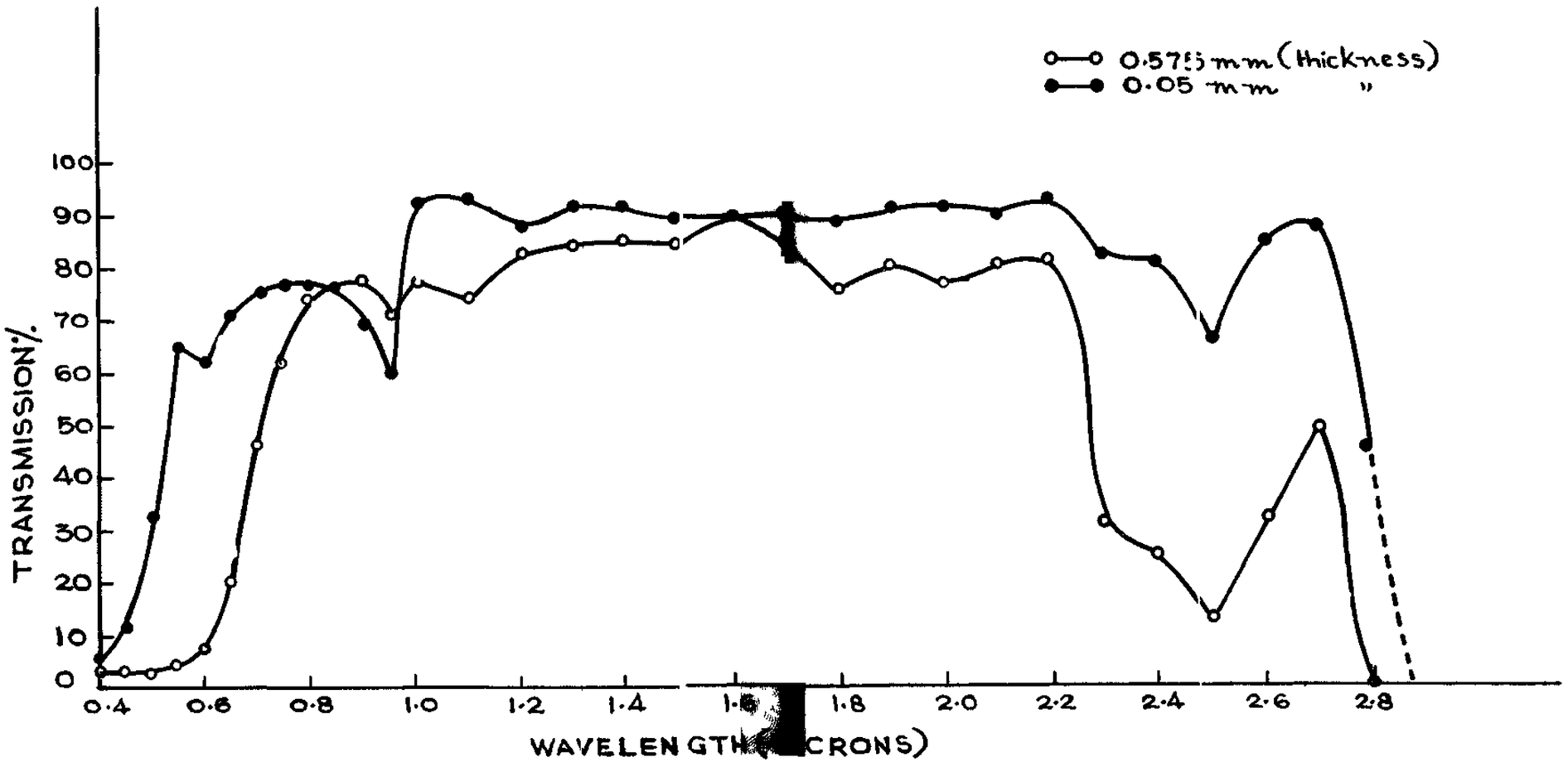
PROVISIONAL SPECIFICATION

SHEET No. . . :- 1

FIGURE - 1.



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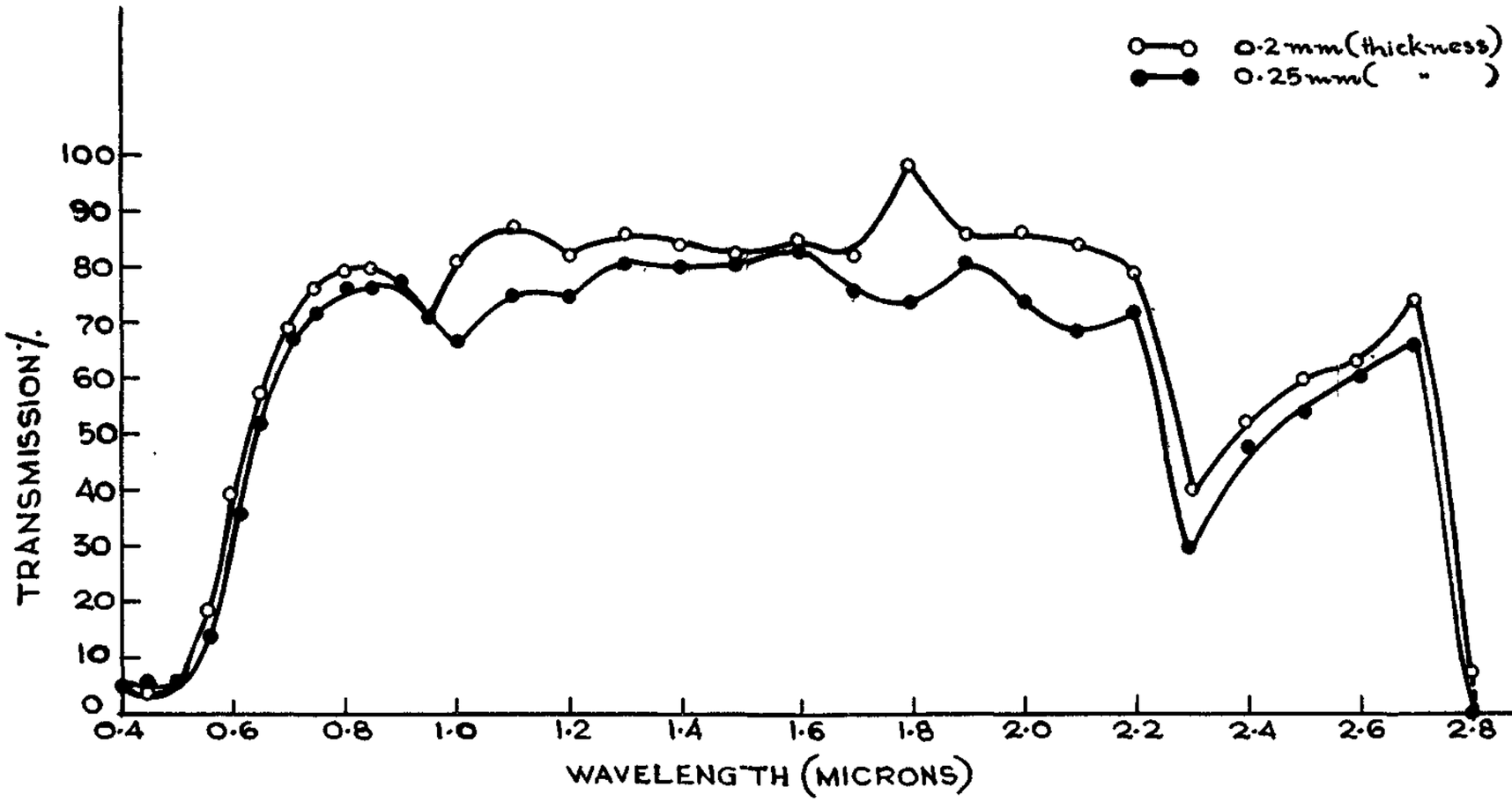
PROVISIONAL SPECIFICATION

FIGURE-2.



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COMPLETE SPECIFICATION

No. OF SHEETS:- 2

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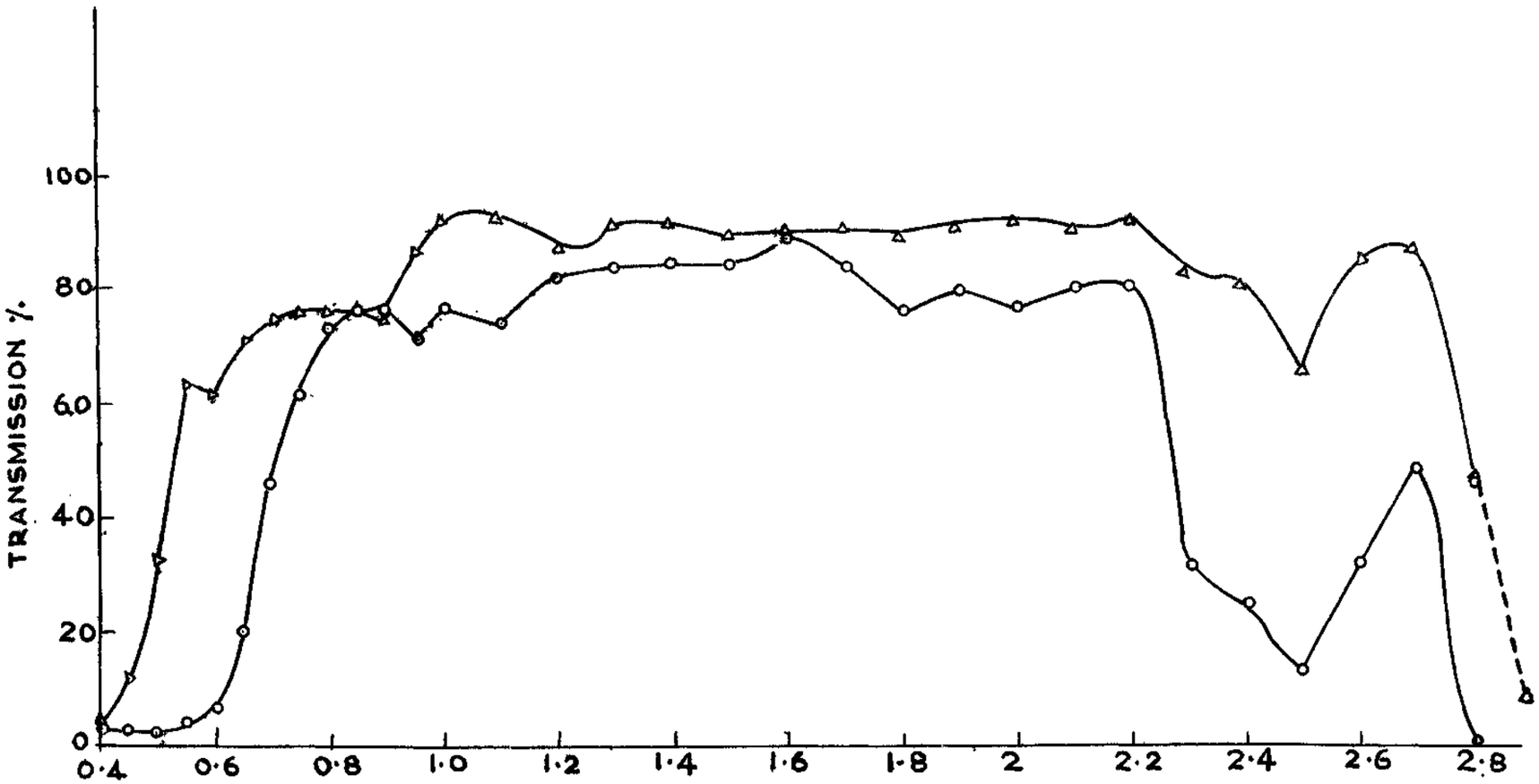
FIGURE - 1



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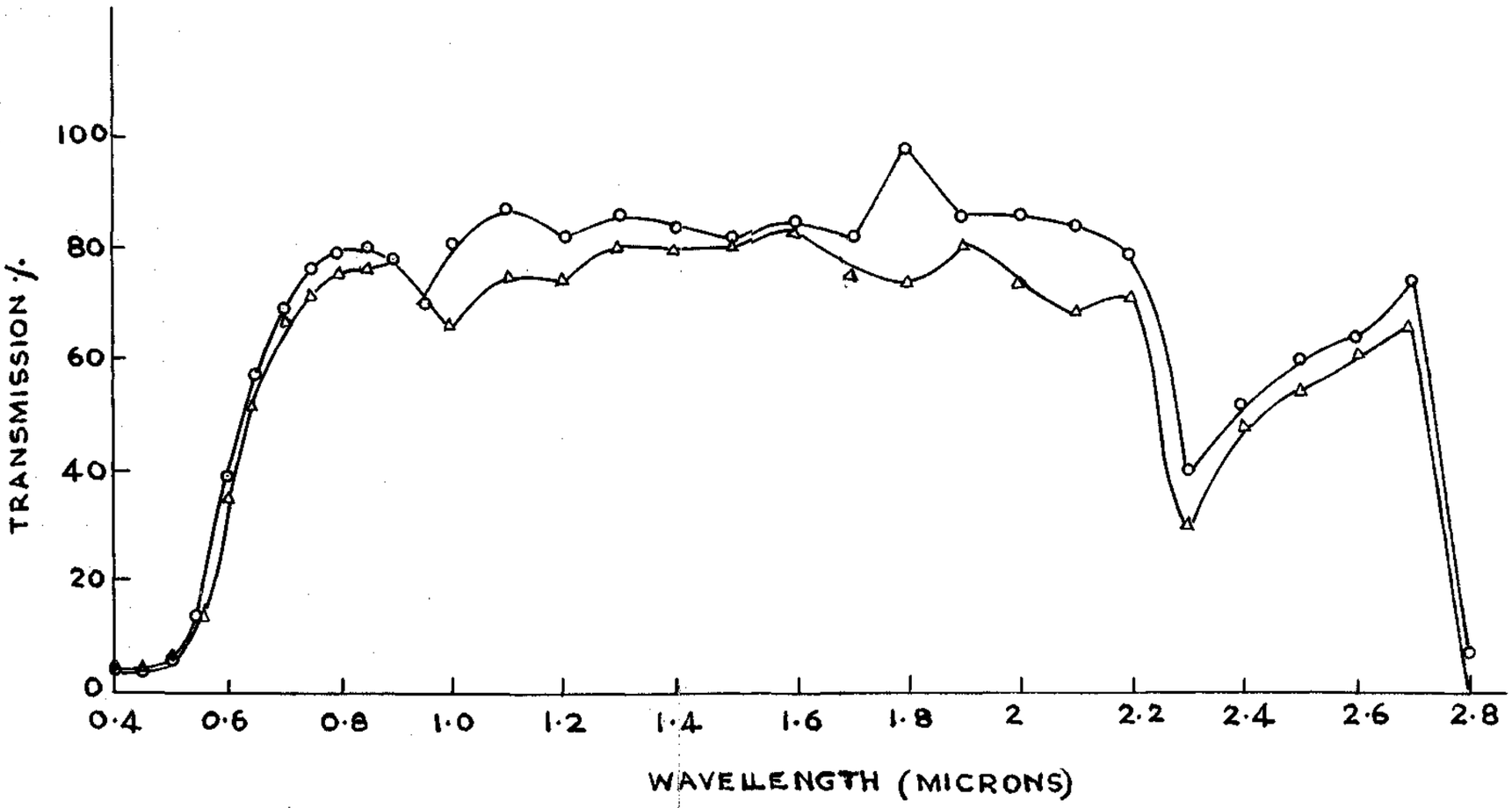
COMPLETE SPECIFICATION

SHEET NO :- 2

FIGURE-2



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