LBL-7825
UC-95d
TID-4500-R66

c.2

RECEIVED
LAWRENCE
BERKELEY LABORATORY

AUG 11 1978

LIBRARY AND
DOCUMENTS SECTION

TWO-WEEK LOAN COPY

This is a Library Circulating Copy
which may be borrowed for two weeks.
For a personal retention copy, call
Tech. Info. Division, Ext. 6782

Energy and Environment Division

High Performance Solar Control Office Windows

William J. King

December 1977

Lawrence Berkeley Laboratory University of California/Berkeley

Prepared for the U.S. Department of Energy under Contract No. W-7405-ENG-48

LBL-7825
c.2

LEGAL NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

HIGH PERFORMANCE SOLAR CONTROL OFFICE WINDOWS

Wm. J. King

KINETIC COATINGS, INC.
Burlington, Massachusetts 01803

Prepared Under Contract No. 1667000

for

LAWRENCE BERKELEY LABORATORY

UNIVERSITY OF CALIFORNIA
Berkeley, California 94701

This work was performed for the Lawrence Berkeley Laboratory, University of California, under DOE Contract No. W-7405-Eng. 48 for the U. S. Department of Energy.

FINAL REPORT December, 1977

FOREWARD

This report was prepared by Kinetic Coatings, Inc., Burlington, Massachusetts 01803, and describes work performed from April through December, 1977 on Lawrence Berkeley Laboratory Contract No. 1667000.

S. Selkowitz was Project Monitor for Lawrence Berkeley Laboratory.

Wm. J. King was Program Manager and was assisted in the research by E. R. Berman.

Consultation on window characteristics and aid in obtaining access to analytical instruments at MIT and Lincoln Labs was provided by T. Johnson of the MIT Department of Architecture.

ABSTRACT

The following report describes investigations conducted over a 9 month period on the use of ion beam sputtering methods for the fabrication of solar control windows for energy conservation. Principal emphasis was placed on colored, reflecting, heat rejecting, office building windows for reducing air conditioning loads and to aid in the design of energy conserving buildings. The coating techniques were developed primarily for use with conventional absorbing plate glass such as PPG solarbronze, but were also demonstrated on plastic substrates for retrofit applications. Extensive material investigations were conducted to determine the optimum obtainable characteristics, with associated weathering studies as appropriate aimed at achieving a 20 year minimum life.

Conservative estimates indicate that successful commercialization of the windows developed under this program would result in energy savings of 16,000,000 barrels of oil/year by 1990 if installation were only 10% of new commercial building stock. These estimates are relative to existing design for energy conserving windows. Installation in a greater percentage of new stock and for retrofit applications could lead to proportionately greater energy savings. All such installations are projected as cost effective as well as energy effective.

A secondary program was carried out to modify the techniques to yield thermal control windows for residential applications. These windows were designed to provide a high heat retention capability without seriously affecting their transmission of incident solar radiation, thereby enhancing the greenhouse effect. This part of the program was successful in producing a window form which could be interchanged for standard residential window material in a cost and energy effective manner. The only variation from standard stock in appearance is a very light rose or neutral gray coloring.

TABLE OF CONTENTS

Section	Page
1 Project Background	1
1.1 General	1
1.2 Window Design Considerations	3
1.3 Deposition Technology	13
2 Material Studies	14
2.1 General	14
2.2 Reflectivity	16
2.3 Growth Mechanism Study	69
2.4 Weathering	77
3 Sample Windows	95
3.1 Office Windows	95
3.2 Residential Windows	104

TABLES

1. Layer Characteristics - Reflectivity Study	19
2. Sample Characteristics - Growth Mechanism Study	71

LIST OF ILLUSTRATIONS

Figure	Description	Page
1	Radiation Regions for Window Design	7
2	Office Window Design Charcteristics	12
3	R&T vs λ : SiO_2 - No reflecting layer	24
4	R vs λ ; Al-Al_2O_3	25
5	T vs λ ; Al-Al_2O_3	26
6	R&T vs λ ; Al-Al_2O_3; G substrate	27
7	R&T vs λ ; Al-Al_2O_3; G substrate	28
8	R&T vs λ ; Al-Al_2O_3 ; PC substrate	29
9	" "	30
10	R&T vs λ ; Al-Al_2O_3 ; M substrate	31
11	" "	32
12	R&T vs λ ; Al-Al_2O_3 ; P substrate	33
13	" "	34
14	R&T vs λ ; Al-SiO_2 ; G substrate	35
15	" "	36
16	R&T vs λ ; Al-SiO_2 ; PC substrate	37
17	" "	38
18	R&T vs λ ; Al-SiO_2 ; M substrate	39
19	" "	40
20	R&T vs λ ; Al-SiO_2 ; P substrate	41
21	" "	42
22	R&T vs λ ; Brass-SiO_2 ; G substrate	43
23	" "	44
24	R&T vs λ ; Brass-SiO_2 ; PC substrate	45
25	" "	46
26	R&T vs λ ; Brass-SiO_2 ; M substrate	47
27	" "	48

LIST OF ILLUSTRATIONS (continued)

Figure	Description	Page
28	R&T vs λ ; Brass-SiO_2 ; P substrate	49
29	" "	50
30	R&T vs λ ; Ni-SiO_2	51
31	"	52
32	R&T vs λ ; Cu-SiO_2	53
33	"	54
34	R&T vs λ ; Ti-SiO_2	55
35	" "	56
36	R&T vs λ ; Inconel 671-SiO_2	57
37	"	58
38	R&T vs λ ; Mo-SiO_2	59
39	"	60
40	R&T vs λ ; Ag-SiO_2	61
41	"	62
42	R&T vs λ ; Au-SiO_2	63
43	"	64
44	R&T vs λ ; Growth Mechanism Study	73
45	Sample W-54-1 ; SEM	75, 76
46	R&T vs λ ; Brass-SiO_2 ; W-54	78
47	" "	79
48	Weatherometer	81
49	Weatherometer Cycles	83
50	R&T vs λ ; Brass-SiO_2 ; 1-4	89
51	R&T vs λ ; Brass-SiO_2 ; 1-1 $\rightarrow$ 1-5	90
52	R&T vs λ ; Brass-SiO_2 ; 1-4	91
53	R vs λ ; Cu-SiO_2	93
54	"	94

LIST OF ILLUSTRATIONS (continued)

Figure	Description	Page
55	R vs λ ; Office Window Sample Set ; U of C	96
56	T vs λ ; Office Window Sample Set ; U of C	97
57	R vs λ ; Office Window Sample Set ; U of C	98
58	T vs λ ; Office Window Sample Set ; U of C	99
59	Performance Curves ; Office Window Sample Set ; U of C	101
60	R vs λ ; Standardization Curves	106
61	R vs λ ; Residential Window Samples ; U of C	108
62	" "	109
63	" "	110

Project Background

1.1 General

The overall problem of providing an energy conscious world with an adequate supply of energy, reduces, in its simplest terms, to a balance in which energy generation must equal or exceed energy consumption. Obviously the problem of everexpanding demand can be met with a multiplicity of approaches including increased energy production, reduced demand, or a combination of the two. Advanced countries, especially technically advanced ones, tend towards the first solution with little consideration for the latter two. In fact, the United States which has the most critical energy problem in the sense that it is the largest per capita consumer, nevertheless continues to be the worst offender in terms of energy wastage. Per capita energy consumption continues to increase drastically in spite of substantial price increases.

The continued lack of emphasis on conservation by the consumer is a difficult problem to overcome. The major difficulty seems to be that those conservation measures which have been implemented such as lower highway speeds, less travelling, lower building temperatures in winter and higher in summer etc., require active participation by the consumer, who for the most part is apathetic or insensitive to the problem. Short of mandatory controls, the only alternatives are financial incentives or passive measures which either do not require individual consumer participation or can be permanently built into his life style. Among the latter, and potentially of great importance is the use of specialized architectural windows for solar heat transmission and retention for reducing heating costs in residential applications, and for solar heat rejection for reducing air conditioning costs in office building applications. The former may require modest consumer participation through the use of shades or screens during non-heating or cooling periods. In contrast, heat rejecting office windows require no consumer action after installation and if properly designed in conjunction with other building design factors can provide energy savings throughout their useful life. Very little has been done commercially for residential applications (however, normal greenhouse effect operates

for ordinary window glass) but some development has been done for office building windows through so-called solar control windows. Due to technical limitations, however, the latter are marginal in their effectiveness. The need for better designs and manufacturing methods has been recognized by the industry, but this need is not satisfiable by standard technology.

Technology capable of producing "high performance" solar control windows was developed at Kinetic Coatings, Inc. (KCI) and the application of the technology to further development of such windows forms the basis of this program. This technology also has potential applications in residential heat retention windows, and due to its compatibility with plastic substrates, has potential applications to plastic laminates for retrofits for both types of windows. All of these applications have been investigated to different degrees under this program, with solar control office windows being predominant.

Actual energy savings resulting from the office windows will depend on the extent of their utilization in new construction, the latter being determined by the building industry based on availability, cost, and architectural factors, as well as on general aesthetic values since they must be acceptable to the public at large. In developments to date, considerable effort has been devoted to the problem of consumer acceptance and the office building glasses, in addition to being designed for "exterior" rejection of the infrared heat load, have also been designed to provide different levels of interior lighting as well as different internal and external coloring. The latter, which permits design coordination with other architectural factors, should help significantly in gaining acceptance by architects and the building industry. For each "typical" office building having a base 200 feet on each side and being nominally 40 stories high, the projected energy savings are 3,000 - 6,000 barrels of oil per year at an operational (electric power) cost savings of $ 54,000 - 108,000/year. On the average, for each 1,000 typical office buildings or equivalent installed, the total energy savings per year would be 4,500,000 barrels of oil. Total savings per year, e.g. by 1990, requires a detailed study of the building industry including design trends. A rough estimate using data calculated for the American Institute of Architects indicates an energy saving of 43,600 barrels/day if only

10% of new commercial buildings use the proposed windows.

1.2 Window Design Considerations

The problem of conserving energy by appropriate window design, as opposed to generating energy through solar converters of various types, is diametrically opposite in office buildings (assumed high rise) to that for residential applications. In the latter, current thinking[1] is directed at retaining solar heat reaching the building interior during heating periods, with shading and/or other techniques used to prevent solar heat from reaching the interior during cooling periods. In contrast, the major problem with new tall energy conserving office buildings will be the avoidance of unnecessary heat loads.

This difference arises because the interior heat sources are radically different in the two cases. Residences usually have modest "indirect" heat sources in the form of lighting fixtures, people, equipment etc. and have high thermal losses through ceilings and walls relative to the amount of floor space. Depending on climate, auxilliary heat sources ranging from small to extensive are required to maintain comfort conditions during periods when the outside temperature is low. Cooling may, or may not, be used when the outside temperature is high, depending on climate and personal circumstances.

The situation for highrise office buildings, particularly for current and projected designs, is radically different. Many of these buildings, particularly in the larger cities, are designed for aesthetic and public relations value as well as for functionality, and as such are highly fenestrated, with external window areas of 80% or greater. As such, they present a particular problem in energy conservation since the design factors for lighting, ventilation, air conditioning, heating, etc. are interrelated in a complex manner which becomes considerably

---

(1) "Energy Conservation and Window Systems", A Report of the Summer Study on Technical Aspects of Efficient Energy Utilization; July 1974-April 1975, Samuel M. Berman et al - Available from NTIS.

more complicated when conservation of energy is emphasized. Designs for buildings of this type are not likely to alter drastically in the near to medium term future, and any conservation measures developed will have to be consistent with present design practices if they are going to be accepted by the industry.

The assumption is therefore made here that office buildings of the type used by insurance companies, banks etc., and even government (GSA) buildings in highly visible locations, will continue to be highly fenestrated and suffer from major solar loads on the air conditioning systems in summer. Techniques such as windows tilted to avoid solar radiation in summer but to capture it in winter are much more difficult to design into such high rise buildings than into low-rise offices or residences. Much smaller window areas and thick highly insulated walls tend in general to give a less pleasing appearance and present additional demands on artificial lighting requirements which tend to compensate for the energy saved by the reduced window area. In addition, for many sites, and certainly in the middle-southern regions, because of other conditions (discussed below) the problem in designing such buildings (if energy conservation measures are used) is one of avoiding solar loads during most of the year.

A major, if not the major, factor in such buildings is the fact that they have very large indirect heat sources (particularly lighting, equipment, and personnel) during the occupied portion of the day. This large heat source coincides with the period in which the solar heat influx is greatest. In the core of the building where there are essentially no heat losses (except for ventilation), the problem during all seasons is essentially one of keeping the temperature sufficiently low. This is the case even for a shaded building in winter in a relatively northern climate. Local heating problems do occur at the building periphery where thermal losses through walls or openings are high, or where there is cold air intrusion. Older design practices accounted for this by providing local space heating, thus duplicating the energy expenditure for cooling in the core with one for heating at the periphery. More modern design practices attempt to avoid this duplication as much as possible by redistributing

the heat from the core to the peripheral areas using air mixers, heat exchangers etc. There can, in fact, be a surplus of indirect heat even under relatively cool external conditions, the basic problem being one of distribution, rather than supply.

Future building designs, even while conserving energy overall may actually increase this surplus. For example, if the building is designed with a "total energy system" in which electric power is generated from fossil fuels on site, the 65% of the fossil fuel which is normally wasted as heat at power generating stations can be used for heating water, preheating cool make-up air in winter, local heating etc. Since the indirect sources such as lighting and people will still contribute heat, such buildings could have a relatively high internal heat surplus during all seasons, requiring extensive cooling. During seasons where the peripheral areas must be heated, surplus heat from the building core during the "loaded" hours would be stored (e.g. in sub-terrestrial water tanks) to provide thermal storage reserves for lower occupancy periods (e.g. early evening) or for preheating building in mornings if necessary etc. It should be noted that the heat removed in mechanical cooling could also be partially recovered (e.g. used for local heating in winter, heating water etc.) if necessary. Obviously cool air can be brought in from the outside in winter, but this presents the problem in many cities of purifying the air which also requires energy. Refrigeration or mixing with air from cooler building regions will be preferable in many cases.

Even in many buildings in which there is not always a net surplus of indirect heat, if a high degree of fenestration is desired, a high solar radiation level may be undesirable because of local "overheating" problems or glare conditions. In such cases the preferred design route might be to reduce the major solar load on the air conditioning in summer, with user comfort dictating the desired conditions in winter.

In general, therefore, for the type of building considered here, with the exclusion of visible light required for natural lighting, solar energy reaching the interior of the building represents an undesirable load on the energy requirements. This forms the basis for window design.

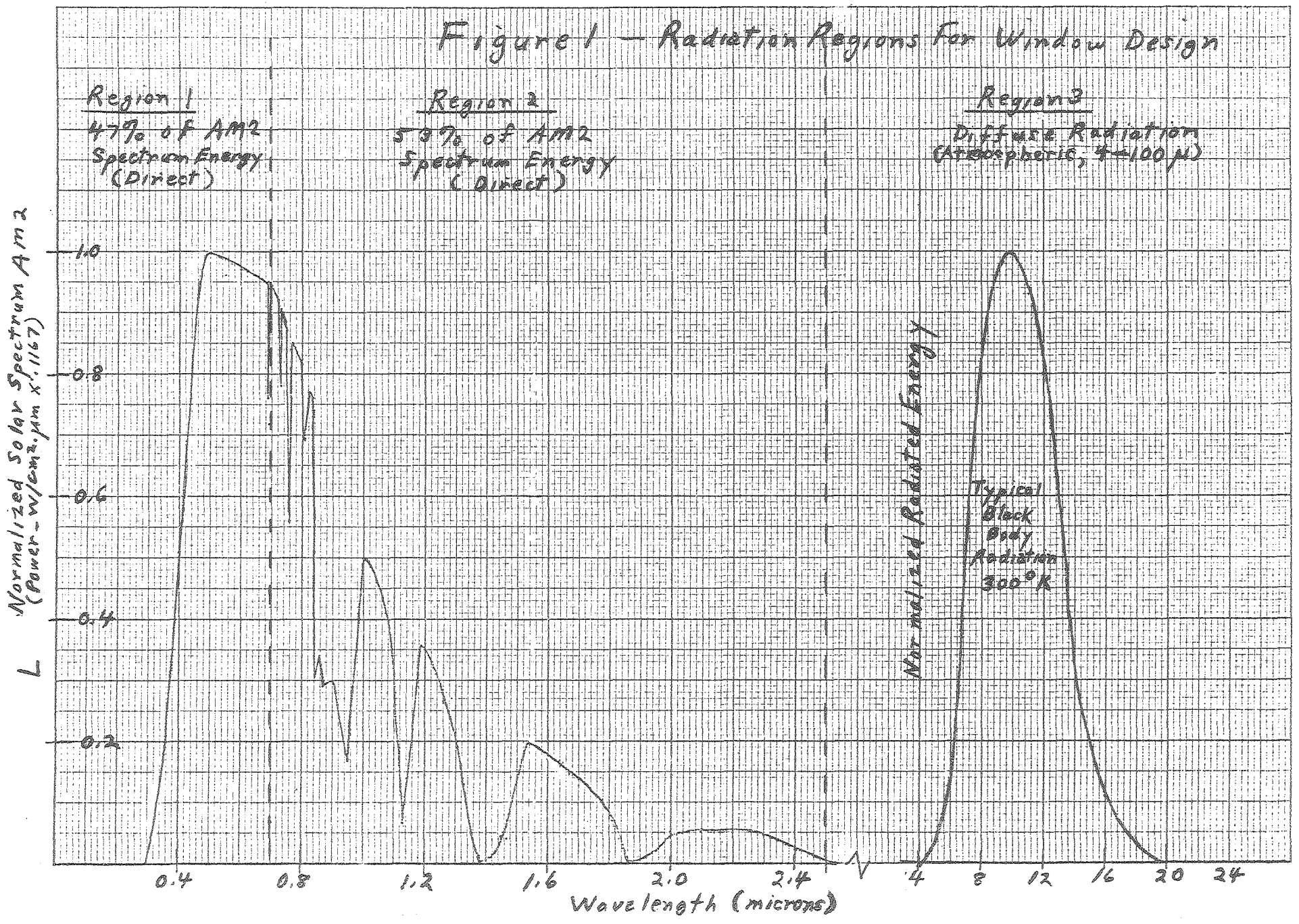
The effect of this difference on window design may be appreciated by referring to Figure 1 which shows the Air Mass 2 (AM 2) solar spectrum and a typical radiation spectrum for an internal heat source operating near 300 °K (approximately room temp.). Slight shifts in the latter to lower wavelengths occur for hotter sources (e.g. motors etc.) but this has little or no effect on overall design criteria unless critical thresholds are reached (see below). The AM 2 spectrum is close to that which actually reaches the earth under practical conditions: Approximately half (45%) of the energy is in the region from .35 - .7 μ, while the rest (55%) is in the near IR from .7 - 2.5μ. This is direct solar radiation. In addition there is incident long wavelength radiation from the sky and clouds, which is in general at wavelengths from 4 μ up, reflected radiation from the ground and thermal radiation from other sources such as other buildings. These sources, as well as climatic and angle effects have been combined by Berman and Claridge[2] to obtain average and cumulative seasonal solar radiation levels on typical vertical building faces (four cardinal directions) at different latitudes as an aid in hard design.

In designing residential windows to collect and retain solar heat, one would like to transmit all incident energy in regions 1 and 2 but retain all energy from internal sources in region 3. Ordinary clear window glass does this to a certain extent through the so-called greenhouse effect. The usual explanation for the latter is that the incident short wavelength radiation ($\lambda < 2.5\ \mu$) after heating up the building interior is reradiated as longer wavelength radiation ($\sim 10\ \mu$) which is absorbed in the glass and cannot escape. It seems fairly obvious that energy radiated by the interior and captured or absorbed in the glass must be "partly" transmitted to the outside by conduction through the glass and convection at the outside surface. The presence of a selective reflector (transmit $< 2.5\ \mu$, reflect $> 2.5\ \mu$) on the

---

(2) Ibid -Section 11B

K+E 20 X 20 TO THE INCH • 7 X 10 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.



inside surface of the glass should reduce such losses by reflecting the interior radiation back into the building. Hewett[3] has presented computer data attributed to Linde Laboratories for total heat losses for single and double glazing, with and without internal reflecting layers. Unfortunately, these calculations are for solar collectors which therefore have fixed internal conditions rather than the comparatively fluid situation existing in a normal residence. However, the data does indicate that the losses for a 50°F differential are approximately 40 BTU/hr. and 20 BTU/hr. per square foot for single glazed applications, without and with an 85% internal IR reflecting layer, respectively. Similar results for a double glazed application are approximately 27 and 15 BTU/hr. ft^2. The results also indicate substantial increases in heat losses for non-absorbing (in thermal IR) glasses, vs ordinary glass, of 33% and 76% for the single and double glazed cases, respectively.

Obviously more work is required in this area in order to determine the best approach to conserving energy, particularly if the economics of the prosessing involved are considered in detail. If one does go the reflecting glass route for residential applications, the layer must be on the inside since otherwise the thermal radiation would simply be absorbed in bulk glass as in the conventional case.

Although the above arguments have significance for office building windows, the situation for the latter is basically quite different. With the assumption of an energy conserving building in a warm to moderate climate one in general is cooling and the need is to reduce heat load as much as possible. Since 55% of the direct radiation is in the .7 - 2.5 μ wavelength region, one would like to prevent this energy from reaching the interior. Obviously this would be best accomplished by having a suitable reflecting layer on the outside of the window to reflect as much of this radiation as possible as well as the longer wavelength sky radiation.

(3) Letters to Optical Spectra; Thomas A. Hewett, Union Carbide Corp.; June, 1976.

For an interior reflecting layer, all of the sky radiation would be absorbed in the glass as well as some of the direct radiation, the percentage for the latter being determined by the characteristics of the bulk glass used. Some of this energy would then be transferred to the cooler interior by conduction and convection at the inside surface. (Colored glasses which absorb heavily in the visible and near IR are, for this reason, relatively ineffective compared to a good reflecting layer on the outside.) Also, the longer wavelength radiation from interior sources would be reflected directly back into the room rather than being absorbed in the glass where it could be partly transferred to the outside. The latter is particularly important for those areas of the building not under direct solar radiation where the outside surface of the window is cooler. Detailed calculations have shown that an outside reflecting layer over a basic absorbing (i. e. colored-e. g. neutral gray or brown) substrate is the optimum configuration.

It should be noted here that for buildings in marginal regions where substantial cooling seasons exist, but where excess core heat is not adequate to heat peripheral areas, these outside reflecting layer glasses will be as effective in retaining interior heat as conventional glasses. However, much less direct solar radiation will be transmitted to the interior.

Another important aesthetic and practical consideration is the appearance of the glass when viewed from the inside and the overall interior lighting level. It is desirable to set the maximum visible transmission at a level which will provide comfortable lighting levels under high direct radiation. The amount and character of transmitted light are dependent on the reflectivity of the exterior reflecting layer as well as the absorptance of the bulk glass. The desirable reflectivity will therefore be much lower in the visible then in the .7 - 2.5 μ range, and what is needed is the capability of adjusting the visible reflectivity while maintaining a high near IR reflectivity. Put another way, a threshold is needed at approximately .7 μ dividing the reflectivities in regions 1 and 2 of Figure 1. When viewed from the inside the glass will also be reflecting in the visible. If the reflecting layer is on the inside the effect could be quite objectionable (e.g. with lights on) if the visible reflectivity is

significant, e.g. 30 - 40%, since one then sees only the inside image, not the outside world. The mirror effect is effectively reduced if the reflecting layer is on the outside and a glass with substantial absorptance (e.g. 40 - 50% per pass) in the visible is used for the bulk material. The internal reflected image (note: 2 passes through glass) for this system can be reduced to non-objectionable levels. The critical factor is that, as the window is viewed from the inside, the amount of light coming from the outside should be considerably higher than the amount of internal reflected light. The reflectivity in the visible when viewed from the outside can still be high enough to provide pleasing architectural effects.

It is also important to note that any visible direct radiation which is reflected will also reduce the solar load on the air conditioning system. (Note: same argument as above applies for having reflecting layer on outside if glass is absorbing in visible.) The relative levels of reflectivity and absorptance must be adjusted to optimize this saving while preserving the aesthetic and lighting conditions as discussed above. A desirable manufacturing process would be one in which these factors could be easily adjustable in manufacturing the glass for a given building and location. Since most bulk glasses are made in only a few basic colors and absorptance levels using well established manufacturing process, it is desirable that such control be exercisable through control of the reflecting layer.

From an acceptance viewpoint, one must also consider the color of the glass as viewed from the inside and outside. The color as viewed from the inside should not be vivid but rather should be a neutral tone such as the neutral grays or browns currently being used. The availability of different external colors rather than only plain metallic would permit coordination with other architectural factors and thereby stimulate the use of energy conserving windows because of building design considerations. It is certainly also desirable that the incremental cost of the reflecting layer be less than or equal to the operational cost savings over the depreciation period.

The decision to go with an external reflecting layer requires that the layer per se be immune to environmental attack or be protected by a suitable

layer. This is also true of inside reflecting layers such as may be used in residential applications, although the detailed requirements might be different in the two cases. In general, the major requirements for protective layers are hardness, hermeticity and resistance to chemicals such as sulfur, carbon monoxide etc. Of these, hermeticity is the most difficult to achieve.

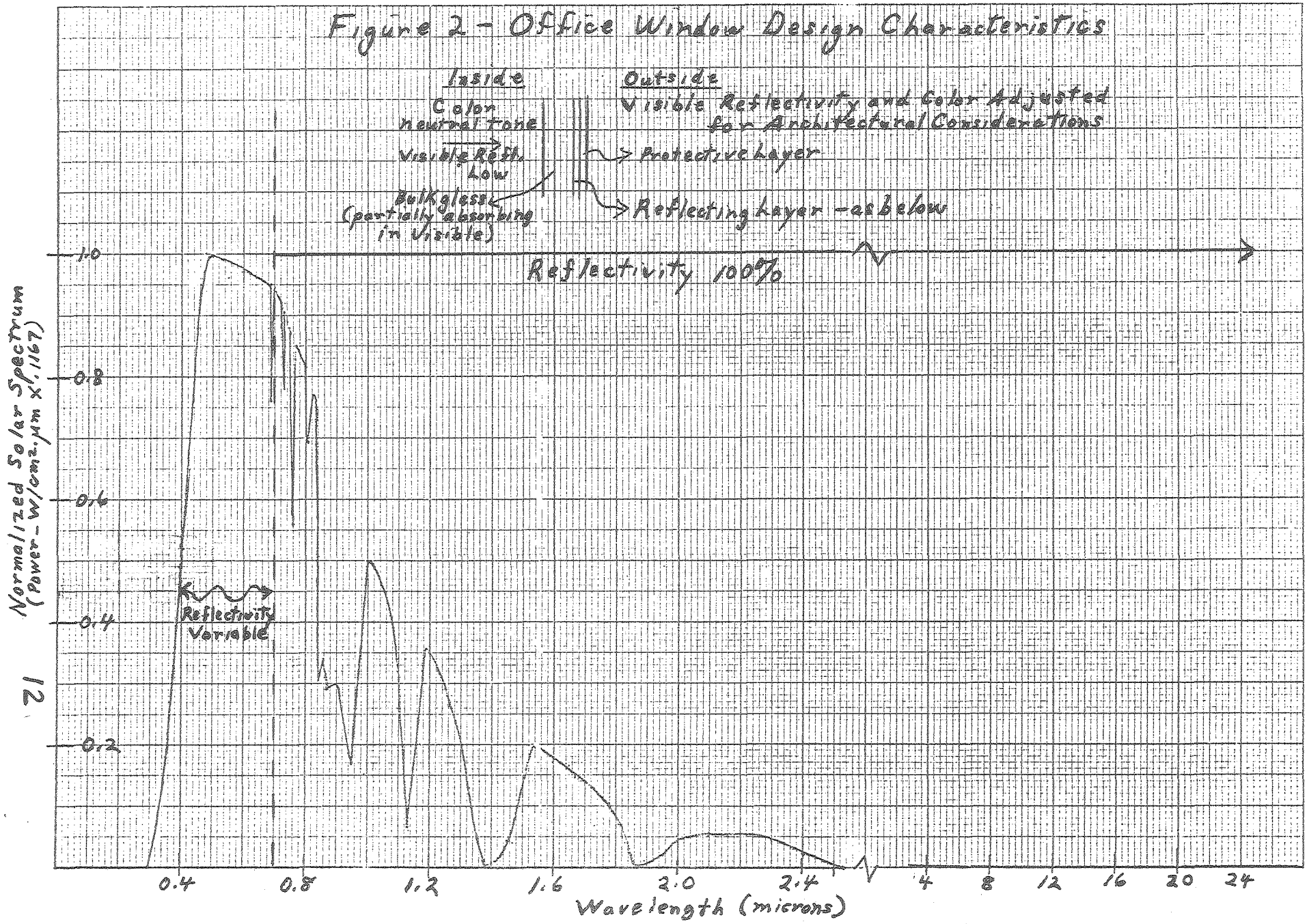
The choice of an outside reflecting layer for energy conservation is based on single glazed applications. Similar energy savings can be achieved with double glazing with the two glass layers being separated by air or gas gaps and with the reflecting layer on the interior of the outside glass. Incident radiation from the outside is effectively prevented from reaching the inside. However, double glazing is more expensive and difficult to install and has in some cases caused some structural stability problems. Single glazing is therefore preferred. In summary, the desirable characterisitcs of an energy saving office building window are (see Figure 2):

1) Single glaze for economy
2) Reflecting layer on exterior surface
3) Reflectivity as close to 100% as possible from .7 - 24 μ
4) Reflectivity variable in visible for light control and architectural factors
5) Absorption in glass for reduced interior reflection
6) Color from inside a neutral tone
7) Color from outside alterable to conform with building design
8) Reflective layer sufficiently hard or well protected to withstand environment for many years
9) Operational cost savings over depreciation period equal to or greater than incremental cost of reflecting layer added to basic glass

In contrast, the desirable characteristics for a residential energy conserving (i.e. heat retaining window) are:

1) Single glaze for economy
2) Reflecting layer on inside surface

K+E 20 X 20 TO THE INCH • 7 X 10 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.



3) Reflectivity as close to 100% as possible from 2.5μ-20μ
4) Reflectivity as low as possible in visible and near IR (i.e. from .4 -2.5μ)
5) Absorption in glass as low as possible from .4-2.5μ
6) Color from inside minimal; i.e. as close to clear aspossible
7) Color from outside as close to clear as possible
8) Reflective layer resistant to inside atmosphere and cleaning
9) Must be inexpensive to be compatible with lower price market

Criteria similar to the above were the basis for developments under this program. Emphasis in the program has been at least 90% on office building windows, with the results on residential applications being secondary in most cases.

1.3 Deposition Technology

All program developments to date have been based on modifications to a basic process called ion beam sputtering (IBS) which was invented by one of the principals of KCI in 1963. This process has been under private development ever since and is now a well characterized process used internally for many specialized applications, particularly in semiconductor and optical areas.

Basically, IBS is a process in which a beam of noble gas ions (Ar, Xe, Kr) is extracted from a plasma source in vacuum and accelerated to energies in the keV range. This beam of energetic ions is allowed to impinge at an oblique angle on a target composed of the material to be sputtered and deposited on the substrate. The latter is normally mounted opposited and parallel to the target.

Vacuum level in the chamber is primarily a function of the pumping system and gas load from the sputtering beam, and can be in the 10^{-6} Torr range at ion beam levels consistent with reasonable deposition rates. Most importantly, the process can be operated without a high background of the sputtering gas, as opposed to other sputtering techniques wherein the plasma

can easily become contaminated with matter such as target support materials etc. which affect the impurity levels in the deposited film. Only the target is sputtered. Equally important is the fact that the partial pressure of background gases, such as O_2, can be much lower than operating levels.

In the IBS system, the beam is completely intercepted by the target, and because the sputtering rates (atoms sputtered/incident ion) are usually much greater than 1, the noble gas ions after interception by the target rapidly become exposed and are pumped away. No evidence has ever been observed in any film deposited by the technique of contamination of the sputtered film by inclusion of the sputtering gas. In general, the deposited material can be made as contaminant free as the target material.

The target per se can be composed of almost any material normally used for coating applications including all metals,and compounds such as SiO_2, Al_2O_3, SiO etc. The only change required to go from one material to another is to change the target. Multi-target systems have been built in which any of four target materials could be selected without breaking vacuum and in essence systems with more available target materials could easily be designed if required.

2 Material Studies

2.1 General

Put briefly, this program has the goal of using IBS methods to create windows having controlled but variable reflectivity as a function of wavelength for energy conservation measures. Also important to the project is the combination of the optical properties of absorption, reflection and interference to provide control of the external window colors, and color intensities, over a broad range. At the same time it is necessary to control the color, appearance, and light intensity as viewed from the inside. The proprietary KCI single glaze method used in the project provides extremely good control over these and other factors outlined in Section 1.2. This method can be extended to commercial scale manufacturing with little modification except scaling. As demonstrated in the following results, the materials used for the coatings are abundant, cheap and non-unique; i.e. similar performance can be obtained with different starting materials if the techniques are properly applied. However, the ability to use different starting

materials does provide a broader range of available characteristics.

Projected costs for this coating method on a large scale basis are consistent with energy conservation criteria and/or with add-on costs based on the building design freedom provided by the availability of reflecting metallic colors rather than the simple gray metallic. Considerably more effort is required on calculations of anticipated energy savings since this is a complex problem depending on present and future building design patterns.

The KCI method of obtaining the desired reflectivity depends more on technique than on material factors, and various materials have therefore been used successfully. Selective reflection in the visible has been used for external color adjustment and control of the amount and character of light transmitted to the interior. For some modifications selective absorption and interference effects have also been combined with reflection to produce desired external colors and/or selective color enhancement. The processing is is never complicated and usually involves only one or two simple steps. Projected large scale manufacturing methods however, are inherently capable of multi-step processing without modification to the basic method.

Since absorbing glass is needed in office applications to avoid undesirable internal reflections, it was decided to make any processes developed consistent with commercial glasses such as PPG solarbronze(light brown) or solargray. Single glaze applications with external reflecting layers protected with a hermetic abrasion resistant layer were the only ones considered. Similarly, an internal reflecting layer with protective overcoat on clear glass substrates was the only one considered for residential developments. Tha basic process and materials are the same in the two cases with the reflectivity characteristics being determined by procedural rather than fundamental changes. In order to achieve the optimum performance consistent with lowest cost and weatherability etc., it was therefore necessary to conduct a detailed investigation of the characteristics obtainable with different reflecting materials and/or different overcoat materials. The most critical factor was the reflectivity, including absolute values over a broad spectrum ($.4 \rightarrow 20\ \mu$), as well as threshold and threshold sharpness values. Modifications in the

overcoat for different coloring or weathering factors were known to be controllable.

In addition to applications for new office buildings and residences, there was a secondary interest in the program in retrofit possibilities since the techniques were known to be compatible with plastic substrates. It a therefore also necessary to demonstrate that the results achieved on glass substrates could be duplicated on various potential plastic substrates. In order to make comparisons easily, the program was organized to conduct reflectivity investigations in the early stages on clear residential window glass and a variety of plastics including mylar, polycarbonate, and polyester film. After demonstration of similarity of results for the first reflecting materials studied, subsequent investigations were restricted to glass substrates on the assumption that the results could be duplicated on plastic. For simplicity, weathering studies were restricted in all cases to clear glass substrates. A final demonstration family of colored office windows was made on PPG solarbronze substrates (see Section 3.1).

2.2 Reflectivity

The major goal of this program was to conduct a broadly based systematic investigation to provide a comprehensive understanding of the practical limitations of the basic process. In particular, various combinations of reflecting and protective materials were used on a variety of substrates to determine the optimum obtainable parameters for office windows and, to a lesser degree, for retrofit and residential applications. Judicious choices were made based on such factors as anticipated IR reflectivity and weathering capability for the reflecting layers, and clarity, durability and hermeticity for the overcoat materials.

The major materials investigated were:

Reflecting Layer	Overcoat	Substrate
Aluminum	Al_2O_3	Glass (ordinary window&plate)
Brass	SiO_2	Mylar
Nickel		Polycarbonate
Copper		Polyester
Titanium		
Inconel 671		
Molybdenum		
Silver		
Gold Alloy (42% Au, 38% Cu, 20% Ag&Zn)		

Aluminum and brass reflecting layers were studied in combination with both SiO_2 and Al_2O_3 overcoats and with all substrate materials (Note: Brass-Al_2O_3 was used in weathering studies only-not in reflectivity). The other reflecting materials were studied in combination with SiO_2 overcoats and glass substrates only. In most cases, optical measurements in the visible and near IR range (up to 2.5 μ) were made on an old Beckman DK-2A spectrometer at KCI, and from 2.5 →50 μ (particularly 10 μ point) using a Beckman Mark IV spectrometer at Lincoln Labs.

For the brass and aluminum cases in which four different substrates were used, the latter were coated simultaneously by placing them in the four quadrant positions (covering an area of 4" x 4") about the center of the distribution. The distribution was sufficiently uniform over this area to produce the same reflecting layer thickness to approximately ±4%. Samples for testing weathering resistance were made in separate runs (see Section 2.4). In all cases the samples were cleaned by vapor degreasing in alcohol followed by ultrasonic cleaning in Alconox.

In all other cases, for reflectivity samples a single glass substrate was mounted at the center of the distribution. In general, samples for weathering studies were made in separate runs and involved twin samples mounted uniformly about a center line. One of the samples was retained for reference and the other was placed in the weatherometer. Some of the

weathering samples were also used in the reflectivity study. Those which were have a hyphenated number in Table 1 which lists some of the characteristics of the various samples. All of the samples made specifically for reflectivity studies had overcoat thicknesses of less than .05 μ (A) while weathering samples had thicknesses of .05 μ (A), .125-.175 μ (B), .275-.325 μ (C) or greater than 2 μ (D). Table 1 indicates this difference where necessary.

Table 1 also gives the thickness of the reflecting layers and the reflectivities at 10 μ. The latter were taken vs. an Al standard and are close to the true absolute values. Full curves from 2.5 → 50 μ were taken for some samples (see Section 3.2) but had to be restricted since there was a large number of samples and an outside instrument was used. The curve shapes were similar in all cases so that the single measurement of reflectivity at 10 μ is sufficient to indicate potential performance for residential applications. These values may be used in conjunction with the reflectivity curves (Figures 3-43) for the region from .35-2.5μ to assess overall performance. For the groups for which four differenct substrates were used, the value from the glass substrate is listed. The others did not give exactly the same value but were withing a few percent. Since this was a specular reflectance measurement (15°), it was quite difficult to maintain the mylar and polyester films (mounted in 35 mm slides) at the correct angle for the maximum value. However, the sample angle could be scanned manually to go through the maximum which usually tested as high or higher than the glass substrate. The polycarbonate substrates usually gave a value a few percent lower. This was believed due to the relative roughness of the surface. In general, it was firmly established that the performance on plastic substrates was comparable to that on glass substrates.

The reflecting layer thickness values were calculated from measurements made on calibration standards made as part of each material set (e.g. Cu-SiO_2) study. In some of the early cases, no calibration standards were available and for these relative thicknesses are given. These relative thicknesses were obtained by controlling deposition conditiona and are believed to be accurate to better than ±2% in most cases. Measurements for the

TABLE 1. LAYER CHARACTERISTICS-REFLECTIVITY STUDY

(Dielectric on Reflective Layer - on Substrate)

KEY

Substrate: G ≡ Glass-common window-1/8" thick
PC ≡ Polycarbonate-Rowland Products Inc-2"x2"-3/32" thick
M ≡ Mylar-Arkwright Xerographic-.004"thick, mounted in 35 mm slide holder(2"x2") after coating
P ≡ Polyester-unknown origin-.004" thick, mounted in 35 mm slide holder (2"x2") after coating
No letter designation indicates window glass substrate only to conform with curves.

Dielectric: 1 ≡ Al_2O_3
2 ≡ SiO_2
A ≡ thickness less than .05 μ
B ≡ thickness .125 - .175 μ
C ≡ thickness .275 - .325 μ
D ≡ thickness > 2 μ

Reflecting Layer : Values are calculated from calibration standards measured on Dektak instrument
* means values are only relative values within group.

Sample No.	Substrate	Reflecting Layer	Reflecting Layer Thickness (Å)	Dielectric	Reflectivity (%) at 10μ (glass substrate)
17	G, PC, M, P	Aluminum	1.4*	1B	63.6
18	"	"	2.8*	1A	77.8
19	"	"	.7*	"	24.7
20	"	"	.35*	"	25.0
21	"	"	.2*	"	25.2
22	"	"	.1*	"	25.1
23	"	"	1.4*	"	68.7
24	"	"	1.4*	"	64.9
25	"	"	2.8*	"	82.9

TABLE 1. (continued)

Sample No.	Substrate	Reflecting Layer	Reflecting Layer Thickness (Å)	Dielectric	Reflectivity (%) at 10 μ (glass substrate)
33	G, PC, M, P	Aluminum	2.8*	2A	91.8
36	"	"	1.4*	"	76.0
37	"	"	.7*	"	24.8
38	"	"	.35*	"	24.0
39	"	"	.2*	"	23.5
40	"	"	.1*	"	24.0
46	G, PC, M, P	Brass	298	2A	91.3
48	"	"	149	"	86.2
49	"	"	75	"	65.0
50	"	"	604	"	94.6
51	"	"	37	"	23.0
52	"	"	21	"	22.9
53	"	"	11	"	23.2
55-1		Nickel	171	2A	94.6
58-1		"	341	"	96.7
59		"	32	"	74.0
60		"	64	"	88.0
61		"	128	"	94.0
62		"	48	"	----
63		"	16	"	56.6
65		"	8	"	38.7
66		"	32	2B	76.1
67		"	31	2A	----
68		Copper	402	2A	98.3
69		"	197	2A	96.9
70		"	398	None	97.1
71		"	104	2A	92.6
72		"	52	"	87.0
73		"	155	"	94.6
74		"	78	"	90.2
75		"	26	"	57.5
76		"	39	"	78.8
77		"	33	"	76.1

TABLE 1. (continued)

Sample No.	Substrate	Reflecting Layer	Reflecting Layer Thickness (Å)	Dielectric	Reflectivity (%) at 10 μ (glass substrate)
86		Titanium	32	2A	28.0
87		"	127	"	65.4
88		"	63	"	48.1
90		Inconel 671	120	2A	63.6
92		"	59	"	48.7
93		"	30	"	35.0
94-2		"	59	2C	41.5
95-2		"	59	2B	43.7
96-2		"	59	2A	48.1
97		Molybdenum	266	2A	75.5
98-2		"	66	2B	38.4
99		"	132	2A	67.2
100-2		"	65	2A	36.1
102-1		Silver	1568	2A	98.4
103		"	418	"	93.0
104-2		"	104	"	91.0
106		"	53	"	86.4
107		"	78	"	90.0
108-2		"	78	2C	86.2
109		"	26	2A	74.4
110		"	13	2A	2 .5
111-2		"	78	2B	90.9
112-2		"	78	2B-C	88.9
113		"	41	2A	84.7
114-2		"	79	2A-B	88.9
115		Gold	560	2A	89.2
116		(42% Au	280	"	85.2
117		38% Cu	140	"	55.0
118		20% Ag	70	"	26.1
119		and Zn)	35	"	22.6

calibrated cases were made on two different Dektak instruments with calibration layers in the 100-500 range, corresponding roughly to real sample values. In general the values are consistent with expected ratios from sputtering coefficients, but the noise levels and drift encountered in the measuring instruments indicate that the absolute values should be considered accurate to only ±50%. The values for the brass samples are extremely suspect and do not fall in line with the other materials. From optical measurements and visual observation, the absolute thickness values for the brass samples seem to be roughly a factor of 3 high. This discrepancy was repeated in a separate study on growth mechanism (Section 2.3) and at present is unexplained.

In all cases, however, the reflecting layer thickness was, in practice, determined by the optical characteristics of the finished sample. Once the correct range of deposition conditions for suitable optical results was obtained, the ratios of reflecting layer thickness between samples was determined by controlling the deposition time. Since the deposition conditions could be controlled quite accurately, these ratios are believed to be quite consistent. In addition, the reproducibility of the samples through control of deposition conditions was excellent.

As stated above, the longer IR measurements were made on a dual beam Beckman Mark IV which gives only a specular reflectance value at 15° angle of incidence. In contrast the Beckman DK-2A used for measurements in the .35-2.5 μ range was ideally suited for this project. The particular instrument used measures total reflectance using an integrating sphere to simultaneously collect both the specular and diffuse reflected components. For total reflectance measurements the angle of incidence is 5°. The diffuse component alone can be obtained by changing the angle of incidence to 0° whereby the specular component is reflected back out of the sphere leaving only the diffuse component to be collected.

MgO reference standards were used for almost all measurements on the DK-2A. These standards have very high absolute reflectance values (.97-.99) in the visible and up to .9 μ. Above this they fall off relative to a fresh Ag or Cu standard. However, the reflectivity had to be measured in

two overlapping segments (.35-.8 μ and .5-2.5 μ) in order to cover the whole spectrum. Using MgO standards for both regions facilitated curve matching and comparison since the .7 μ point was in good agreement in most cases. The error in doing this occurs in the 1.5 to 2.5 μ area where the measured values run from approximately 0-5% high at 1.5 μ to 0-10% high at 2.5 μ. The spread is due to the difference in reflecting layer thickness and varies with thickness and spectral region. There is no similar effect in the transmittance measurements.

After the present program was completed, an internal KCI program was carried out to establish cross-referenced standards. The results of this program have been incorporated into the conclusions on the performance of the windows developed under the present program (see Section 3).

Basically, the reflectivity studies were designed to yield comparisons of the performance of different reflecting layer-overcoat combinations on a variety of substrates. An enormous amount of data was generated, not all of which could be presented in this report. However, the curves etc which are included are believed adequate to support all of the pertinent conclusions. Many of the curves on the DK-2A were repeated many times and were reproducible within the stability (maximum error ±2% - usually ± 1%) and calibration of the instrument. The error due to calibration being not precisely flat has been ignored as inconsequential in drawing conclusions. This error which was a much as 5-6% in some spectral regions when the instrument was obtained, was reduced through overhaul to 2-3% or better by the end of the program.

Figures 3-43 present the main reflectivity data in the same order as Table 1. Basically there is a set of two curves for each reflecting layer-overcoat combination and, where applicable, there is a set for each substrate used. For obvious reasons, the highest reflectivity curves on each set represent the thickest reflecting layer and vice versa. The following symbolism has been used throughout this report:

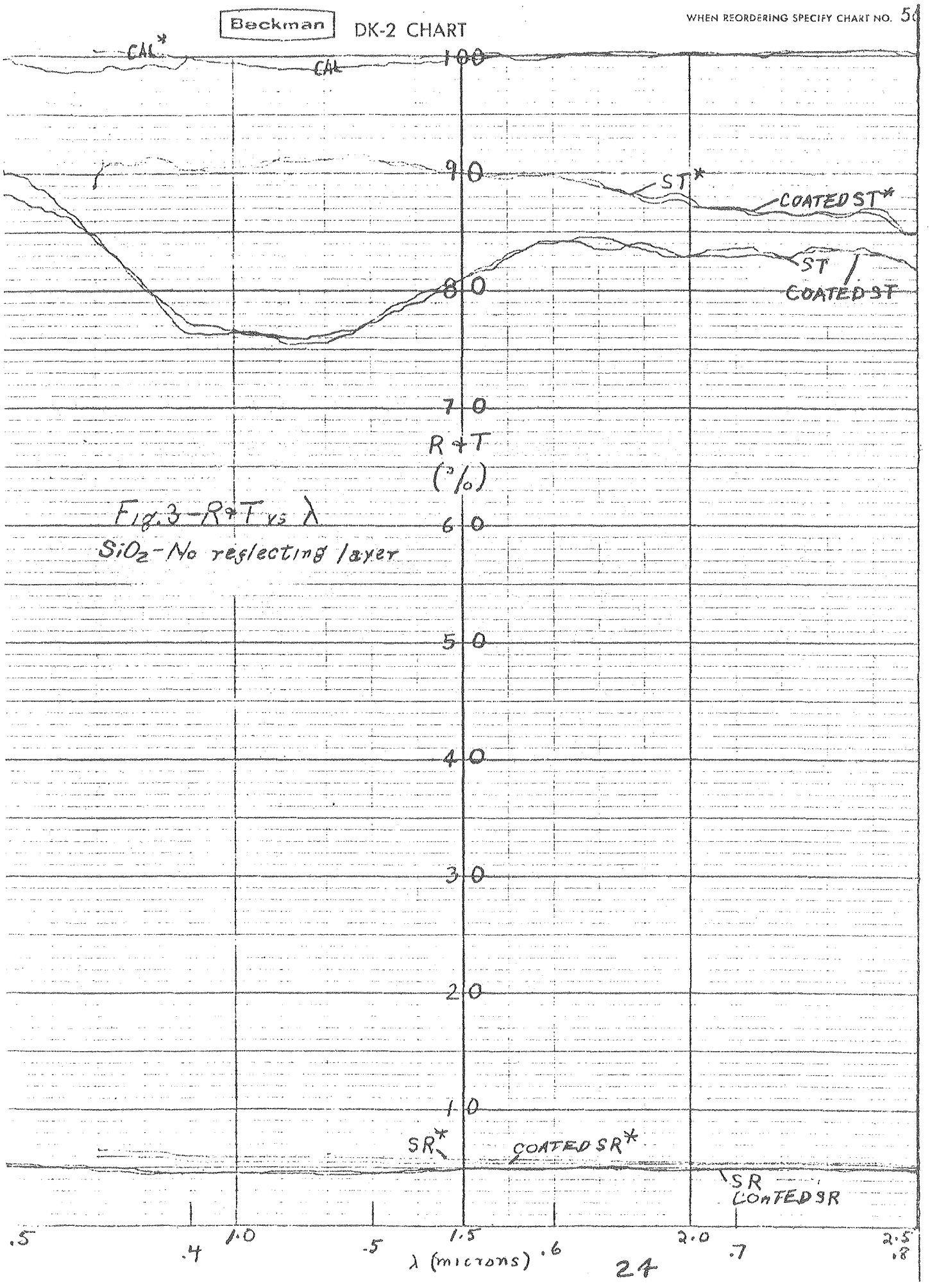


Fig. 3 – R+T vs λ
SiO_2 – No reflecting layer

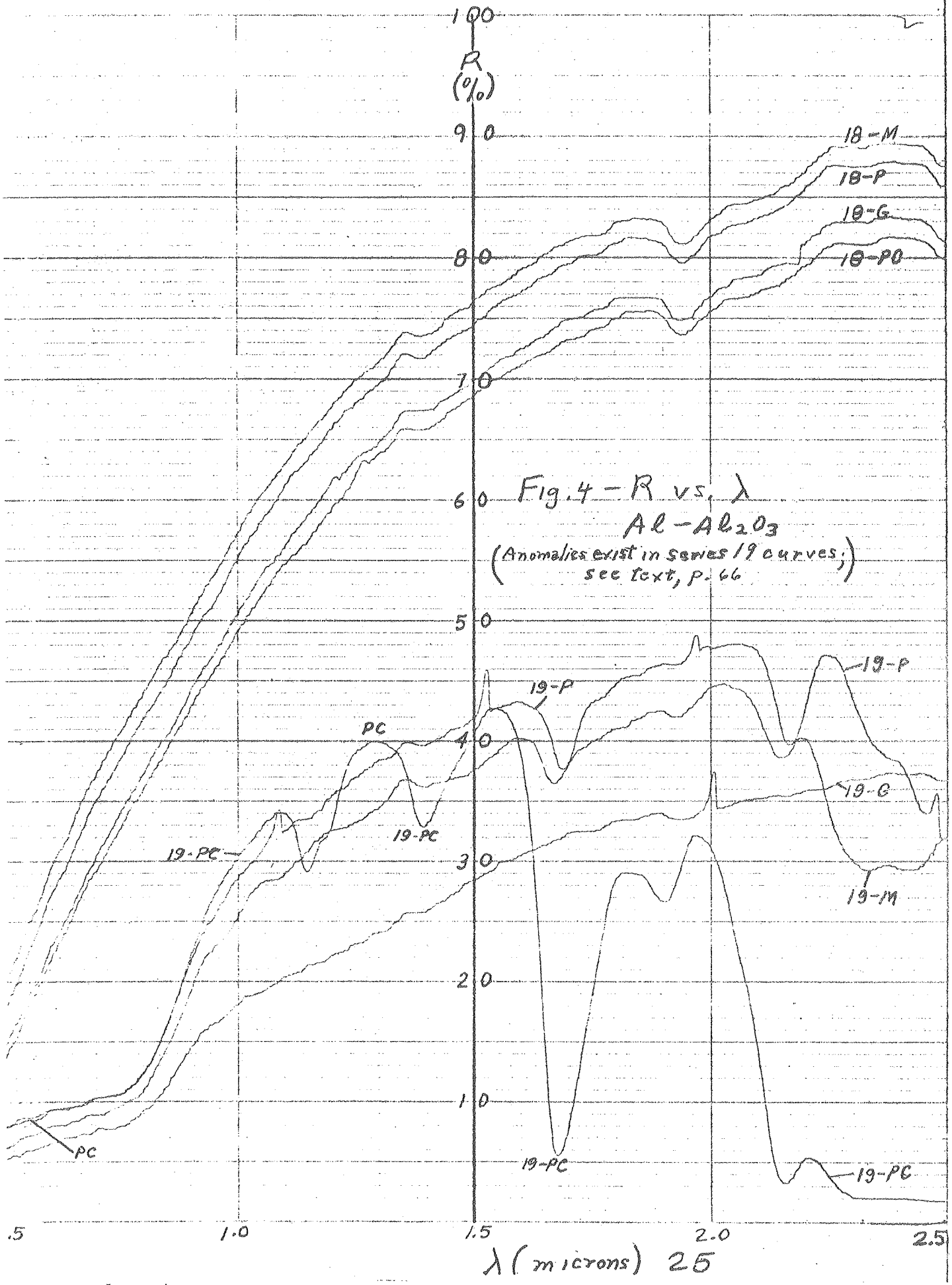


Fig. 4 – R vs. λ
Al – Al_2O_3
(Anomalies exist in series 19 curves; see text, p. 66)

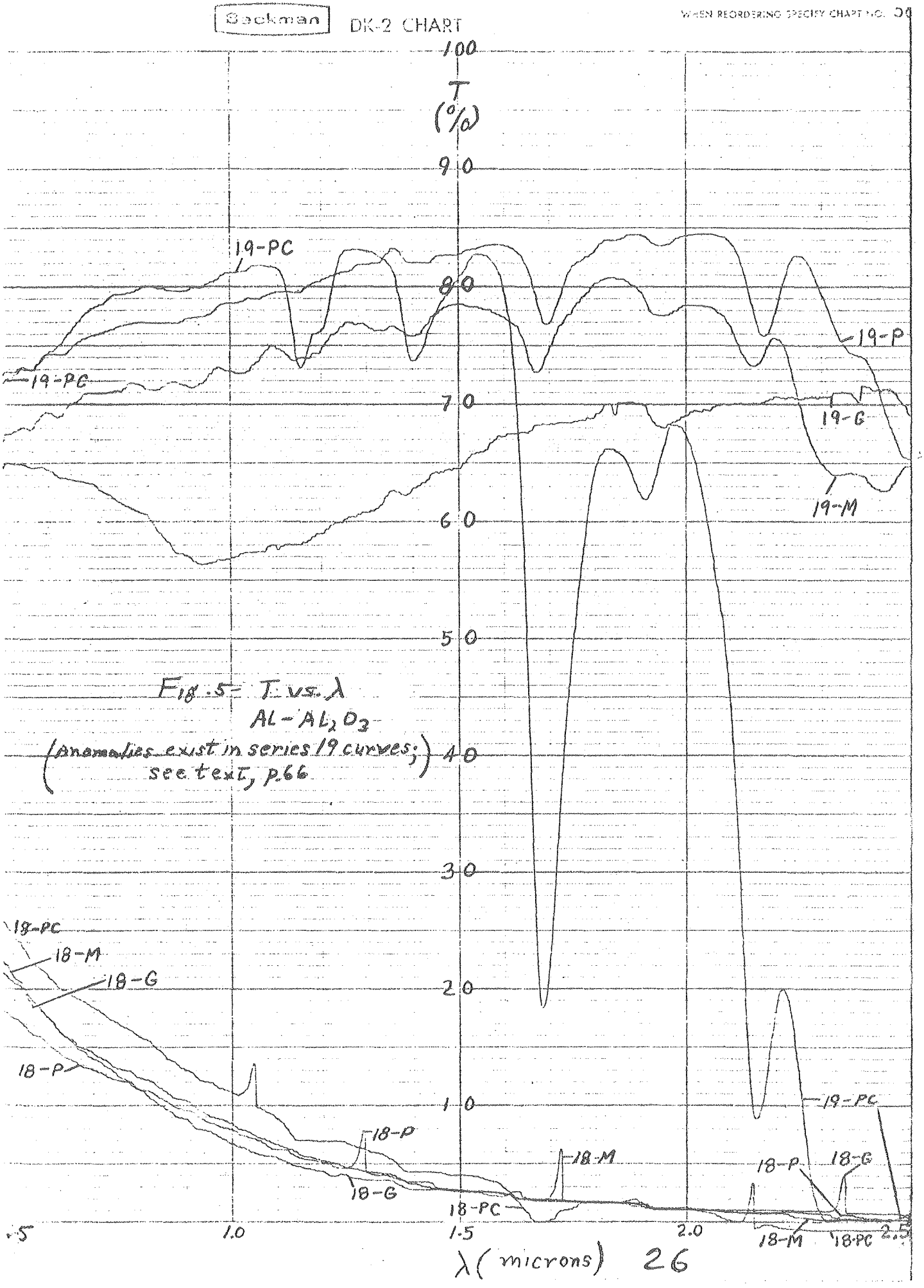


Fig. 5 – T vs. λ
Al – Al_2O_3
(anomalies exist in series 19 curves; see text, p.66)

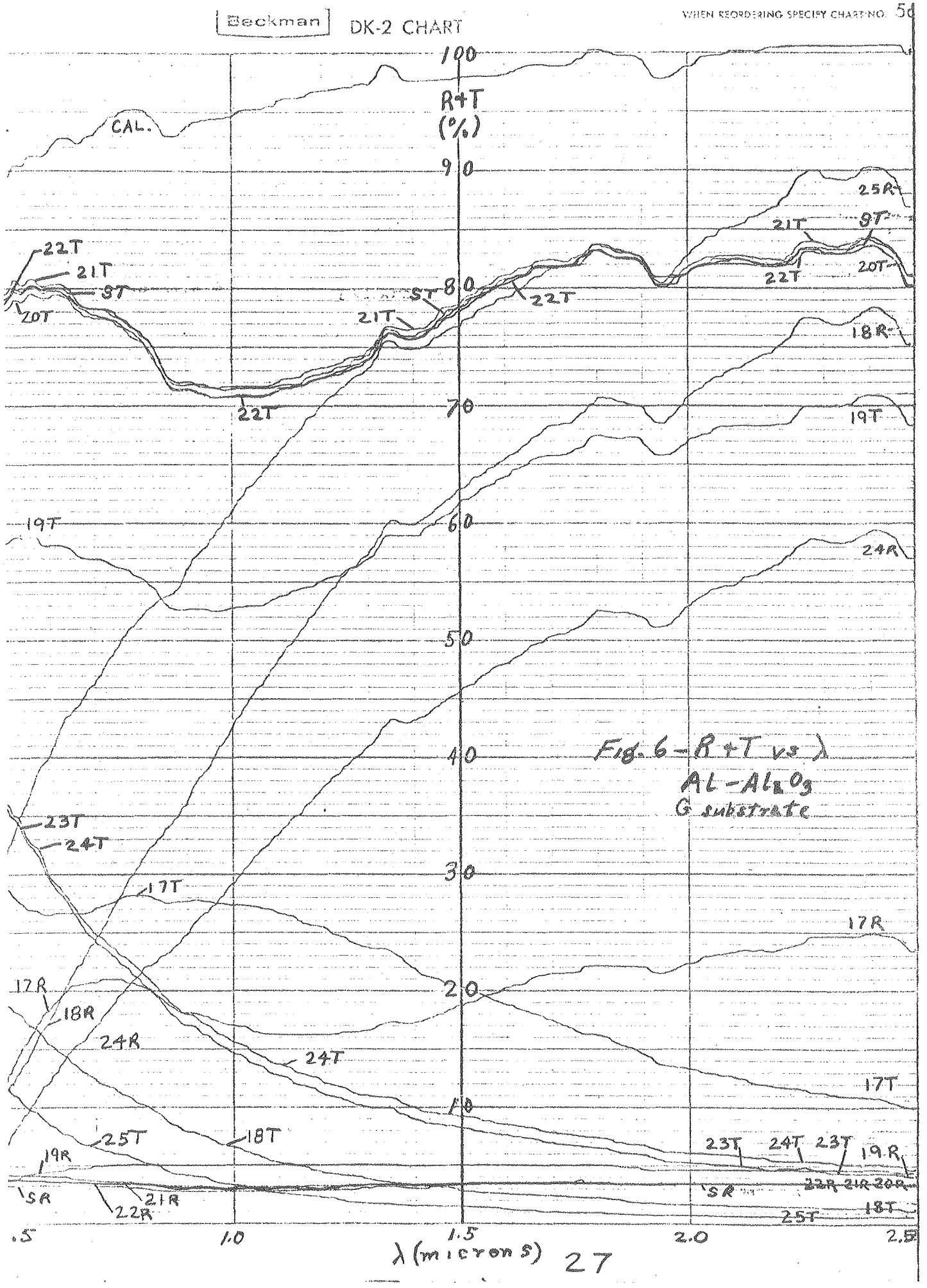


Fig. 6 - R + T vs λ
Al - Al_2O_3
G substrate

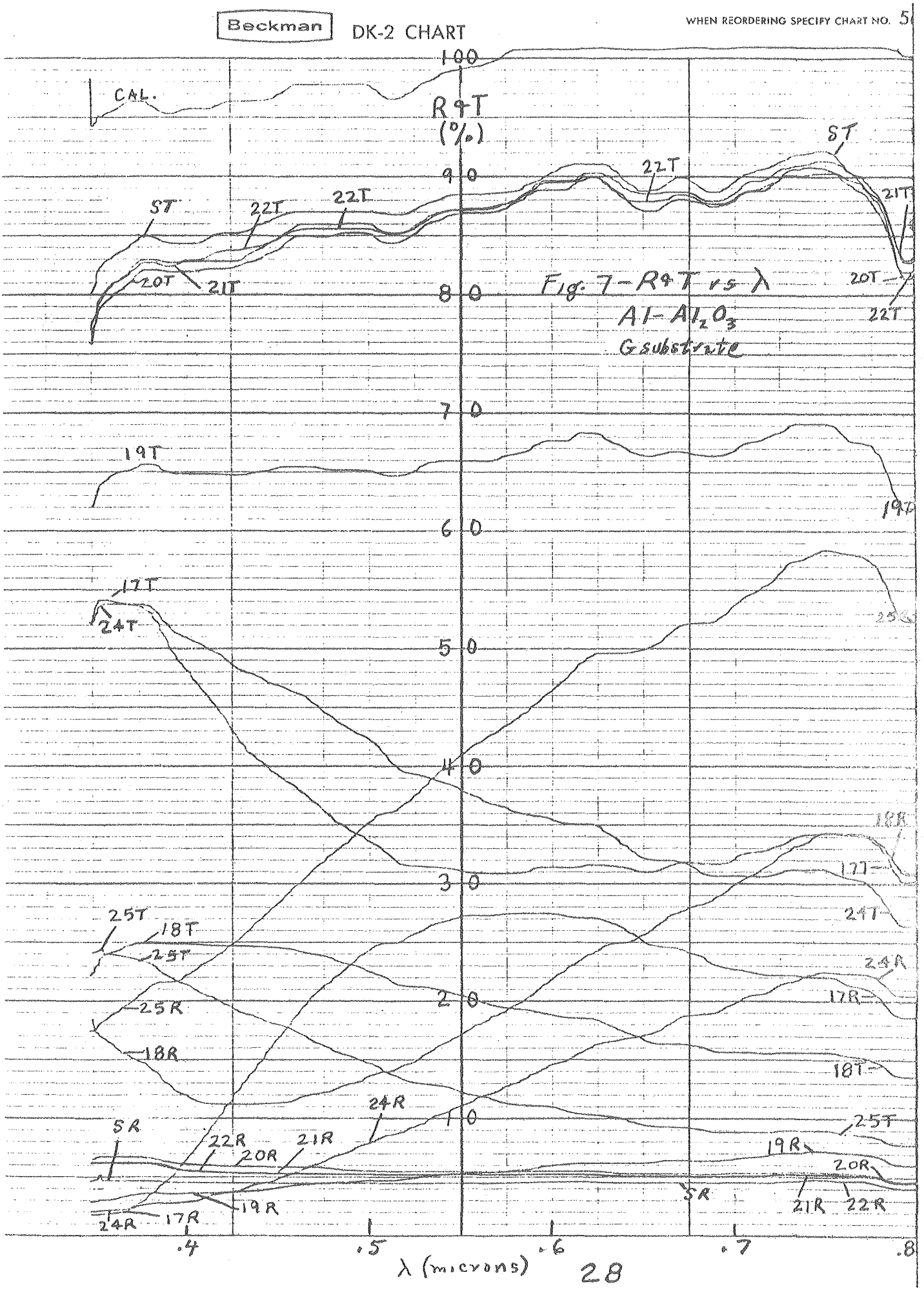
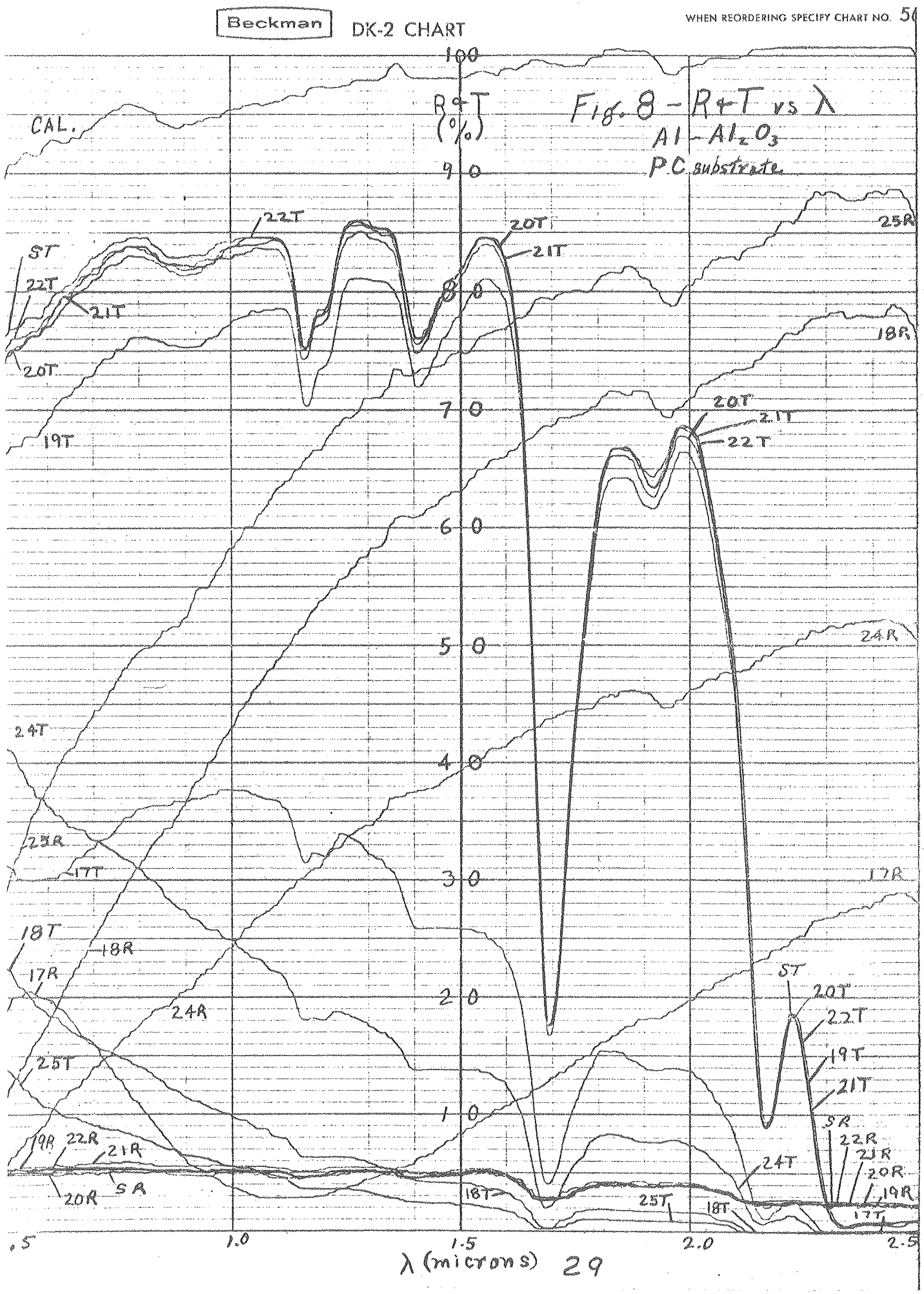


Fig. 7 – R+T vs λ
Al – Al_2O_3
G substrate


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 50
Fig. 8 - R+T vs λ
Al - Al2O3
P.C. substrate
R+T (%)
100
90
80
70
60
50
40
30
20
10
CAL.
22T
20T
21T
5T
22T
21T
20T
19T
25R
18R
20T
21T
22T
24R
24T
23R
17T
17R
18T
18R
17R
24R
5T
20T
22T
19T
21T
25T
19R
22R
21R
20R
5R
18T
25T
18T
24T
5R
22R
21R
20R
19R
17T
.5
1.0
1.5
2.0
2.5
λ (microns)

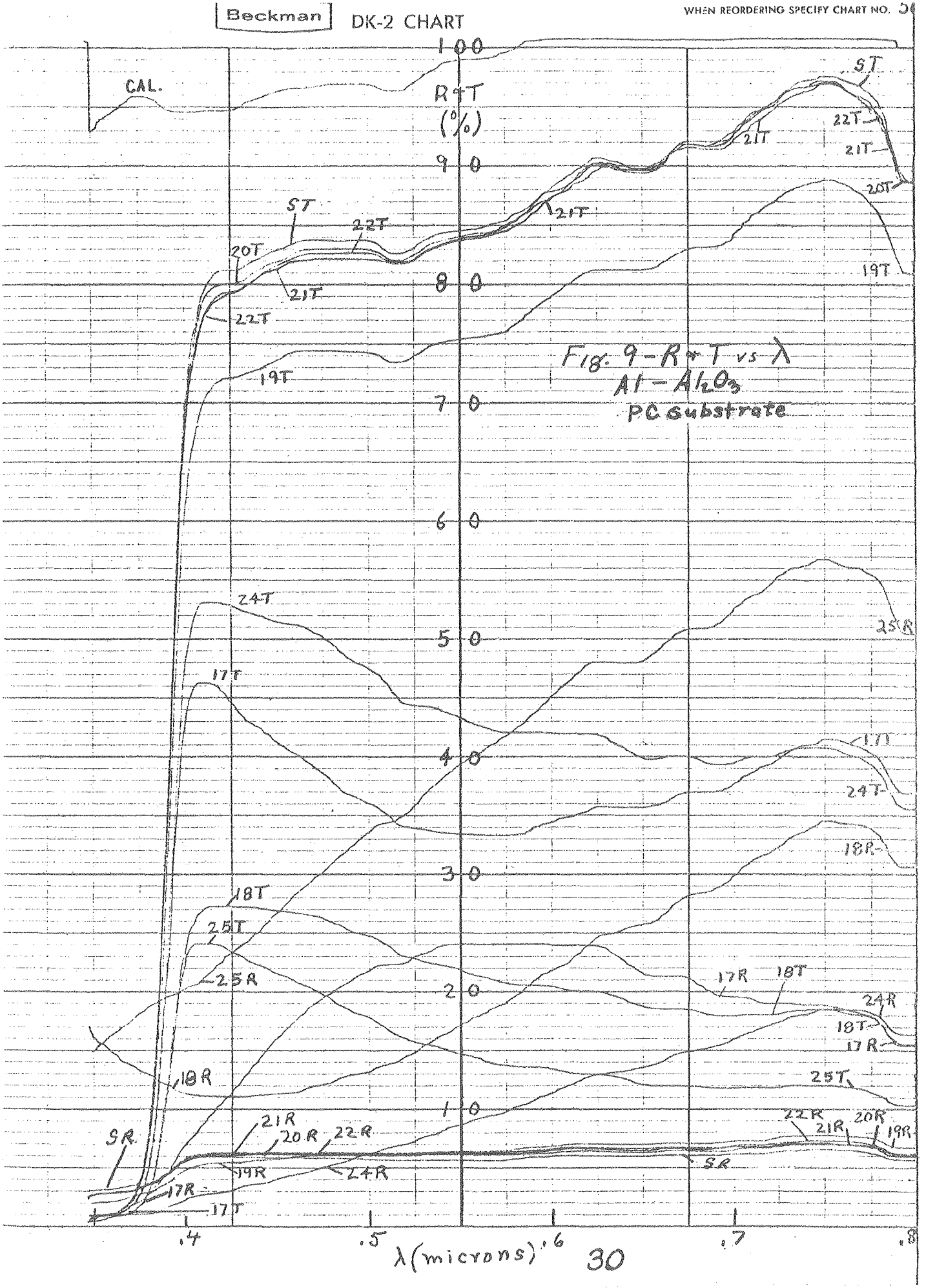
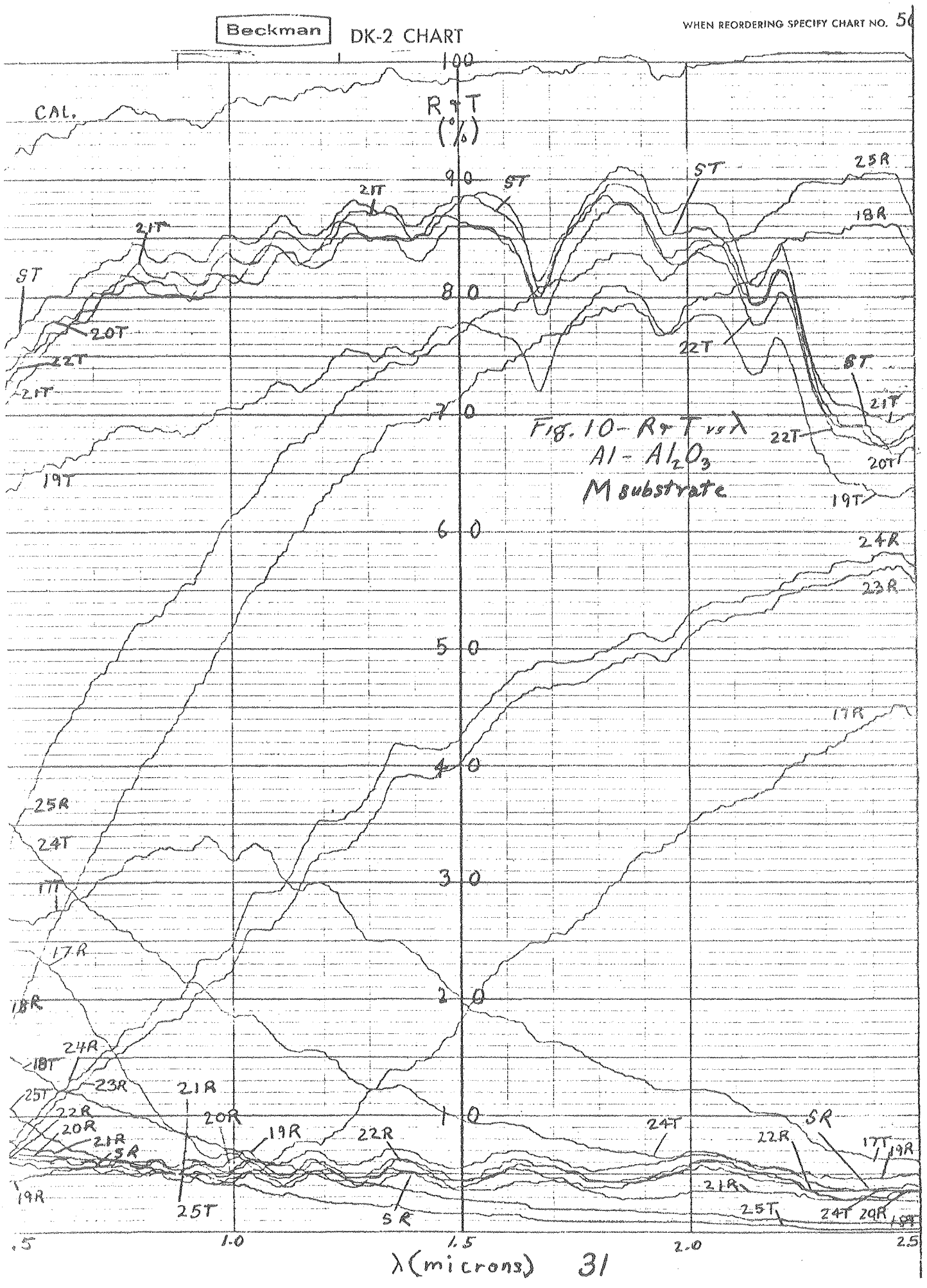
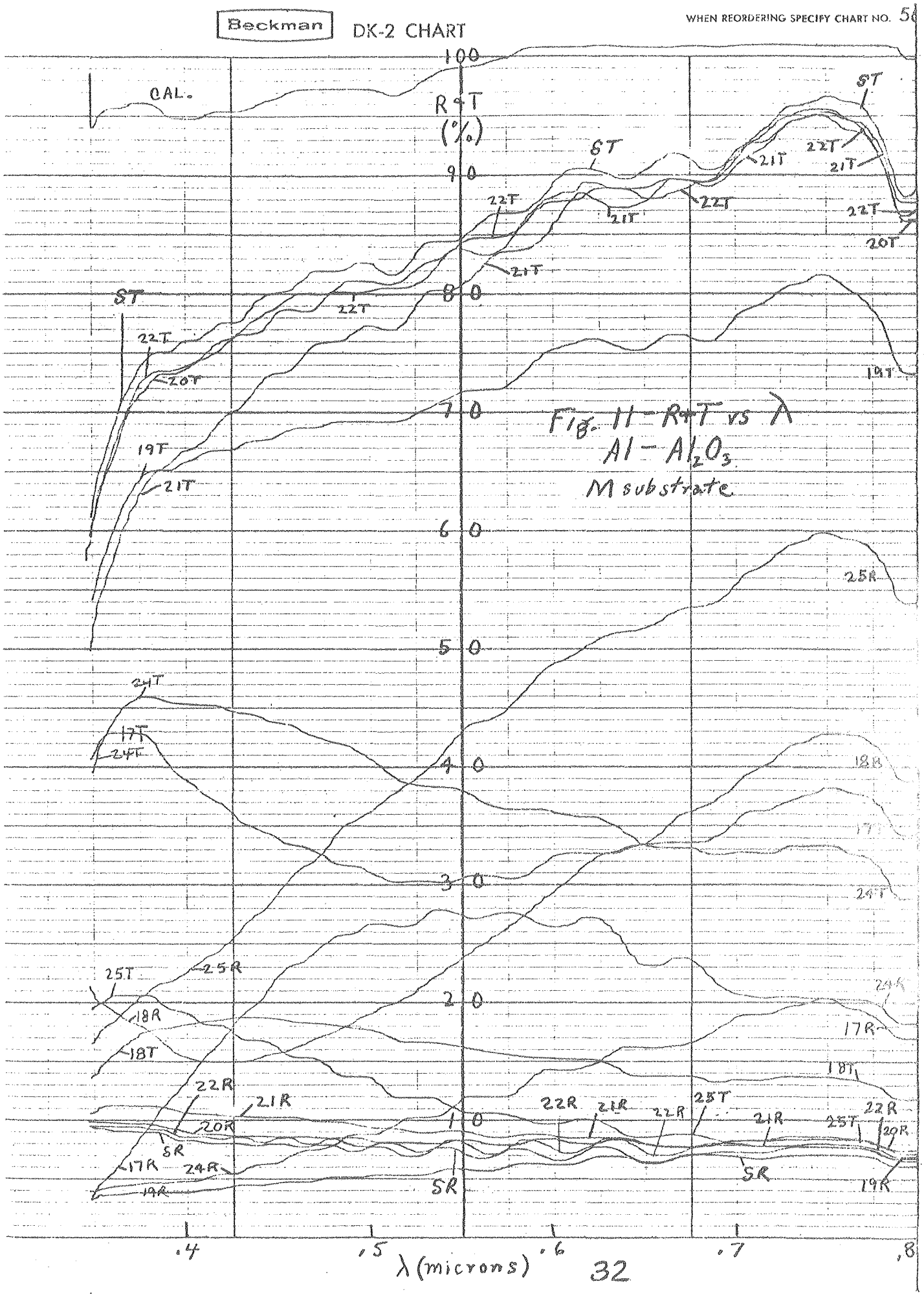


Fig. 9 - R + T vs λ
Al - Al_2O_3
PC substrate


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 50
R+T
(%)
100
90
80
70
60
50
40
30
20
10
CAL.
5T
21T
20T
22T
19T
25R
18R
24R
23R
17R
24T
17T
18T
25T
21R
20R
19R
22R
5R
.5
1.0
1.5
2.0
2.5
λ (microns)
Fig. 10 - R+T vs λ
Al - Al_2O_3
M substrate


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 56
CAL.
R+T
(%)
100
90
80
70
60
50
40
30
20
10
.4
.5
.6
.7
.8
λ (microns)
Fig. 11 - R+T vs λ
Al - Al2O3
M substrate
ST
6T
22T
21T
20T
19T
25R
24T
17T
18R
18T
17R
24R
19R
25T
SR

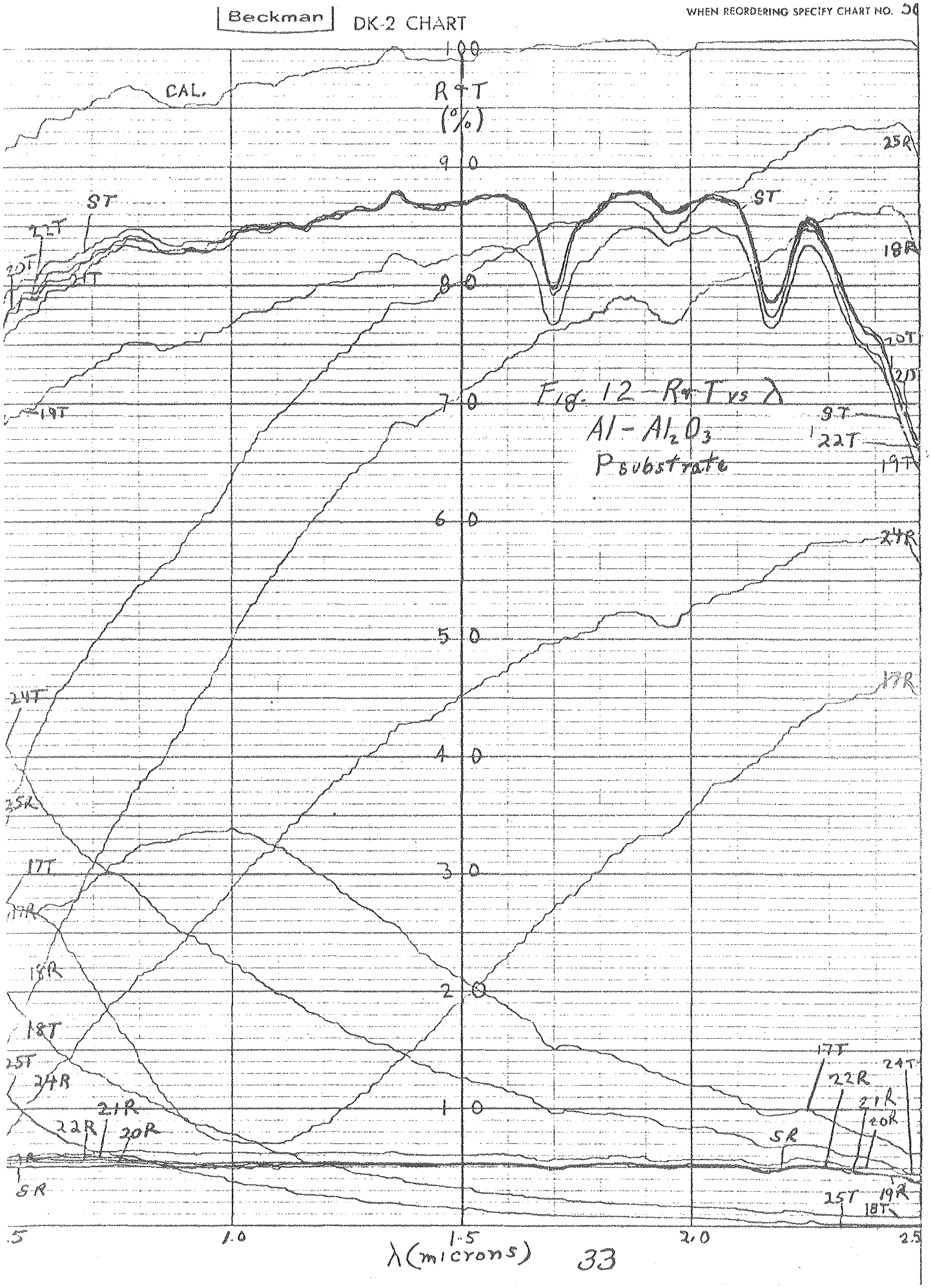


Fig. 12 – R+T vs λ
Al – Al_2O_3
P substrate

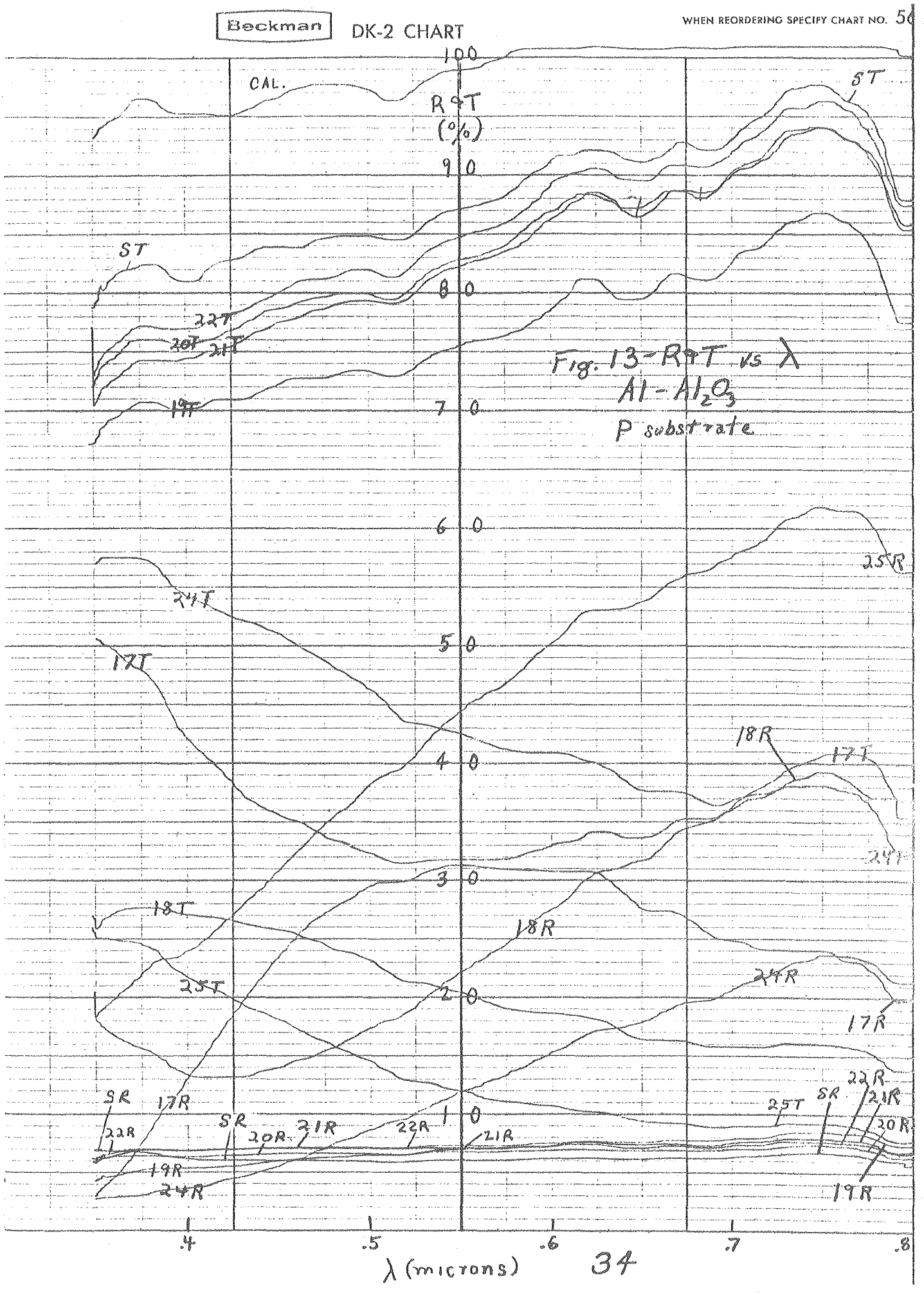


Fig. 13 - R&T vs λ
Al - Al_2O_3
P substrate

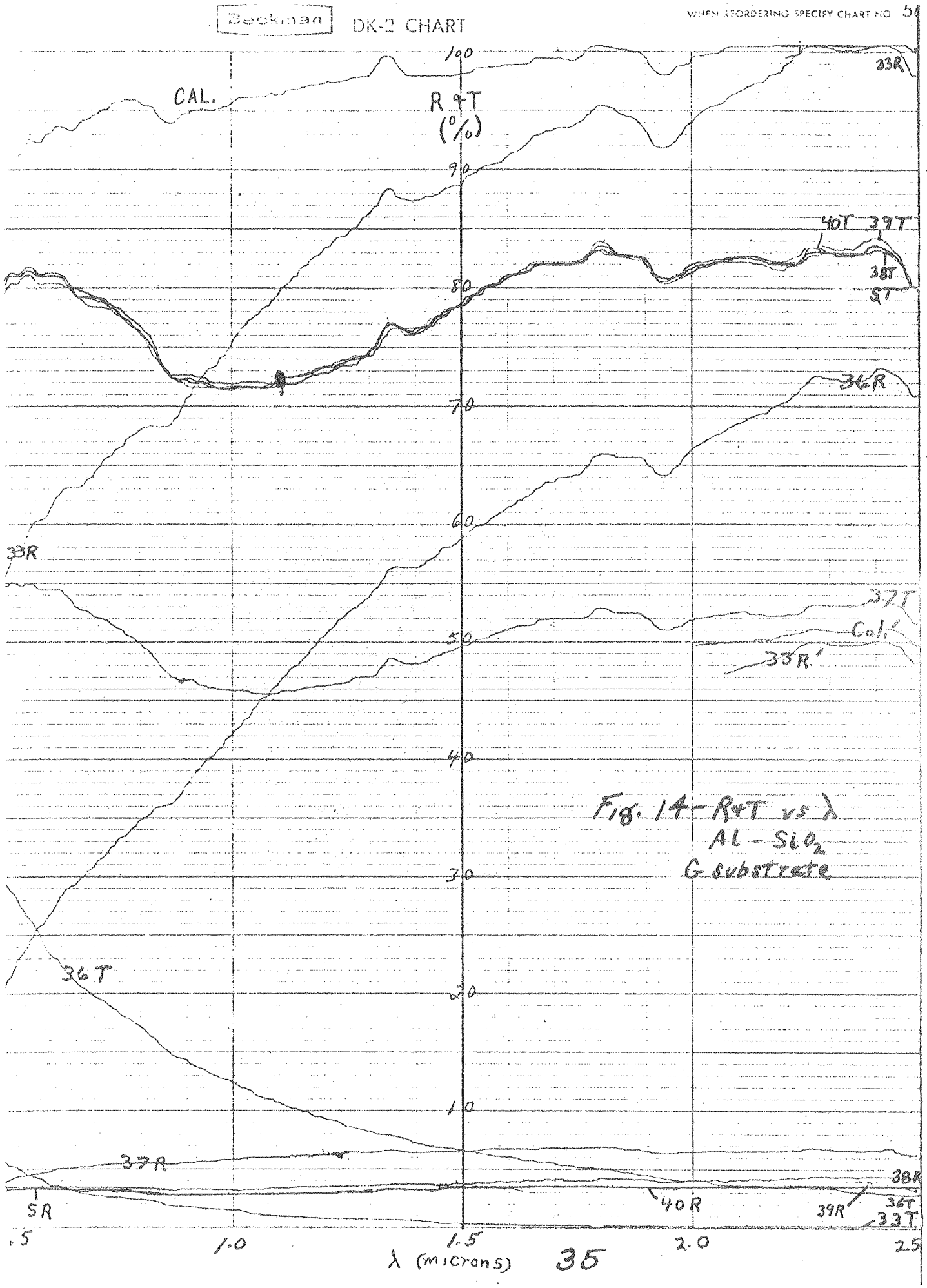


Fig. 14 – R&T vs λ
Al – SiO_2
G substrate

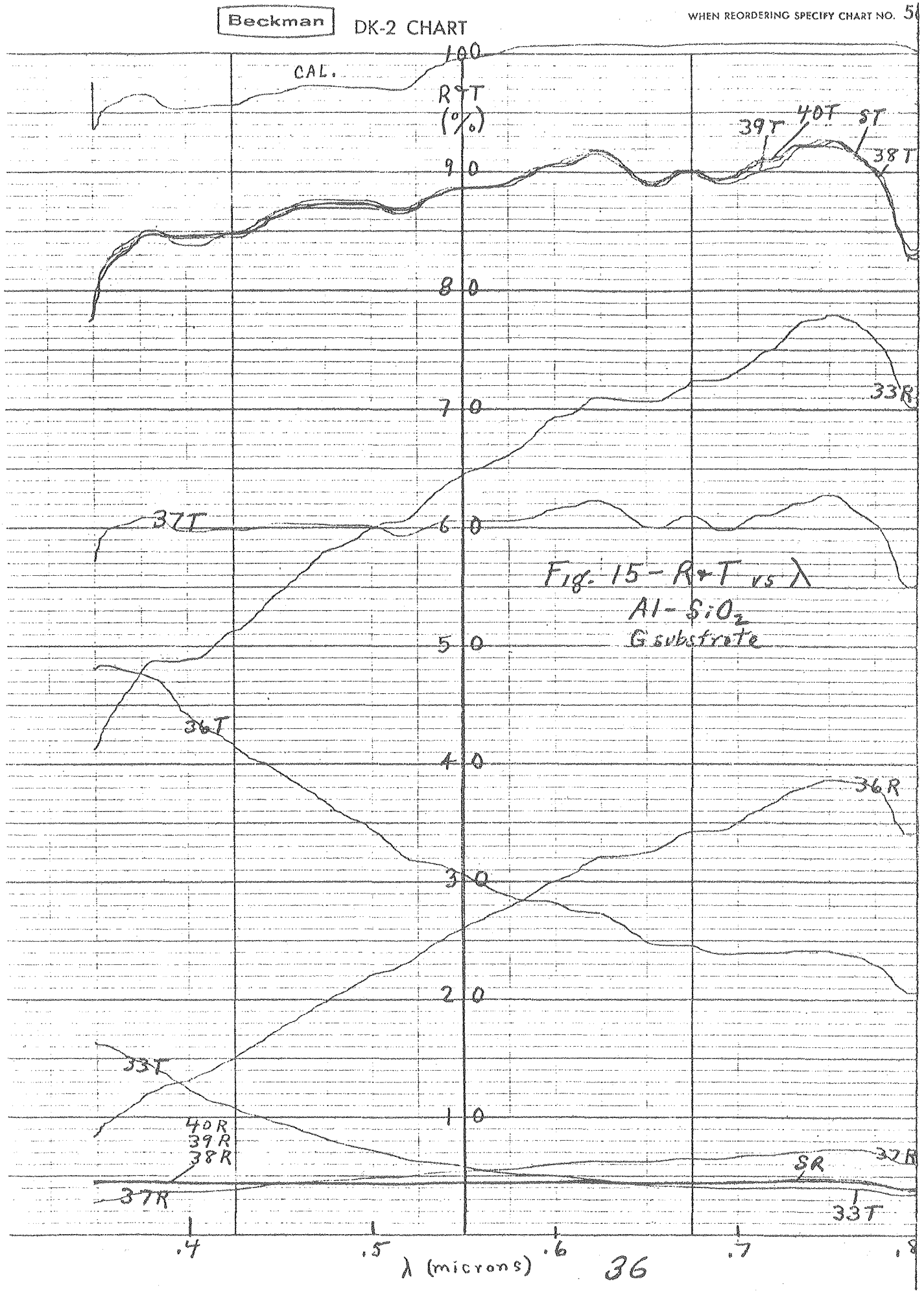


Fig. 15 – R+T vs λ
Al – SiO_2
G substrate

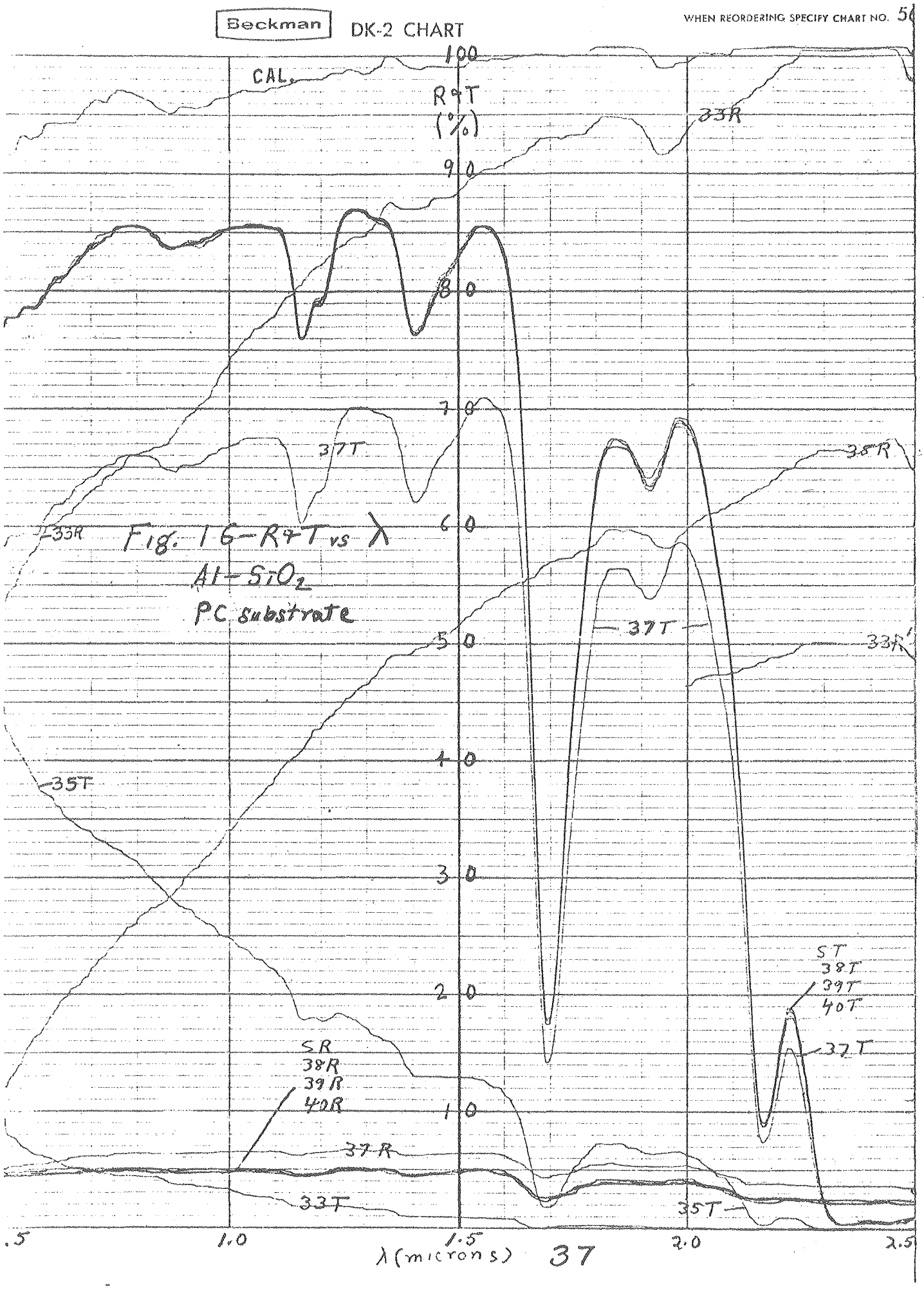
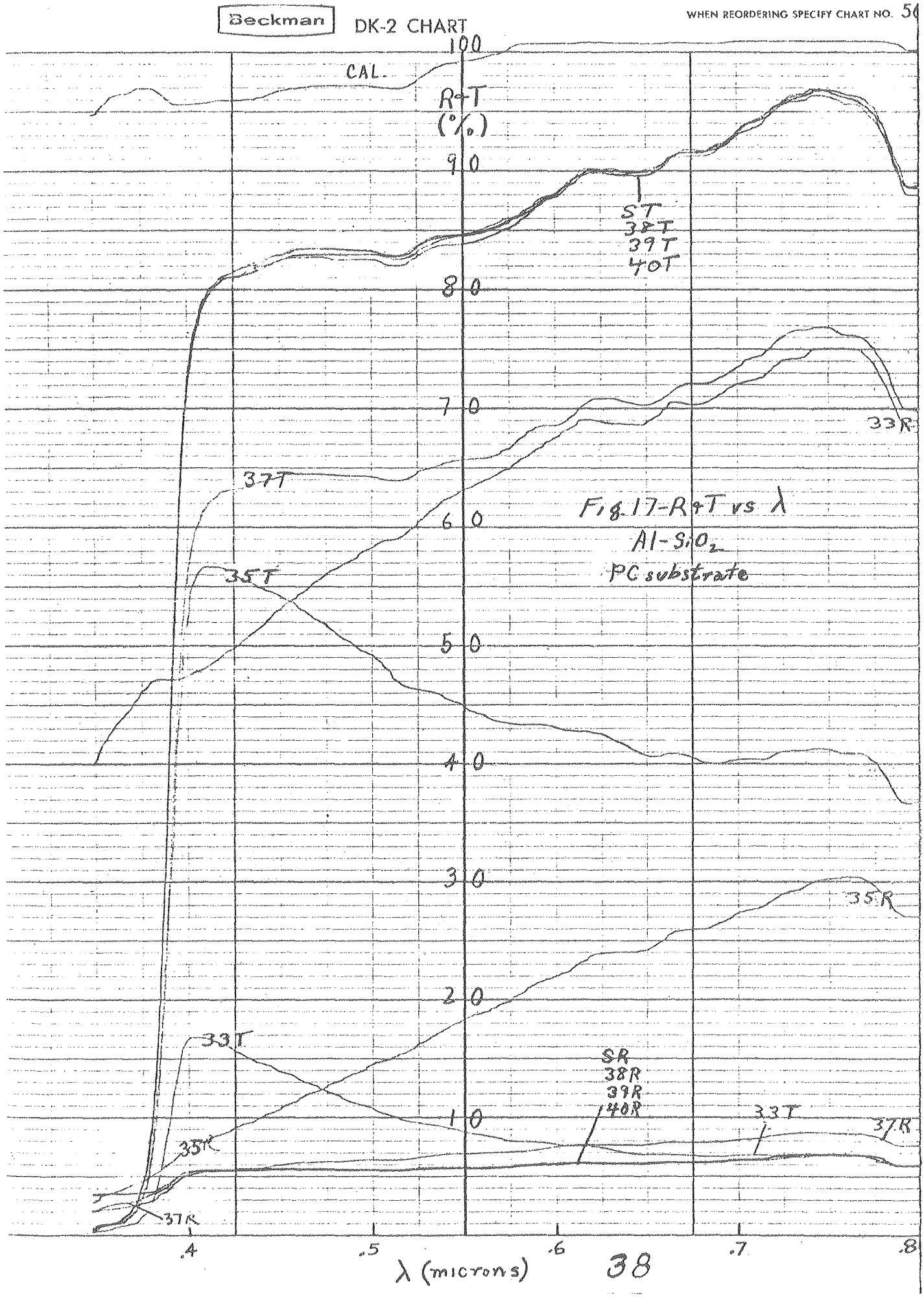


Fig. 16 – R+T vs λ
Al – SiO_2
PC substrate


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 56
CAL.
R+T
(%)
100
90
80
70
60
50
40
30
20
10
5T
38T
39T
40T
33R
37T
35T
35R
33T
5R
38R
39R
40R
33T
37R
35R
37R
.4
.5
.6
.7
.8
λ (microns)
Fig. 17-R+T vs λ
Al-SiO2
PC substrate

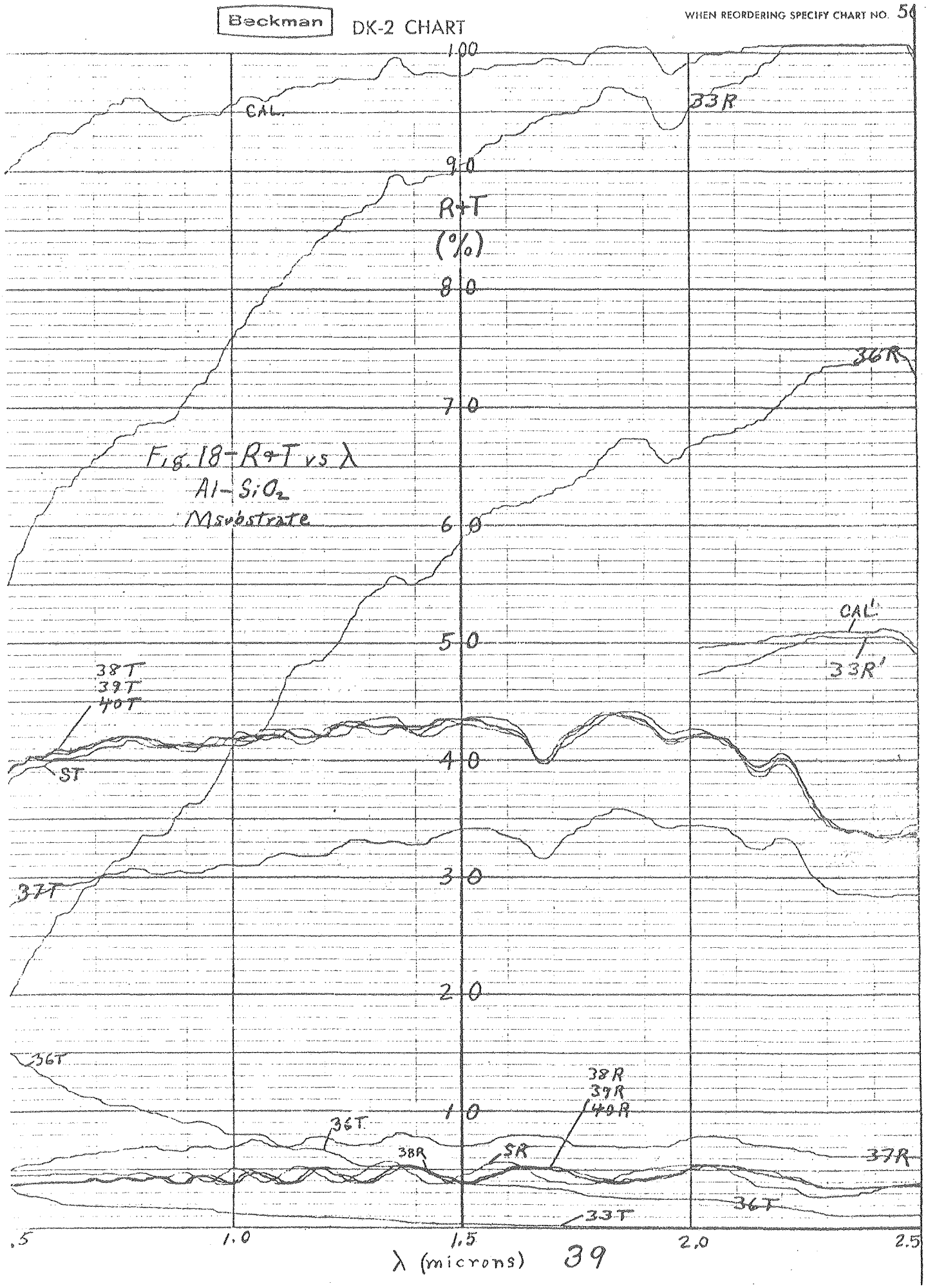


Fig. 18 - R+T vs λ
Al - SiO_2
Msubstrate

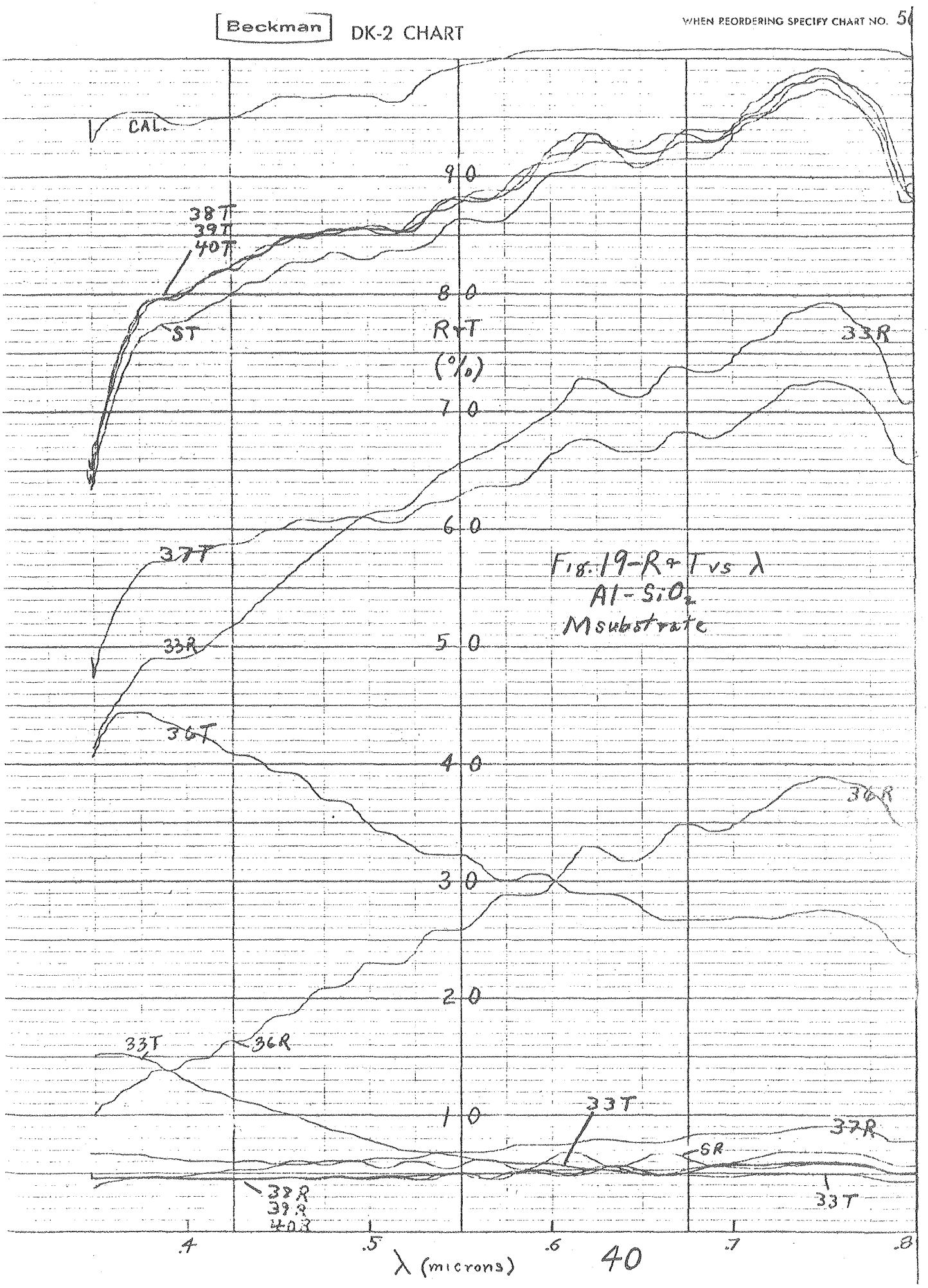


Fig. 19 - R + T vs λ
Al - SiO_2
M substrate

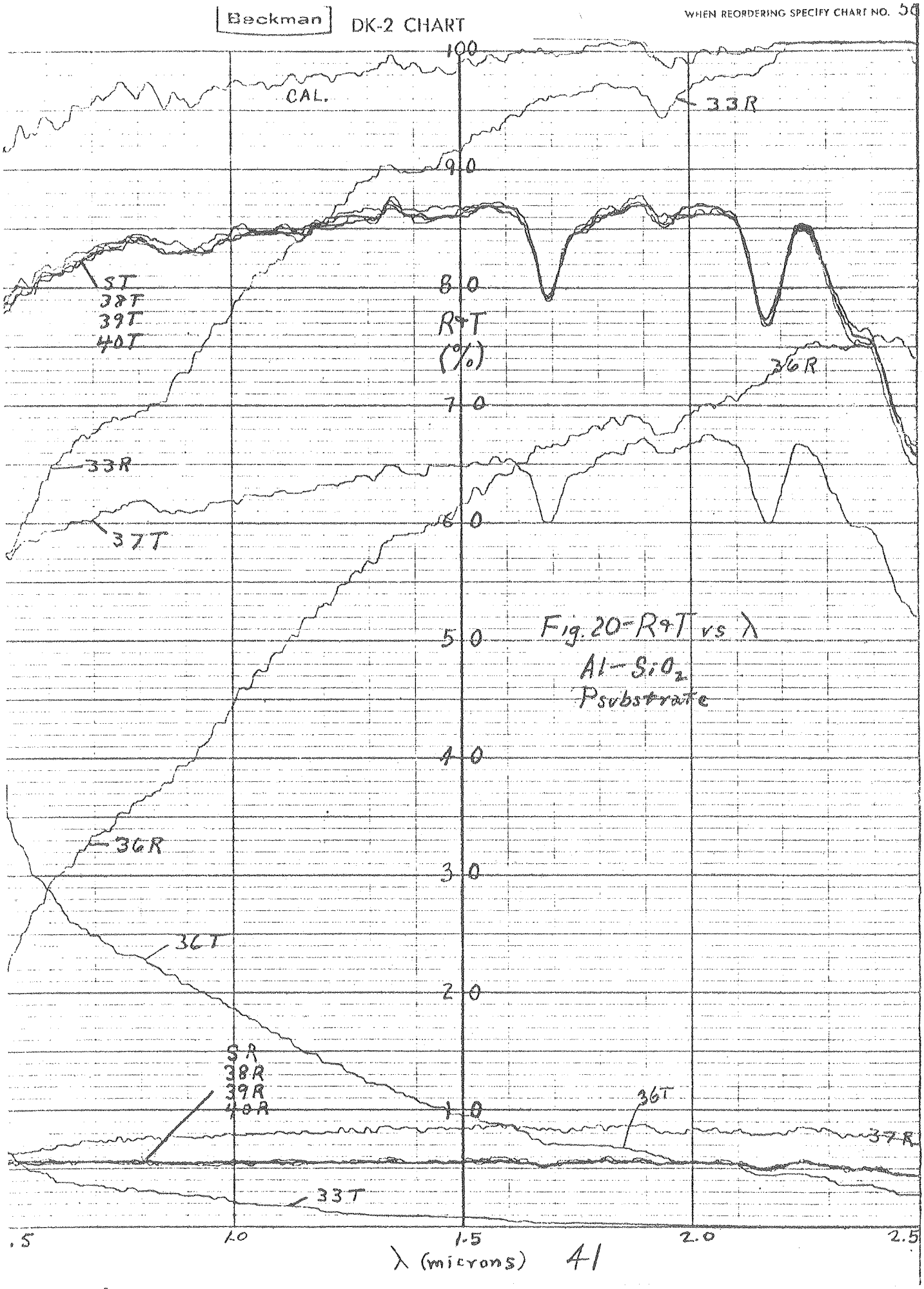


Fig. 20 - R+T vs λ
Al - SiO_2
Psubstrate

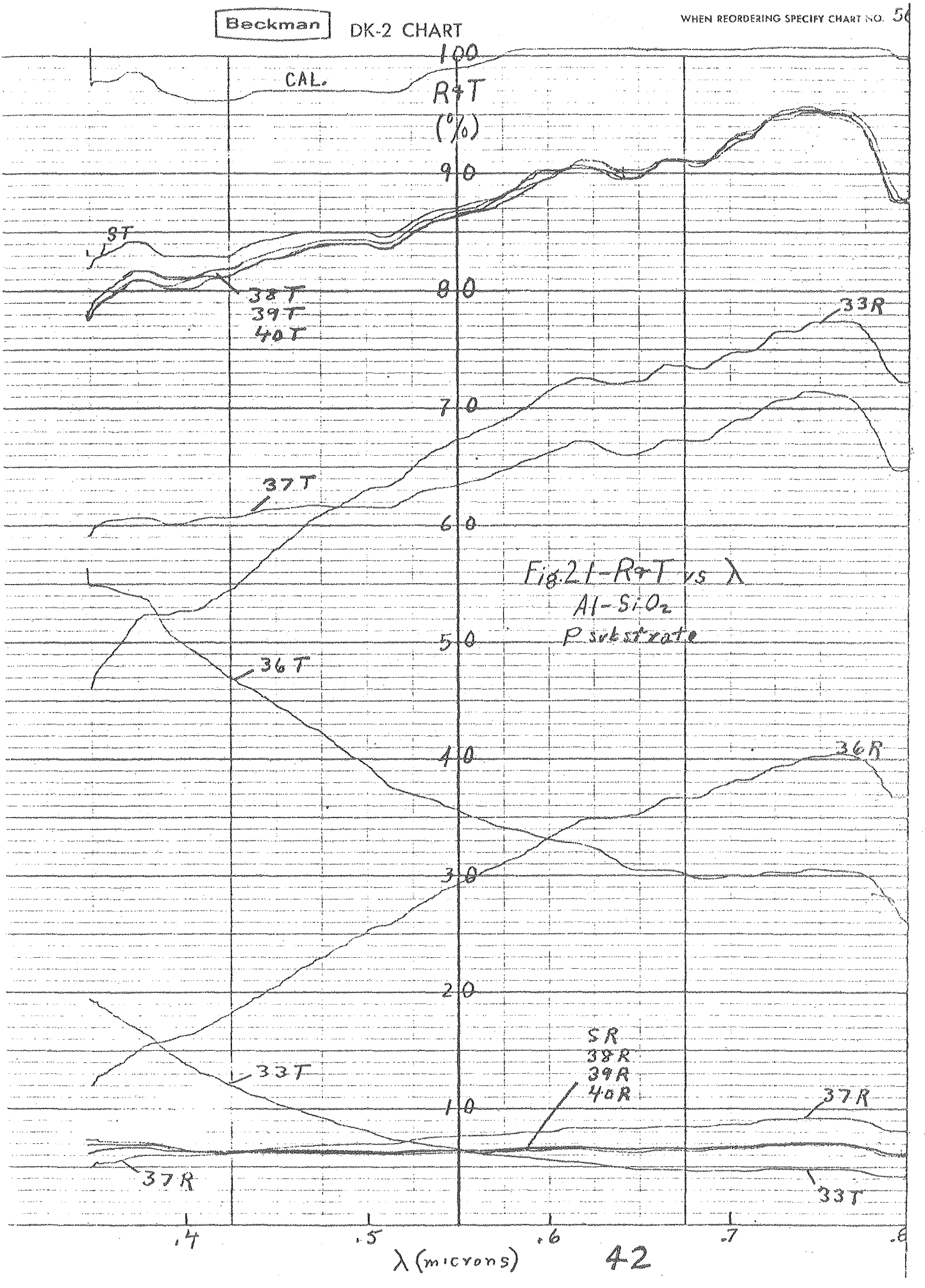
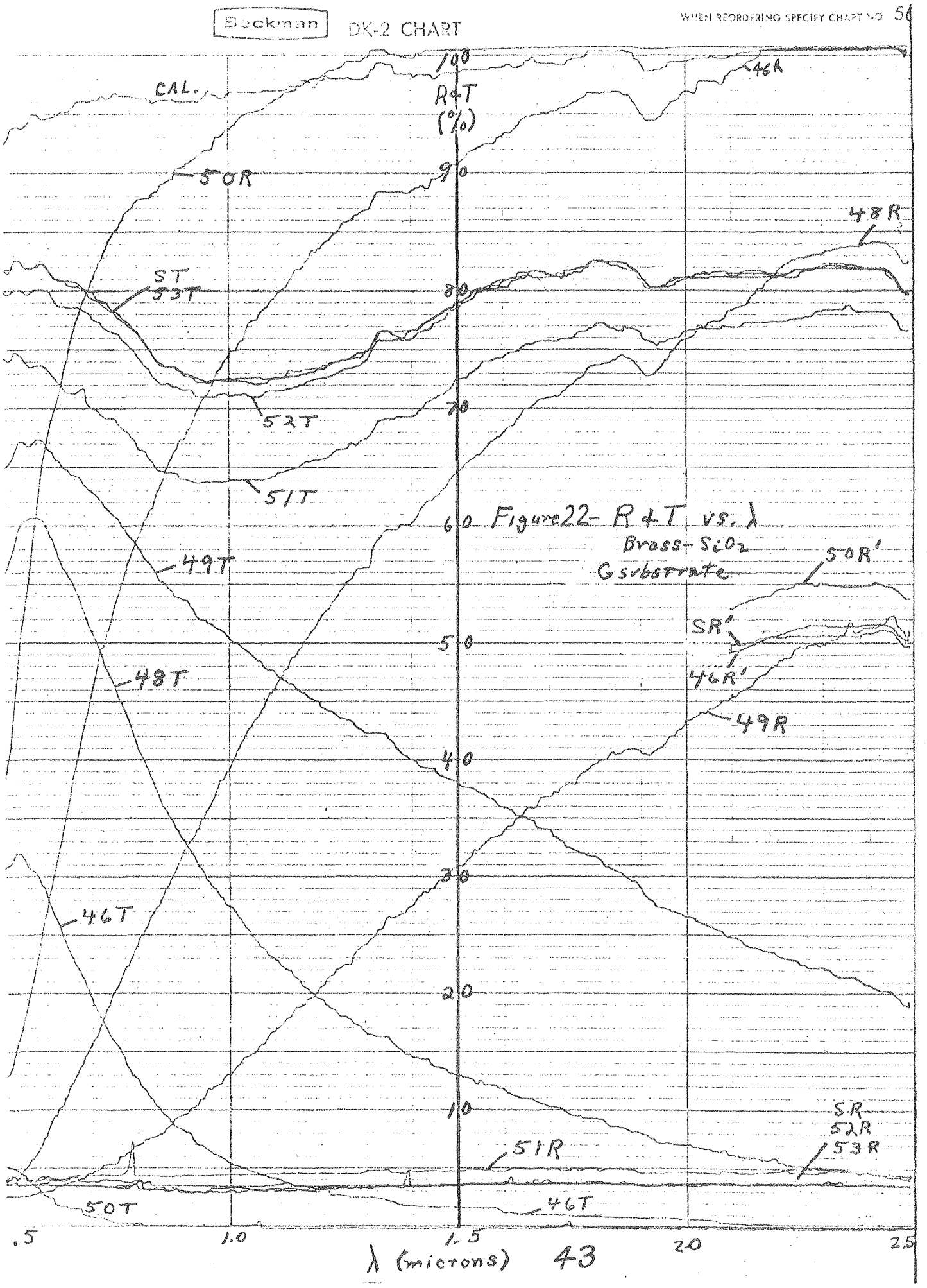
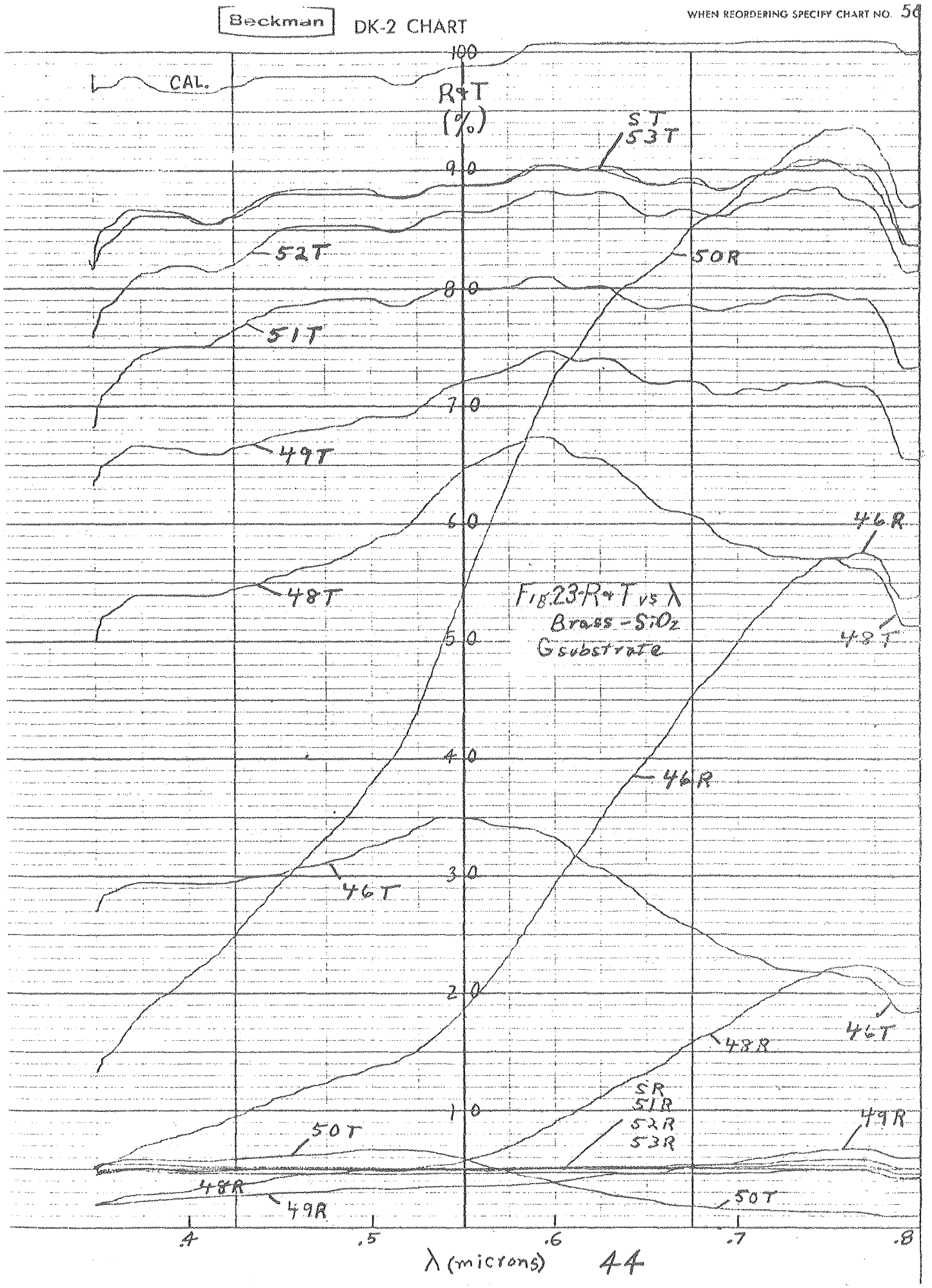


Fig. 21 - R+T vs λ
Al-SiO2
P substrate


Beckman DK-2 CHART
WHEN REORDERING SPECIFY CHART NO 56
CAL.
R+T (%)
100
90
80
70
60
50
40
30
20
10
46R
50R
48R
ST
53T
52T
51T
49T
48T
46T
50R'
SR'
46R'
49R
SR
52R
53R
51R
50T
46T
.5
1.0
1.5
2.0
2.5
λ (microns)
Figure 22- R & T vs. λ
Brass-SiO2
G substrate


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 56
CAL.
R+T
(%)
100
90
80
70
60
50
40
30
20
10
5T
53T
52T
50R
51T
49T
46R
48T
48T
46R
46T
46T
48R
5R
51R
52R
53R
49R
50T
48R
49R
50T
.4
.5
.6
.7
.8
λ (microns)
Fig.23-R+T vs λ
Brass-SiO2
G substrate

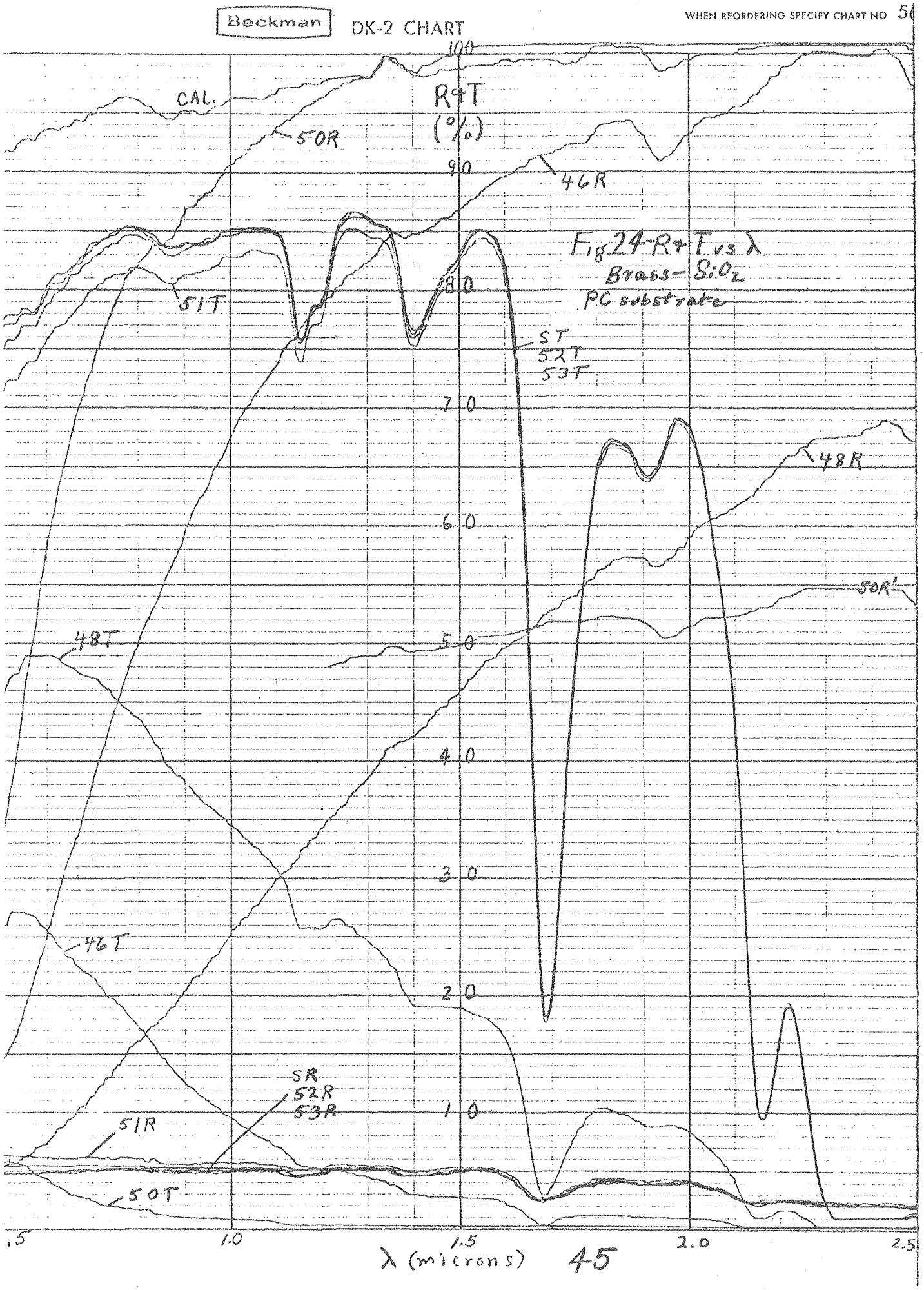


Fig. 24 R+T vs λ
Brass – SiO_2
PC substrate

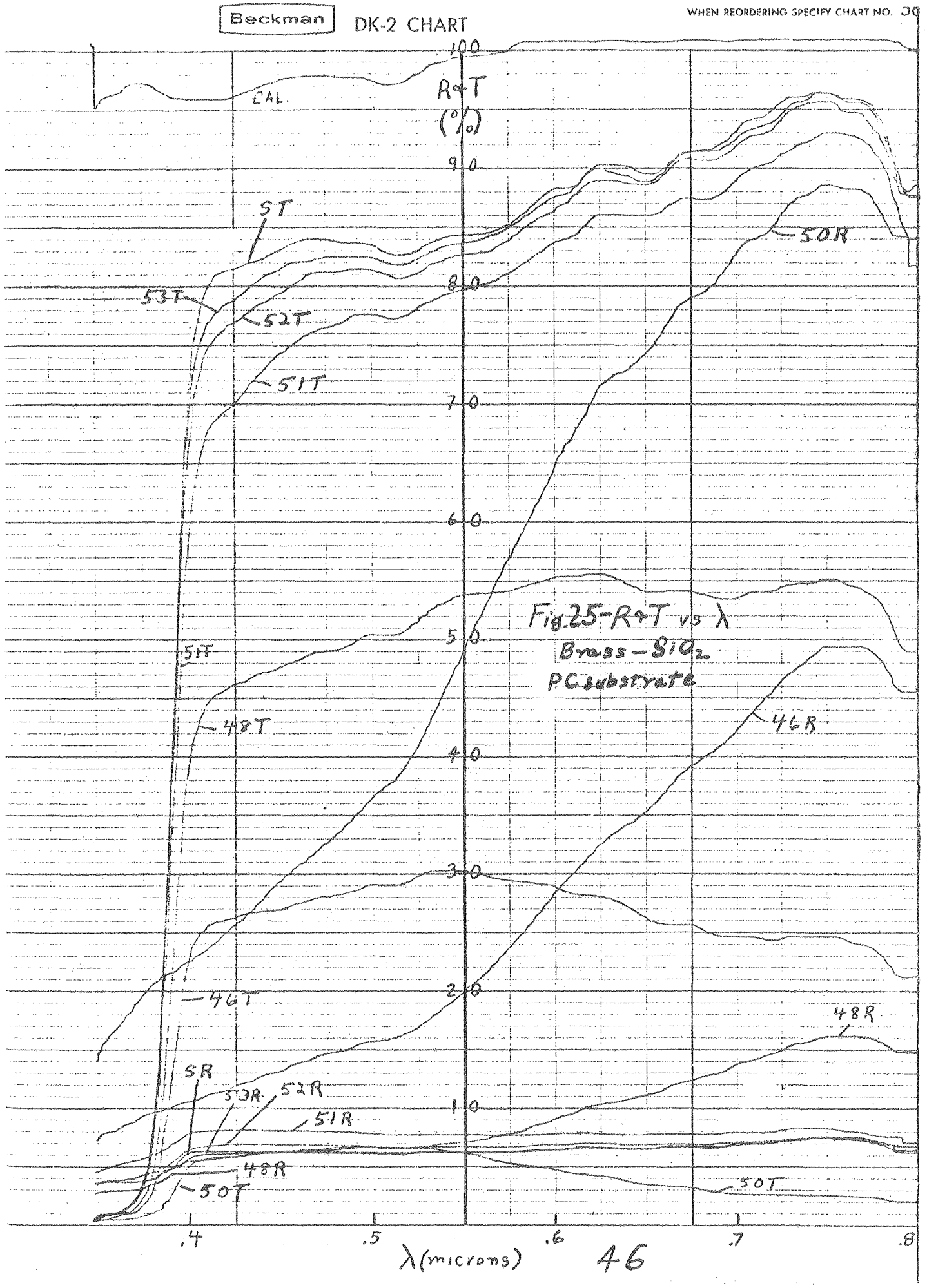
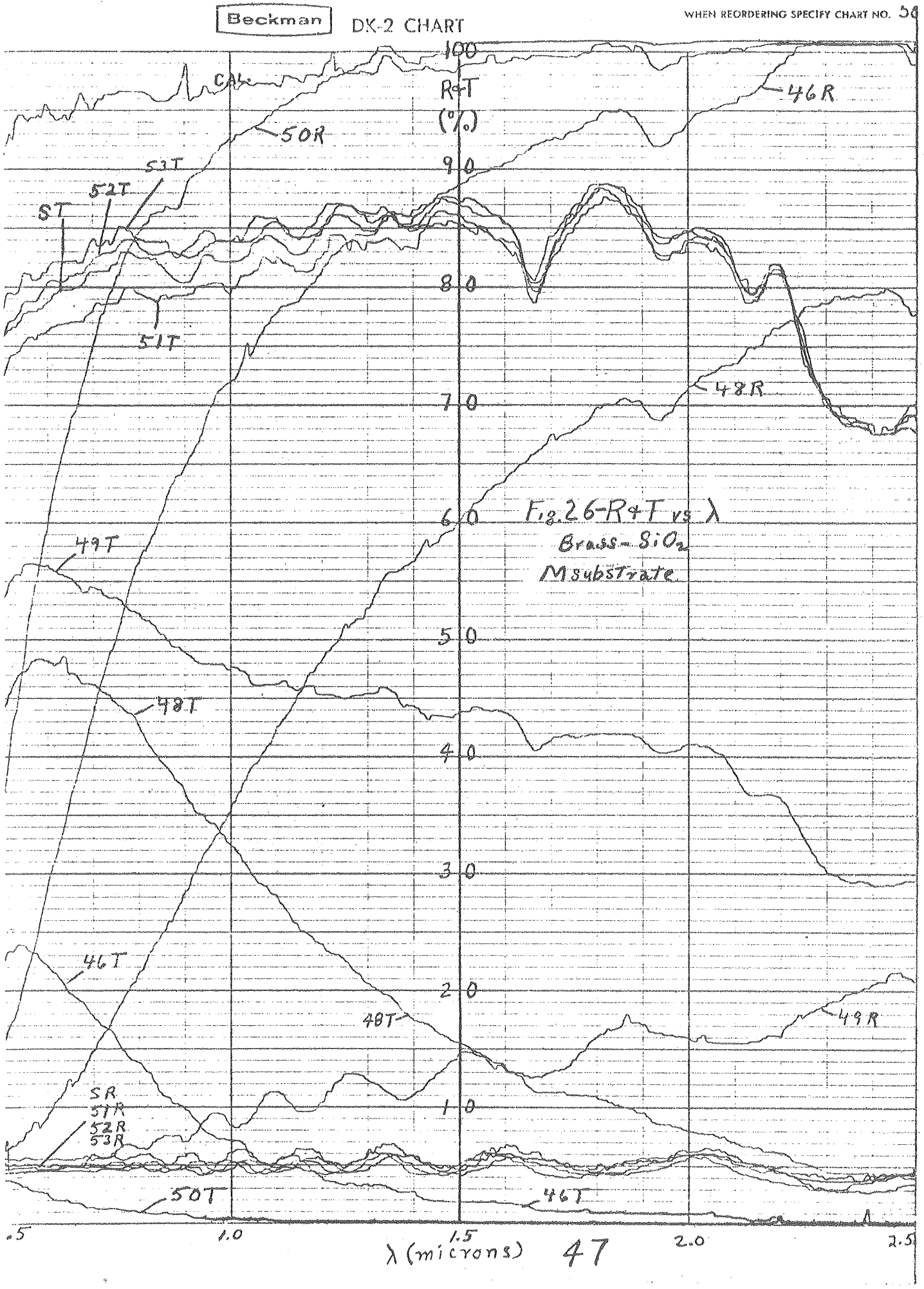
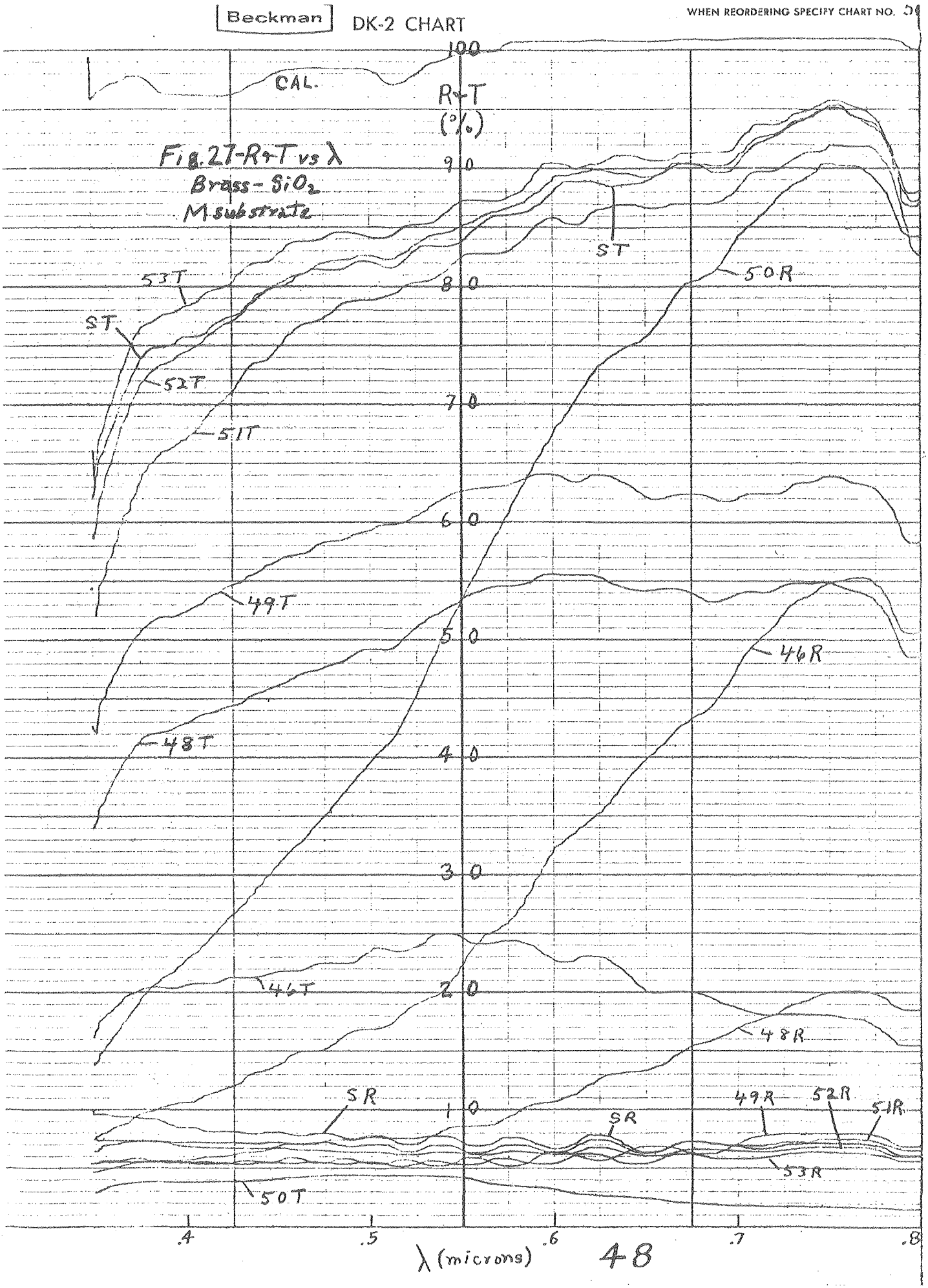


Fig.25-R&T vs λ
Brass-SiO_2
PC substrate


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 50
100
R+T
(%)
90
80
70
60
50
40
30
20
10
CAL
46R
50R
53T
52T
5T
51T
48R
49T
48T
46T
48T
49R
5R
51R
52R
53R
50T
46T
.5
1.0
1.5
2.0
2.5
λ (microns)
Fig. 26-R+T vs λ
Brass-SiO2
M substrate


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO.
CAL.
R+T
(%)
Fig. 27-R+T vs λ
Brass - SiO2
M substrate
100
90
80
70
60
50
40
30
20
10
53T
ST
52T
51T
49T
48T
46T
50T
ST
50R
46R
48R
SR
SR
49R
52R
51R
53R
.4
.5
.6
.7
.8
λ (microns)

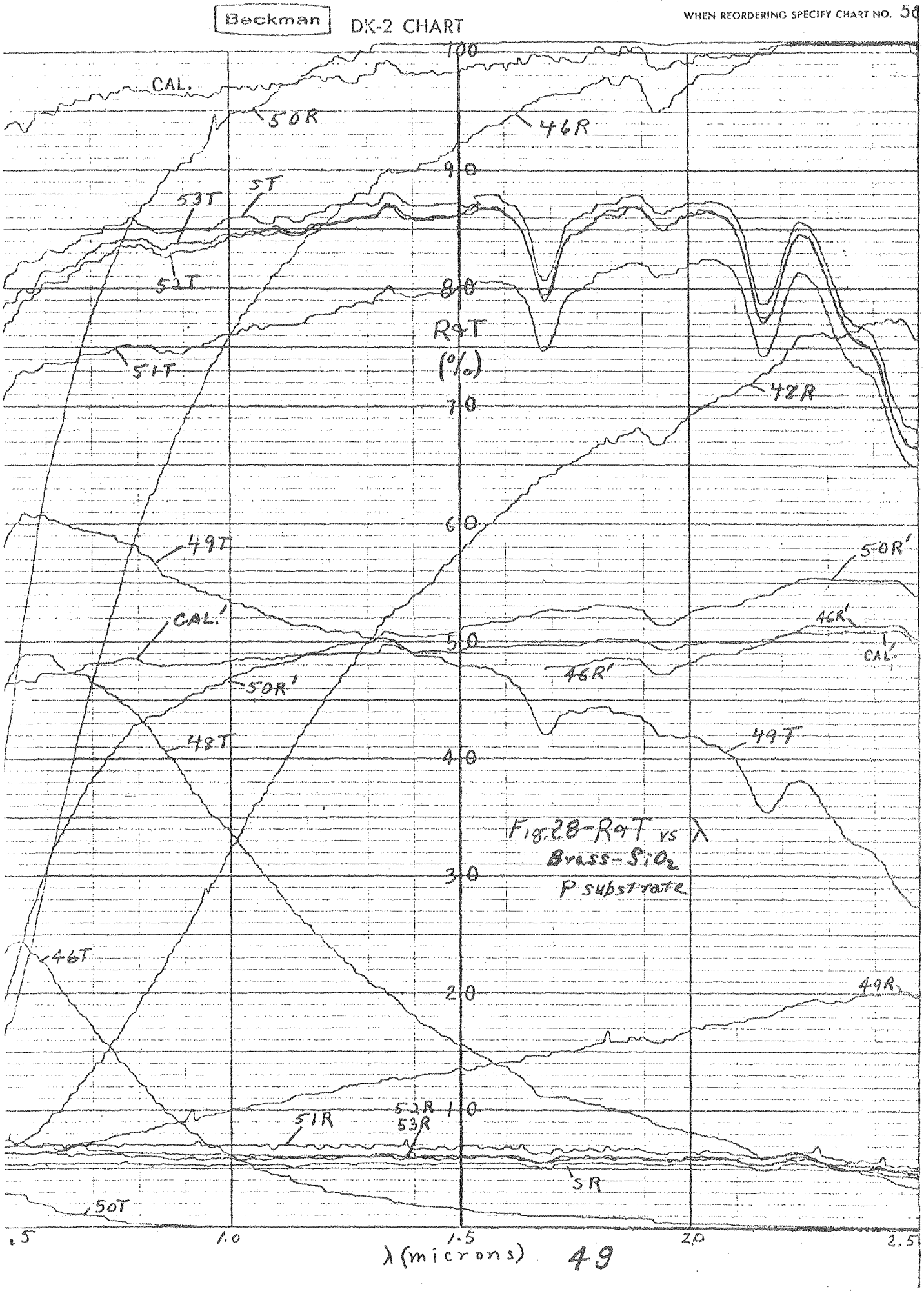
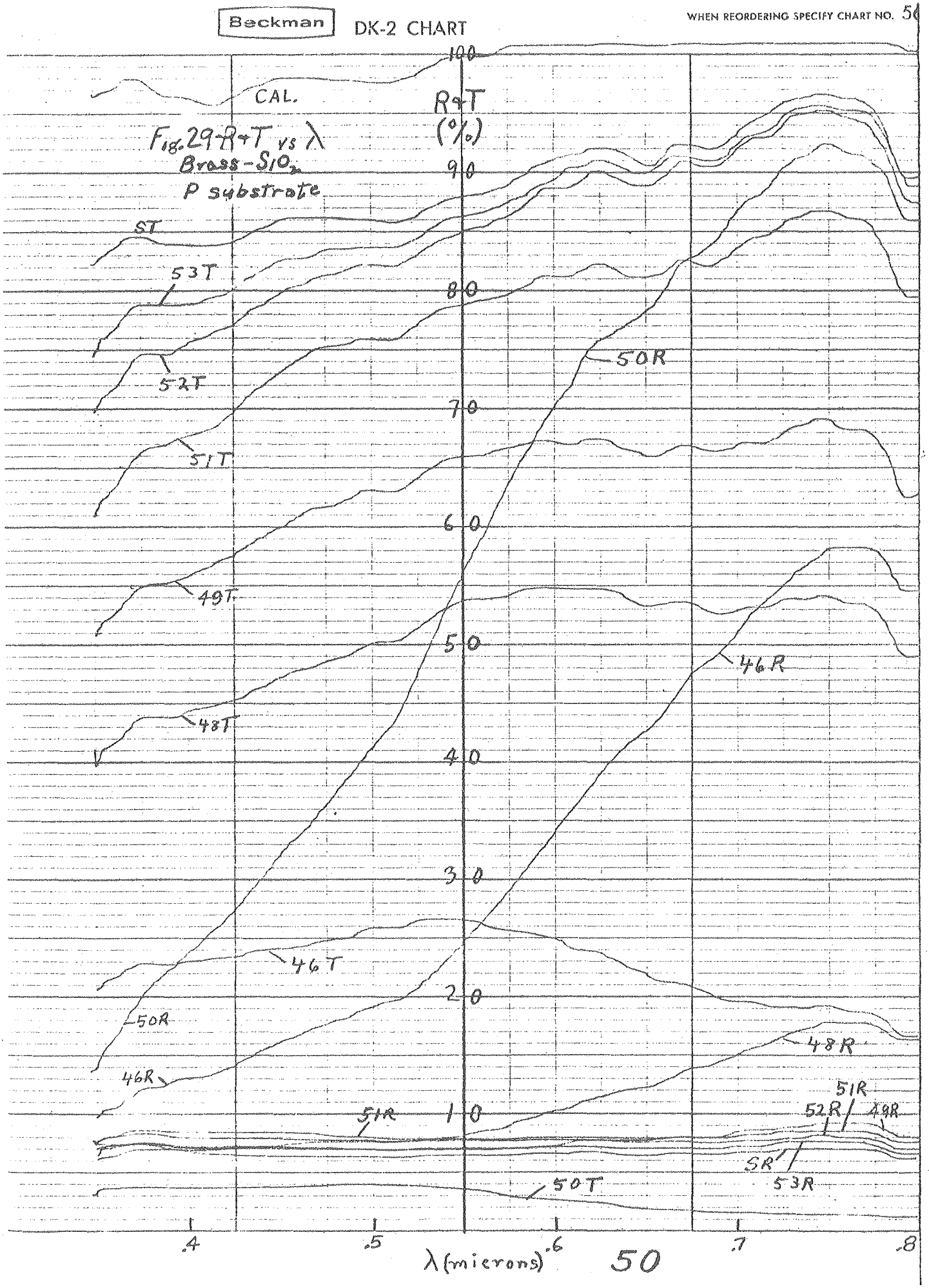
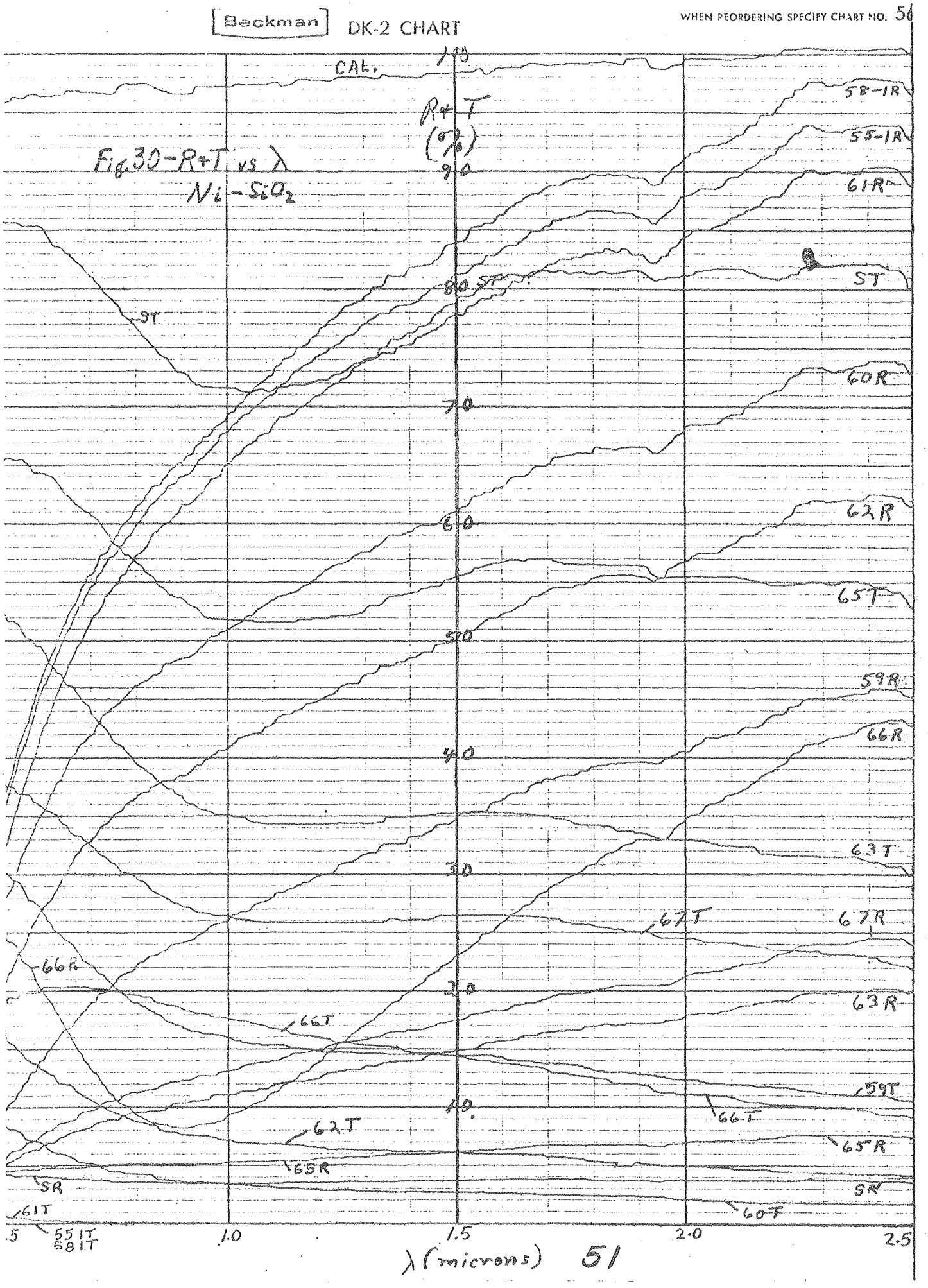


Fig. 28 - R+T vs λ
Brass - SiO_2
P substrate


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 56
Fig. 29 R+T vs λ
Brass-SiO2
P substrate
R+T
(%)
100
90
80
70
60
50
40
30
20
10
CAL.
ST
53T
52T
51T
49T
48T
46T
50R
46R
51R
50R
46R
48R
51R
52R
49R
SR
53R
50T
.4
.5
.6
.7
.8
λ (microns)


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 56
Fig. 30 - R+T vs λ
Ni - SiO2
R+T
(%)
CAL.
100
90
80
70
60
50
40
30
20
10
58-1R
55-1R
61R
5T
9T
60R
62R
65T
59R
66R
63T
67T
67R
66R
63R
66T
59T
66T
62T
65R
65R
5R
5R
61T
60T
.5
55 1T
58 1T
1.0
1.5
2.0
2.5
λ (microns)
51

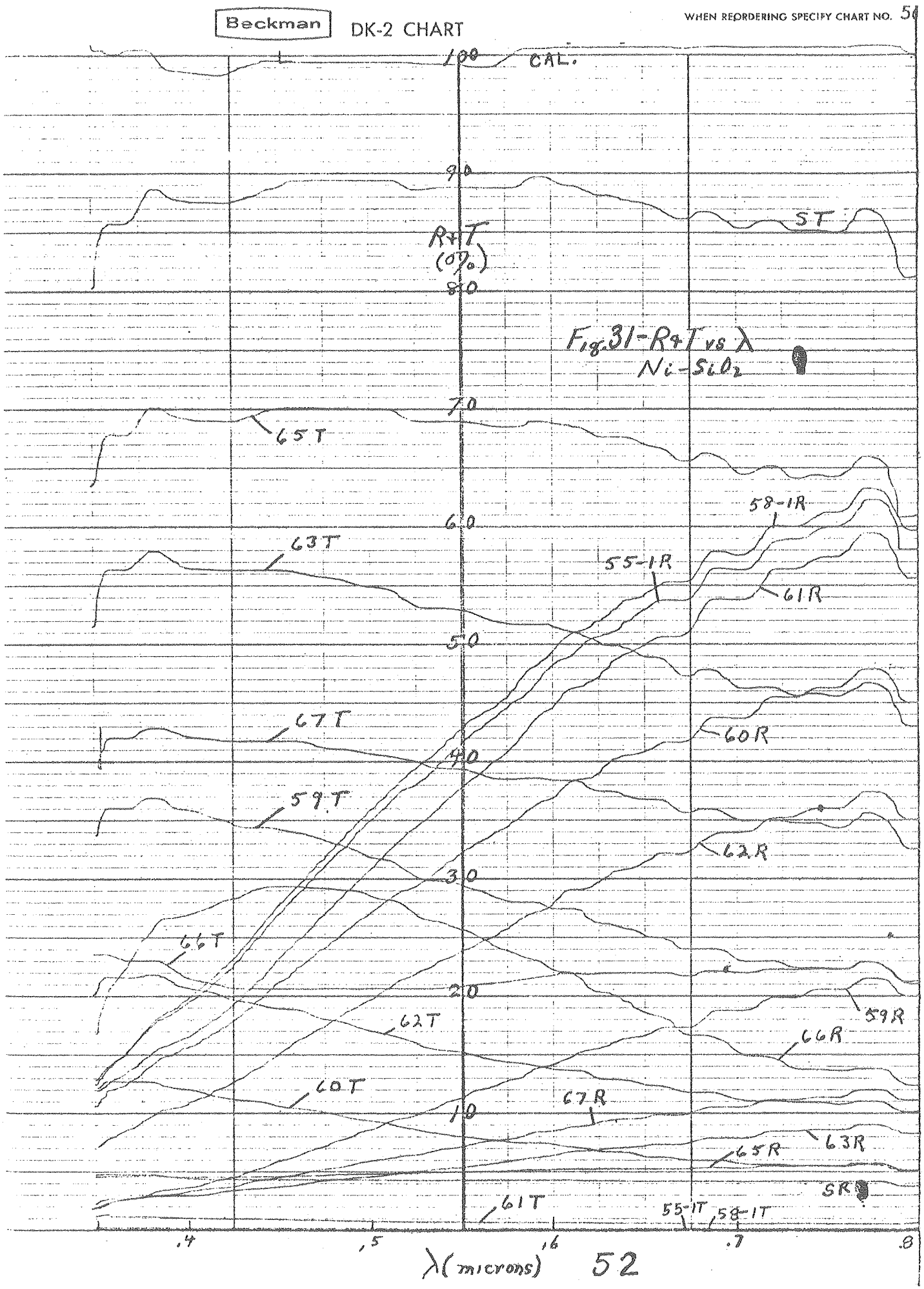


Fig. 31 - R+T vs λ
Ni-SiO2

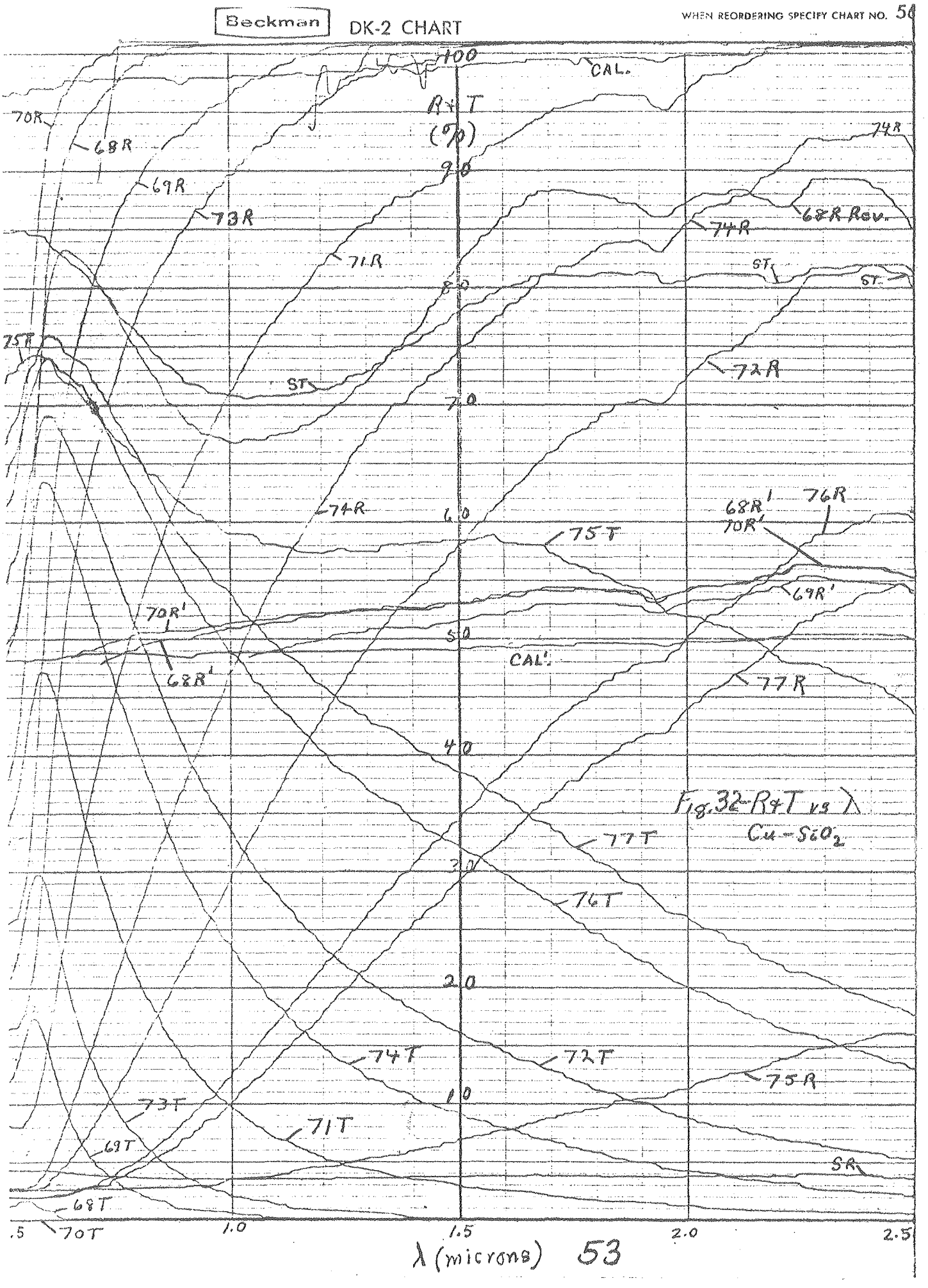


Fig. 32 - R+T vs λ
Cu-SiO2

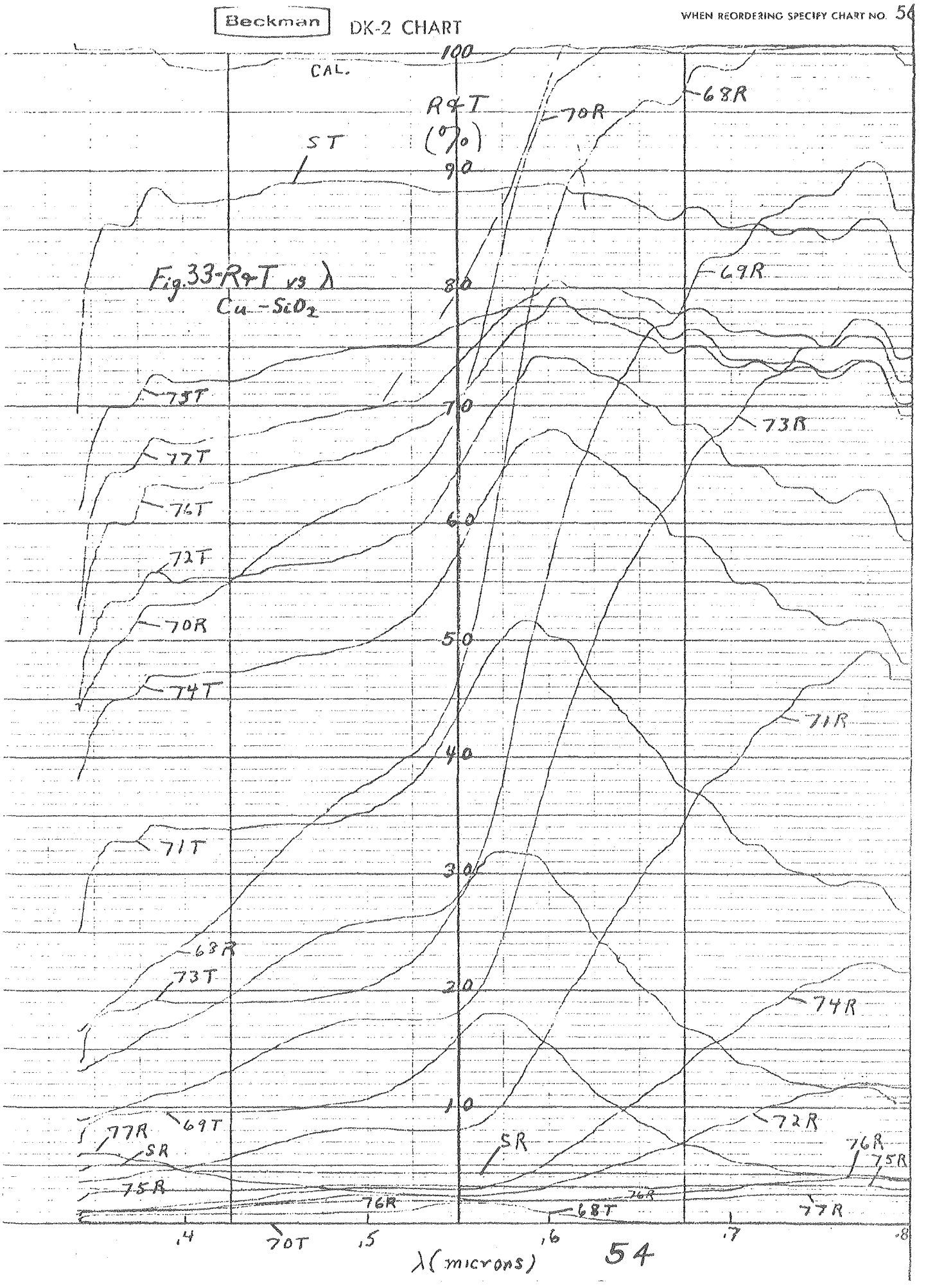


Fig. 33 - R&T vs λ
Cu - SiO_2

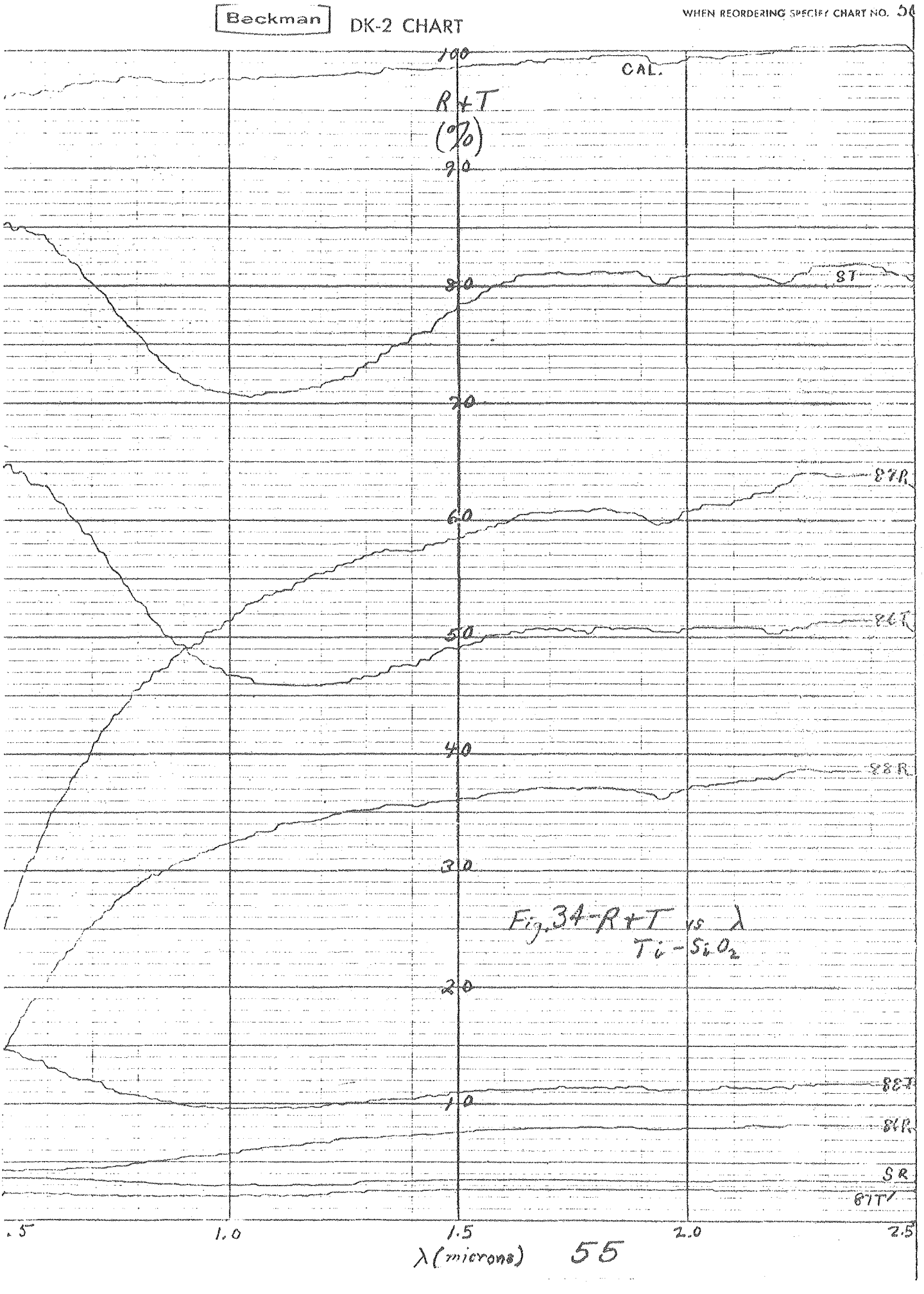


Fig. 34 - R+T vs λ
Ti - SiO_2

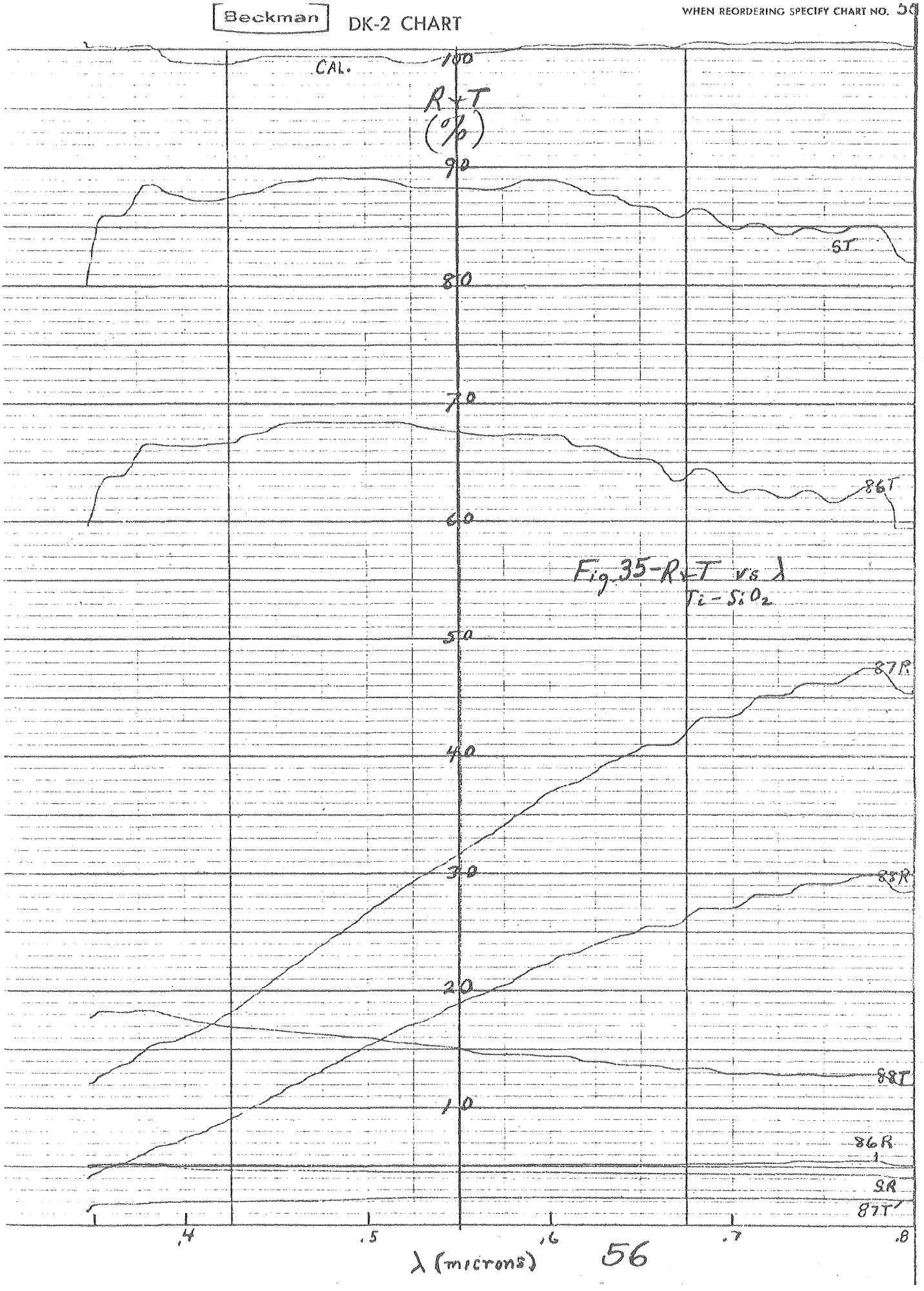


Fig. 35 - R+T vs λ
Ti - SiO_2

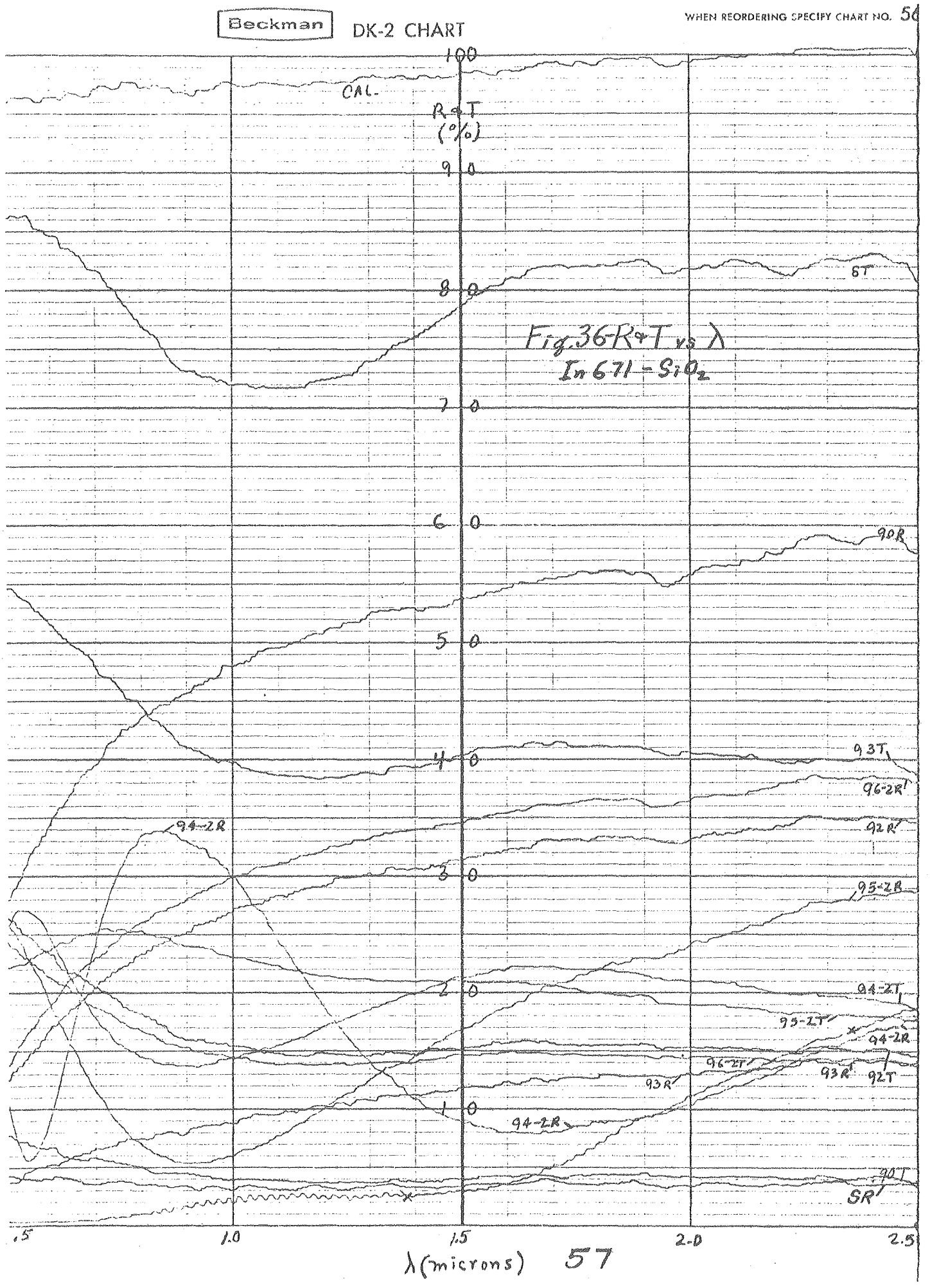
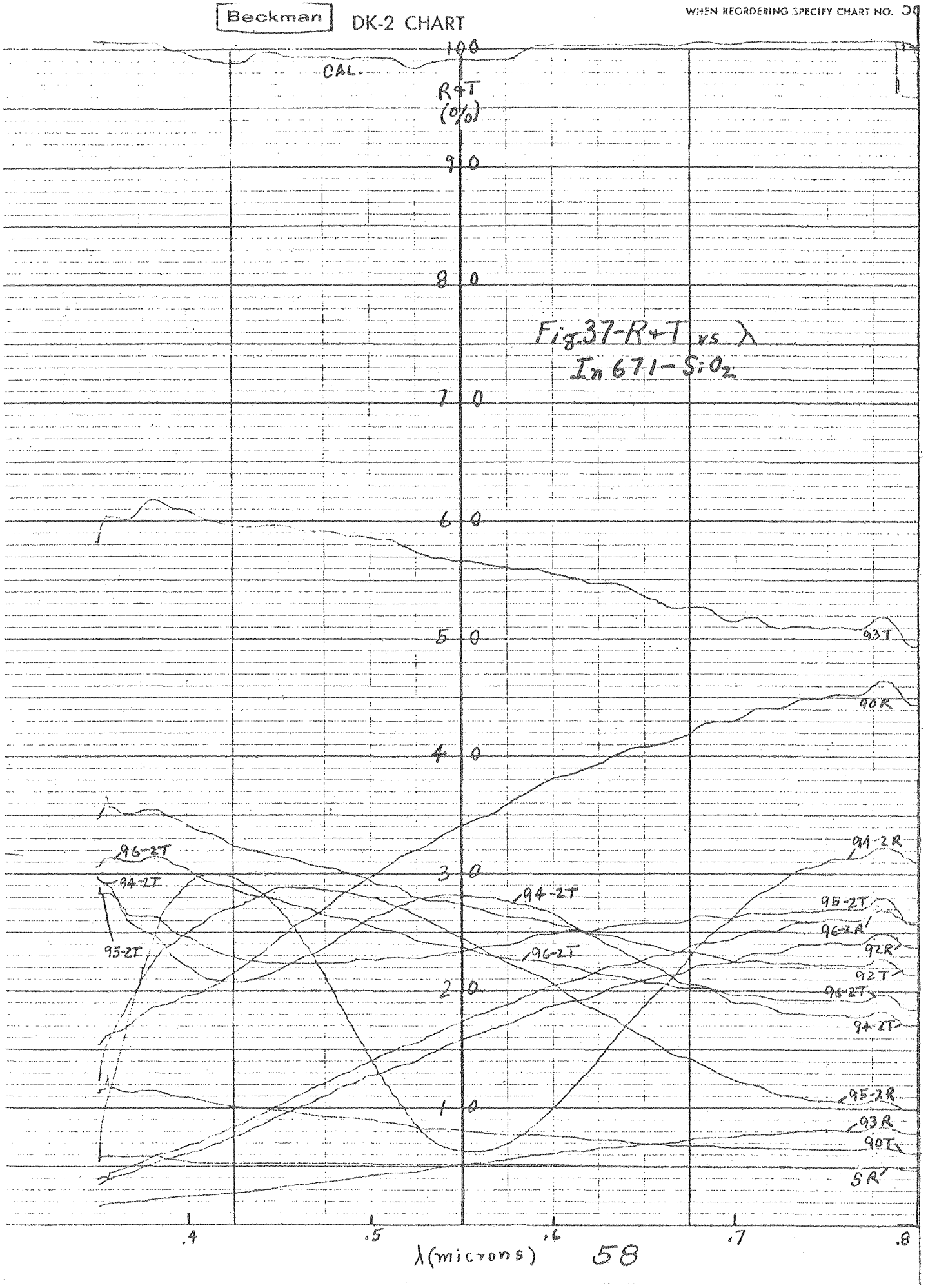


Fig. 36 R+T vs λ
In 671 - SiO_2


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 30
CAL.
R+T
(%)
100
90
80
70
60
50
40
30
20
10
Fig. 37-R+T vs λ
In 671-SiO2
93T
90R
96-2T
94-2T
95-2T
94-2T
96-2T
94-2R
95-2T
96-2R
92R
92T
96-2T
94-2T
95-2R
93R
90T
5R
.4
.5
.6
.7
.8
λ(microns)

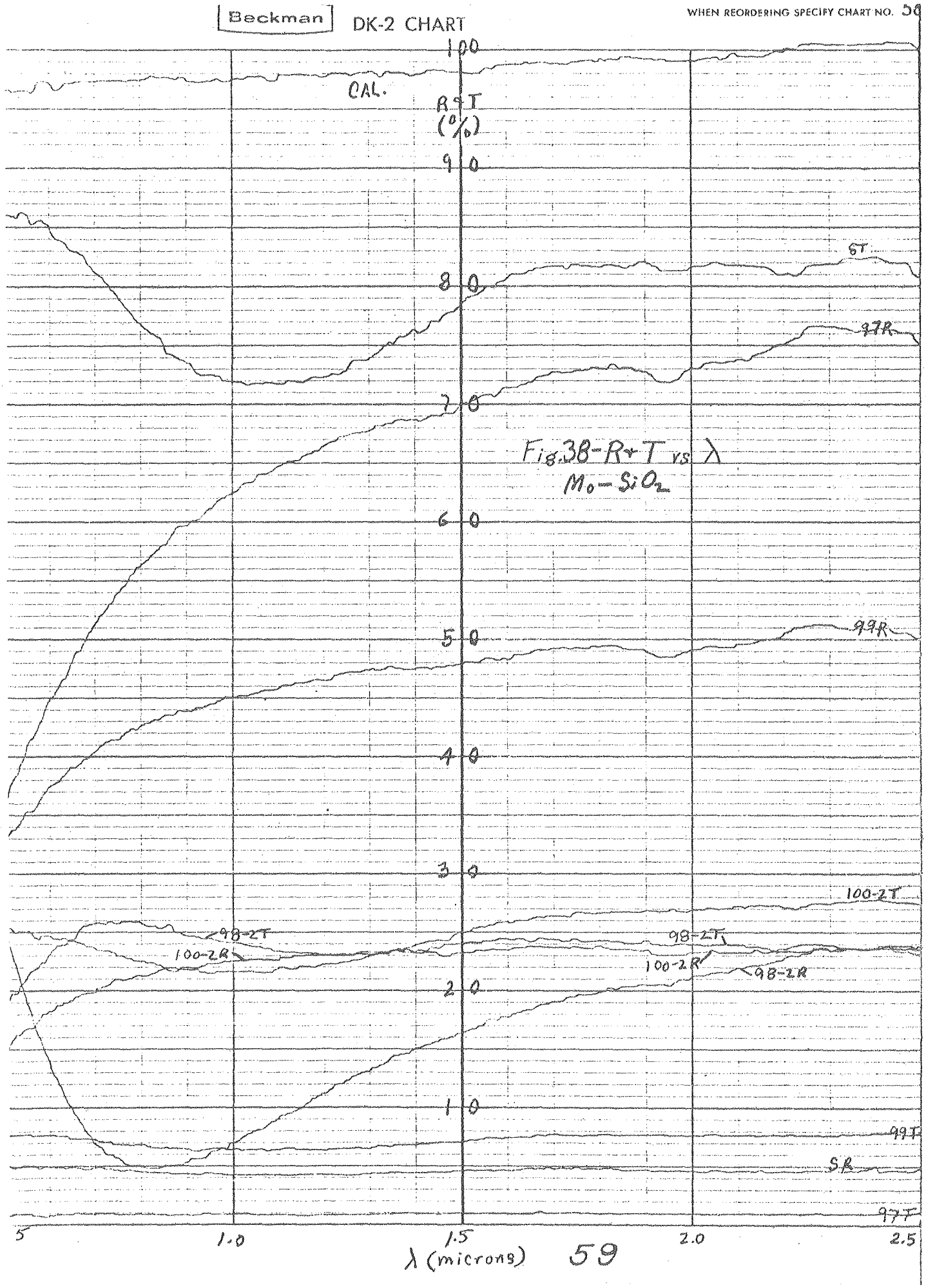


Fig. 38 - R+T vs λ
Mo - SiO_2

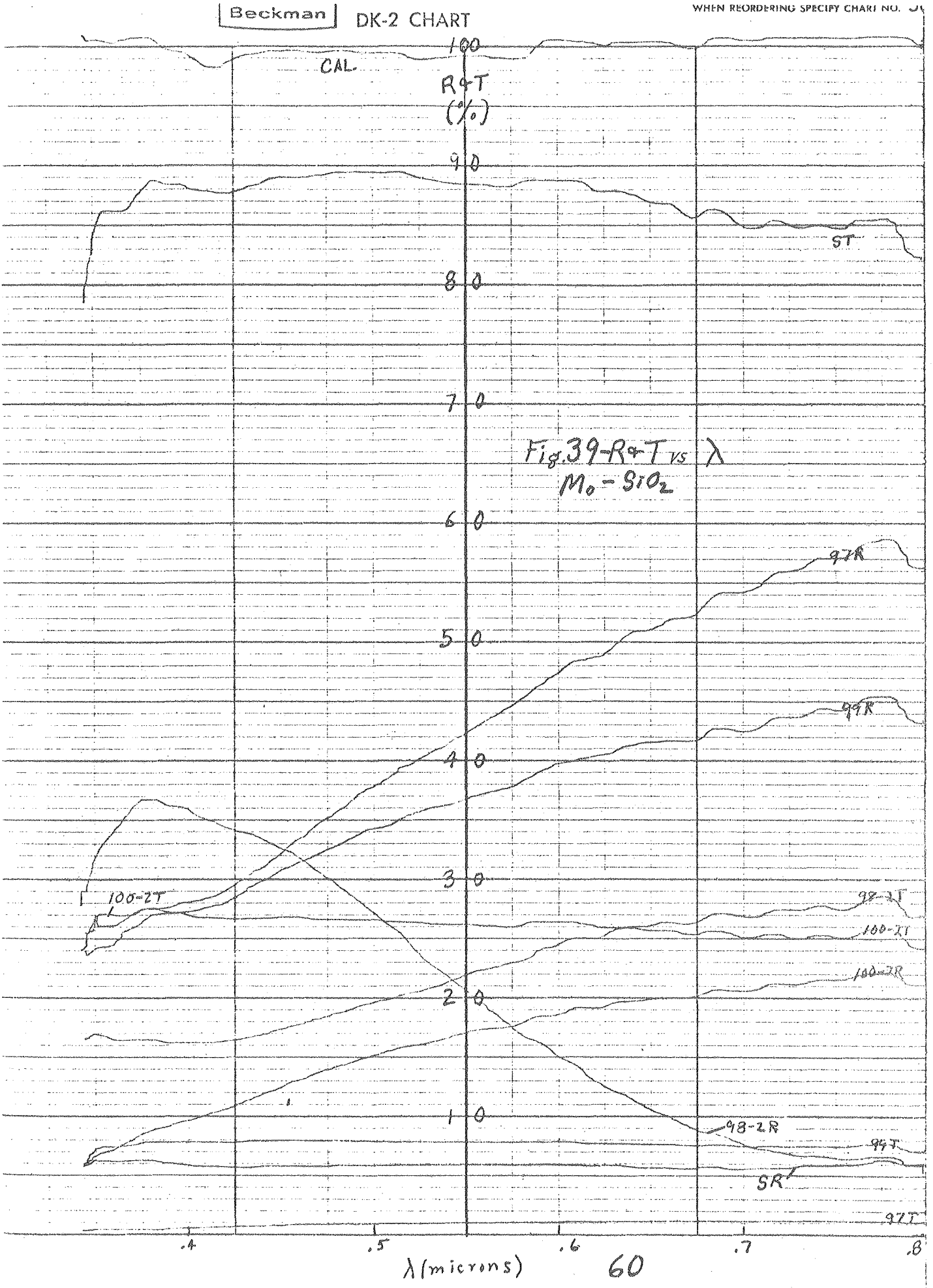
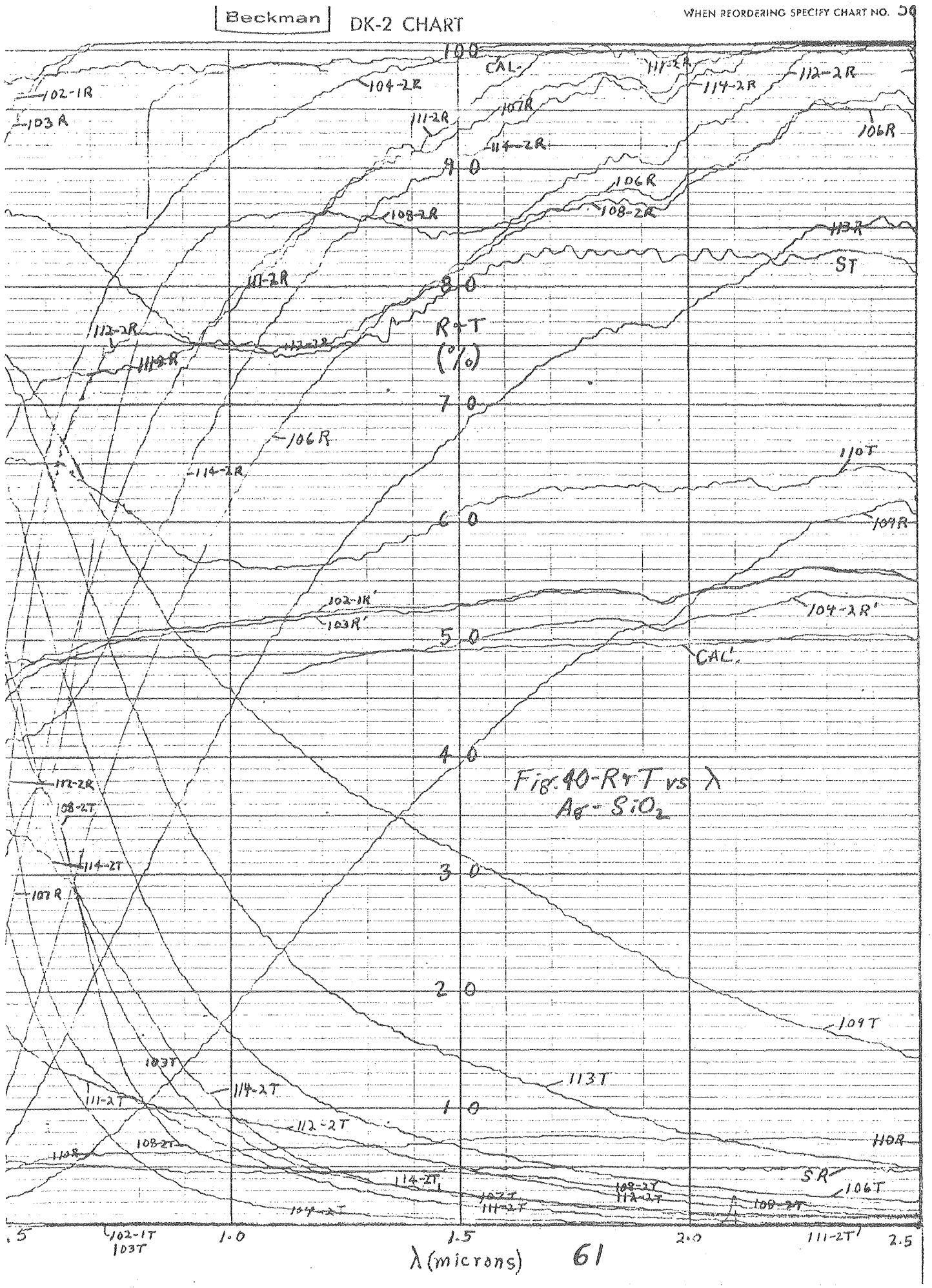
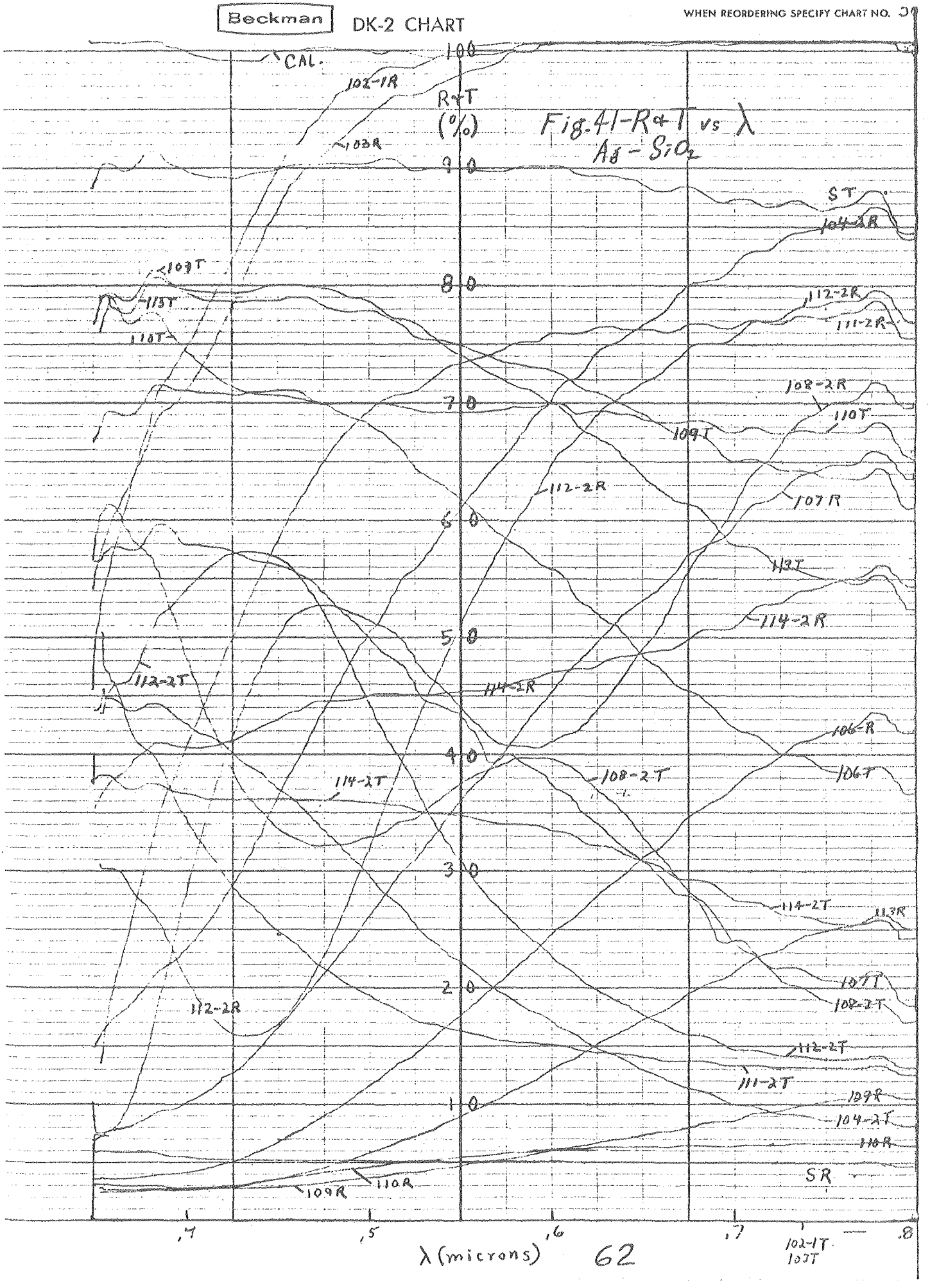


Fig. 39 - R+T vs λ
$M_o - SiO_2$


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO.
100
90
80
70
60
50
40
30
20
10
R+T
(%)
.5
1.0
1.5
2.0
2.5
λ (microns)
CAL.
CAL'.
ST
SR
102-1R
103R
104-2R
107R
111-2R
114-2R
112-2R
106R
108-2R
113R
110T
109R
102-1R'
103R'
104-2R'
109T
113T
110R
106T
108-2T
114-2T
107R
103T
111-2T
112-2T
102-1T
103T
111-2T'
Fig. 40-R+T vs λ
Ag-SiO2


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO.
Fig. 41-R&T vs λ
Ag - SiO2
R+T
(%)
100
90
80
70
60
50
40
30
20
10
CAL.
102-1R
103R
ST
104-2R
109T
113T
110T
112-2R
111-2R
108-2R
110T
109T
107R
112-2R
113T
114-2R
112-2T
114-2R
106-R
106T
114-2T
108-2T
114-2T
113R
107T
108-2T
112-2R
112-2T
111-2T
109R
104-2T
110R
SR
109R
110R
.4
.5
.6
.7
.8
λ (microns)
102-1T
103T

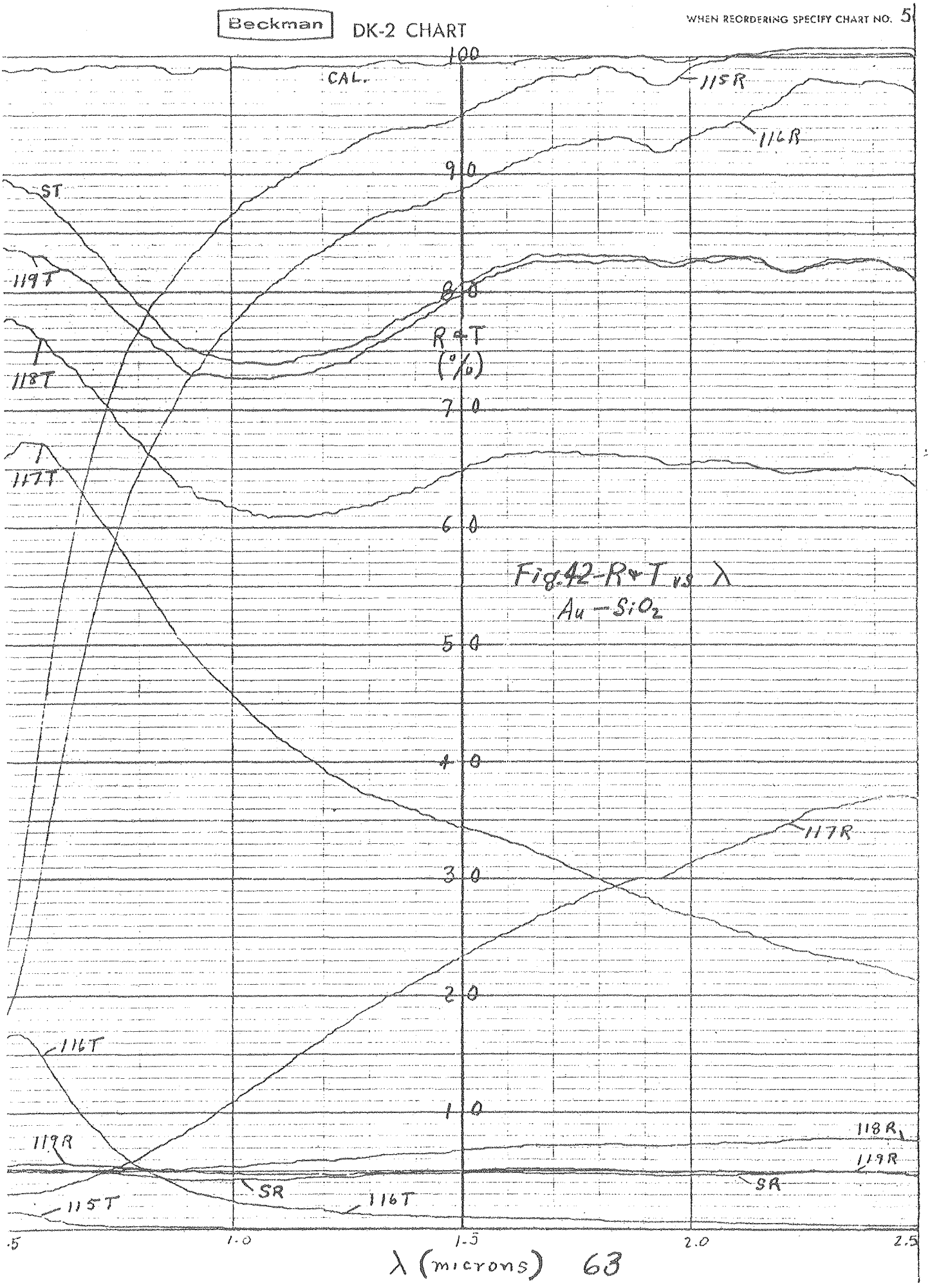


Fig. 42 - R & T vs λ
Au - SiO_2

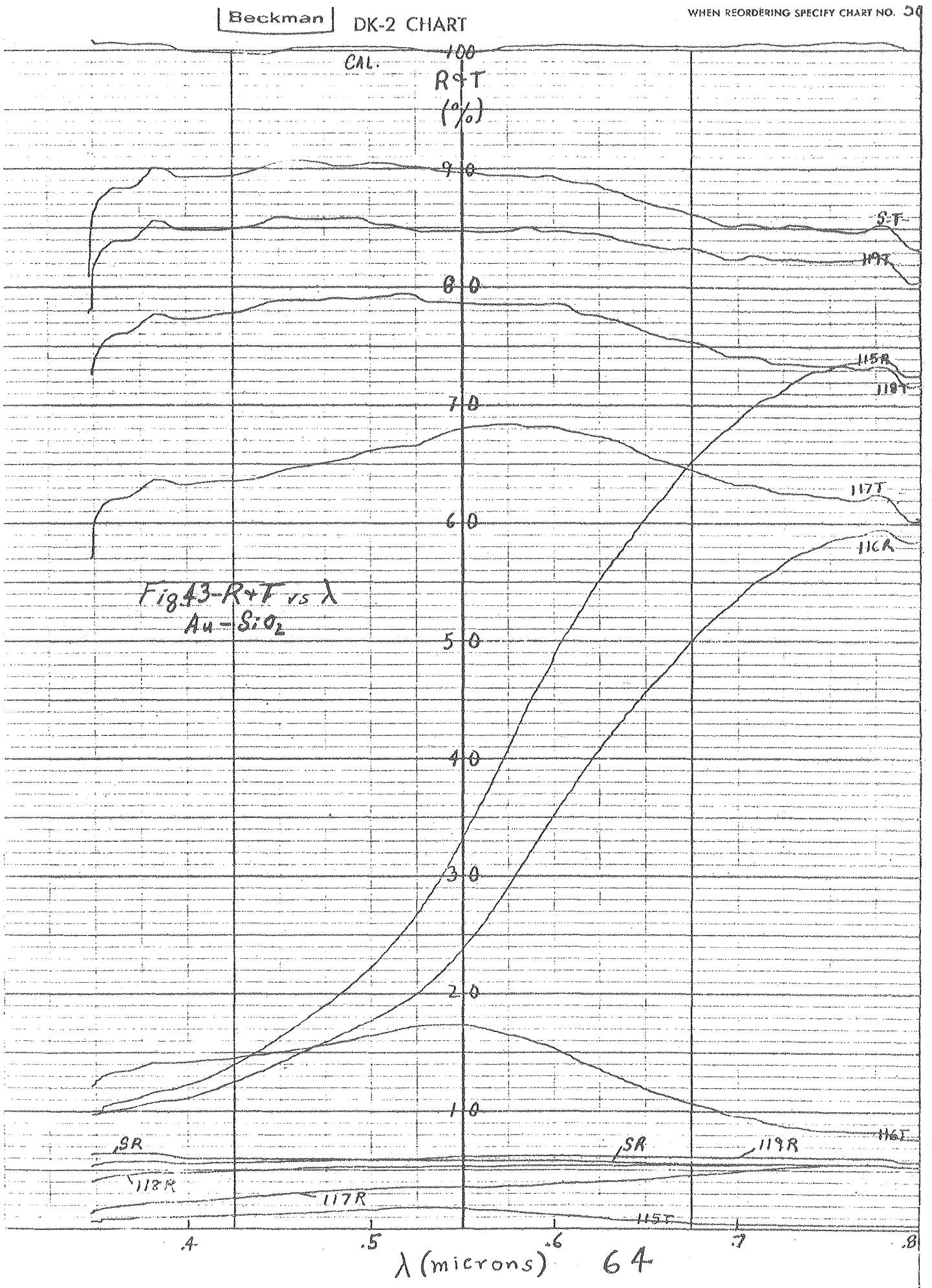


Fig 43-R+T vs λ
Au-SiO_2

Cal	≡	calibration curve(set for 100% at 2.5 μ or 100% at 0.8 μ)
S	≡	standard or uncoated substrate curve (one brand of glass was used up to sample 54 and another for the remainder)
R	≡	reflectance
T	≡	transmittance
Curve #	≡	sample #, e.g. 72 and S; if both reflectance and transmittance are on one set of curves, the curve number is suffixed accordingly, e.g. 72 R and SR; 72T and ST
G, PC, M, P	≡	glass, polycarbonate, mylar, polyester-as in Table 1
'	≡	double scale, i.e. 50 is 100% and 100 is 200%
*	≡	.35 - .8 μ range when both spectral ranges are on one set of curves

Calibration curves were run by setting 100% at 2.5 μ on the .5 -2.5 μ ranges and at .8 μ on the .35 - .8 μ range. In both cases, there was usually a jump upwards due to an unknown factor (probably mechanical lag) as the scan started. This was small and of no consequence on the higher scale. However on the lower scale it was sufficient to affect the region between .7 and .8 μ. The point at .8 μ, in fact, seems to be low in most cases and the lower scale curves are considered correct only between .35 and .7 μ. Matching of the two curves from .7 μ down was usually good to excellent.

The curves shown in Figure 3 were taken at the end of the program to determine the effect of coating one of the standards with a thin (< 500 Å) layer of SiO_2 without a reflecting layer;i.e. to look for absorption in the SiO_2. There was no apparent change in transmission and only a small change in the visible reflectance.

Figures 4 and 5 for Al-Al_2O_3 are included to show directly the comparative results on glass (G), polycarbonate (PC), mylar (M) and polyester(P). The M and P substrates, during deposition, were at comparable positions which received somewhat thicker reflecting layers than the G andPC substrates which were also at comparable positions. The M material was smoother than the P and gave slightly higher R values, both of them being

higher than those obtained for G and PC as demonstrated by samples 18. The PC material was slightly rough and generally gave lower R values than the G.

The R curves for samples 19 show an anomaly of the DK-2A instrument. The R values were much higher than expected for the Al thickness, an effect which occurred in many sets of curves. This was traced to a plunger used to hold the samples in place during measurement. For very thin reflecting layers, the radiation passing thorugh the sample was reflected from the cupped plunger as a diffuse component. Since the instrument integrates all reflected components, this diffuse component was collected. However, when the samples were held by moving the plunger to one side so it did not see the beam transmitted through the sample, the effect completly disappeared as shown by Figures 6, 8, 10, and 12. This component from the plunger is also evident from the detailed PC curves for sample 19 which show the R and T values peaking and dipping at the same λ. The IR absorption peaks for the plastics are, of course, evident in all of the related transmission curves.

After this point was cleared up, all prior curves were retaken as necessary and Figures 6-43 are correct as shown. Although there is a great deal of secondary information in these figures, only the factors important to the present project are discussed below. The reader is referred to Figures 1 and 2 and the accompanying discussion of office and residential window requirements (Section 1.2) for help in evaluating the significance of the data. Put briefly, for office windows one would like variable reflectivity from .4-.7 μ consistent with lighting etc. requirements and 100% reflectivity from .7- 20μ. For residential windows, the requirements are for lowest possible reflectivity from .4-2.5 μ and 100% reflectivity from 2.5-20 μ. The transition between these regions should be as sharp as possible, but for practical purposes does not have to be extremely sharp. The sharpness is more important for office windows than for residential windows since the transition region occurs near the peak of the AM2 spectrum for the former, but in the low power area for the latter.

Conclusions from Figures 6-43 may be anticipated by stating that

the best results for office window applications were obtained for the brass-SiO_2, Cu-SiO_2 and Ag-SiO_2 systems. From results on weathering samples where the brass-Al_2O_3 system was studied, it is known that results for brass-Al_2O_3, Cu-Al_2O_3 and Ag-Al_2O_3 systems would have been equally good. All of these systems are capable of excellent performance for office windows. Relative performance for residential windows is not as clear cut due to more difficult design considerations. However performance for many of the systems is considered more than adequate in view of the potentially inexpensive manufacturing process. Detailed discussion on completed window samples is given in Section 3.

Figures 6-13 show the results for the Al-Al_2O_3 system. (Note: In evaluating this and other systems, the reflectivity curves must be considered in conjunction with the visible transmission curves, bearing in mind that, for office windows, enough light must be transmitted for suitable viewing and/or lighting. Since these results are on transparent substrates, some allowance must also be made for additional absorption in colored glass substrates used for office windows). Basically the results for all substrates are similar. The transitions are not sharp enough to provide top performance for office windows and samples with sufficiently low visible and near IR reflectance for residential applications have 10 μ reflectance values below those of the better systems (cp. Cu-SiO_2, Figures 32, 33, and Table 1). Results for the Al-SiO_2 system (Figures 14-21) were quite similar resulting in the same relative deficiencies.

In contrast to the previous two systems, the brass-SiO_2 system (Figures 22-29) has sharp transitions in the thickness range for office windows (roughly 0.8 to 1 times thickness of sample 46) and was the system used in initial investigations which lead to the present program. (It should be borne in mind that the curves as shown are those obtained within the time limitations of the program, Intermediate values for more refined performance may be directly interpolated). Results for residential window thicknesses were similar to those of the Al systems, and in practice might be slightly inferior due to the sharper transition. The latter, which occurs in the 1-2 μ region, has the

effect of allowing somewhat less of the incident energy to penetrate into the interior. The lower slope of the Al system (for the same 10 μ reflectivity) in the spectral region before 2.5 μ is therefore preferred. However, as the spectral region (4-25 μ) corresponding to the radiation from internal sources is approached, the relatively higher values for the sharper transition curve (for the same 10 μ reflectivity) are preferred since this leads to greater retention of internal heat. The actual choice in practice will be a complicated design problem which must be conducted to optimize performance and economic factors. This is more obvious from the complete curves given in Section 3.2 for a few of the preferred samples. Again, results for the other substrates were comparable to the results on glass.

The Ni-SiO_2 system (Figures 30-31) had low slopes inconsistent with good office window performance. The surprisingly high reflectance at 10 μ (e.g. Sample 59; 74% at 10 μ vs. ave. 11% in visible), in view of the durability of Ni, indicates some potential for residential windows should the superior systems (Cu-SiO_2 and Ag-SiO_2) not be sufficiently durable.

The Cu-SiO_2 system (Figures 32-33) has superior performance for both types of window. With one of the sharpest transitions regions it can readily be manipulated to match almost any office window design characteristics (see e.g. Sample 71). In addition, the very low visible reflectance coupled with relatively high 10 μ reflectance (see e.g. Samples 72, 74, and 76) can yield good residential window characteristics. Because of absorption, it may be desirable in this system to give up some of the 10 μ reflectance in order to get better visible transmittance (cp. Samples 72 and 76). More discussion is given on this system in Sections 3.1 and 3.2 since it was used for the final office window samples and some of the residential window samples.

Although only a few samples were made for the Ti-SiO_2 system, it was clear that it was of no potential use for the present application. Basically, the transmittance is too low for any reasonable degree of reflectance (e.g. Sample 87). The Inconel 671-SiO_2 and Mo-SiO_2 systems which were included primarily for their anticipated stability, suffer from the same deficiency.

General performance for the Ag-SiO_2 system was very similar to the Cu-SiO_2 system. The transition slopes are slightly less steep and the 10 μ reflectivities are somewhat smaller for the same general parameters in the .35-2.5 μ region. Overall, the Cu-SiO_2 system has the edge for both office and residential windows in terms of reflectivity characteristics. For residential windows, however, the Ag-SiO_2 system has a less noticeable coloring, being, for samples of interest, only a very slight neutral gray which is readily apparent only by direct comparision with clear glass. In contrast, the Cu-SiO_2 samples at residential window levels have a pleasant slight brown coppery cast. In both systems the coloring level is sufficiently low that it would probably not be objectionable to the user and might even be preferred for many applications.

The final system studied was Au alloy-SiO_2 where the Au alloy was 42% Au, 38% Cu, and 20% Ag and Zn. This combination also suffered from too much absorption for a given reflectivity so that performance was always considerably poorer than for the good systems. The rationale for using a Au alloy was the possibility of high stability. In view of the already available Cu-SiO_2 and Ag-SiO_2 results, the presence of the Cu and Ag in the alloy was not expected to adversely affect its relative performance. However, the combination did not work as well as the pieces, apparently due to higher absorption in the gold component. Since the Au is expensive, the other systems with superior performance look relatively attractive. Should other Au alloys become available from other KCI programs, they may be investigated in future programs. The prognosis from present results, however, did not warrant the purchase of additional targets in the present program.

Further interpretation and use of the data included in this section is presented in Section 3.

2.3 Growth Mechanism Study

Investigations and developments on other KCI ion beam sputtering programs had previously indicated that, due to their energetic nature, the IBS deposited atoms or molecules penetrate into the substrate. The net

effects of this penetration are twofold. First, the deposited atoms do not migrate over the surface and agglomerate but, rather, remain where they strike in a distribution determined by the distribution of the depositing material. Second, the energetic nature creates a bombarding or packing effect which densifies the deposited material.

Early results from the present program in general agreed with this premise. Reflection curves, both in the IR and visible, were seen to progress smoothly from long wavelengths to shorter wavelengths, even with the thinnest reflecting layers studied. The latter were so thin in some cases as to be barely detectable in the visible region by the naked eye, but still had appreciable 10 μ reflectivity (e. g. sample 49-10 μ reflectivity of 65%). One possible explanation for the behavior is that the reflectivity is strictly a function of the probability of the incident light wave, of a given wavelength, interacting with an atom of the reflecting layer material. This probability would obviously be higher for the longer wavelength and would get proportionately higher at shorter and shorter wavelengths as the atoms got closer and closer together, i. e. as more were deposited. If, however, the atoms did not penetrate but agglomerated as in other deposition techniques, the function would not be smooth but might have discontinuities at bridging thicknesses etc. Such discontinuities would be more likely to occur with very thin layers corresponding to rising far IR reflectance while the visible reflectance is still low.

In order to define the condition of the reflecting film at every stage of deposition, a series of 23 samples consisting of 2" x 2" window glass coated with varying thicknesses of brass (see Table 2) was prepared.

It is unfortunate that this experiment was initiated before the apparent anomaly in the brass thicknesses (see Section 2. 2) was discovered. As discussed previously, the brass thicknesses calculated from a measured calibration standard appear to be a factor of 3 too high when compared with other materials and optical characteristics. The relative thicknesses are, however, quite consistent.

Most samples were deposited as soon as the vacuum level was

TABLE 2

SAMPLE CHARACTERISTICS - GROWTH MECHANISM STUDY

(Brass Reflecting Layer)

Sample #	Brass Thickness(Å)	Reflectivity(%) at 10 μ(SiO_2 overcoat)
54-1	1880	96.8
2	940	96.3
3	470	94.5
4	236	90.1
5	224	89.2
6	211	87.6
7	201	87.3
8	189	86.0
9	177	83.6
10	165	82.5
11	154	80.9
12	142	76.2
13	130	75.6
14	118	69.3
15	106	68.5
16	95	66.9
17	83	39.7
18	71	27.9
19	59	25.8
20	47	25.5
21	24	25.5
22	35	25.5
23	106	73.0

adequate. However sample 54-5 (hereafter labelled 5 etc.) was left in the vacuum overnight before deposition. Because it produced an anomalous reflectivity curve, sample 16 was left in vacuum an additional 1-1/2 hours before deposition to see if the effect could be repeated. Sample 23 was then done at the same brass level as 15 but was left in the vacuum overnight before deposition.

No overcoating was used, and the samples, after immediate measurement of the 0.5 - 2.5 μ R and T values, were stored in vacuum (10^{-6} torr) until just before they were taken to MIT for SEM evaluation. Total air exposure before SEM was less than 3 hours.

Figure 44 shows the R and T curves for the various samples taken on the Beckman DK-2A. Basically the curves progress in reflectance and associated transmission from the thickest brass sample (# 1) to the thinnest (# 21). Number 5 was slightly anomalous, having a reflectance very close to that of # 4 rather than being less. As discussed above, # 16 which was also left in vacuum for an additional period before deposition, had reflectance values greater than rather than less than # 15. Finally, # 23 which was a repeat of # 15 but having long vacuum exposure, had greater reflectance than 15 or 16. Clearly the reflectance of the deposited layer is a function of the vacuum exposure time before deposition, as well as of the brass thickness and beam conditions. Since the SEM studies (see below) indicate an extreme similarity of the physical appearance of the deposited material at all thicknesses, the difference in reflectivity as a function of vacuum exposure is probably due to the degree of oxidation during deposition; i.e. to the residual oxygen or water vapor in the system. The latter is a function of pumping time before deposition. In most cases this was approximately 1/2 hour whereas for the higher reflectance cases (5, 16, 23) it was 2 hours to 15 hours.

Although the reflectance curves with the shorter pump down times are quite acceptable, in the long run control of the reflectance characteristics would appear to depend on vacuum conditions. However, for a production process, the system would not be opened, exposed to air and then repumped for each sample as in the present case. The required vacuum conditions for control and reproducibility are readily achievable in a production machine.

Beckman DK-2 CHART

WHEN REORDERING SPECIFY CHART NO. 56

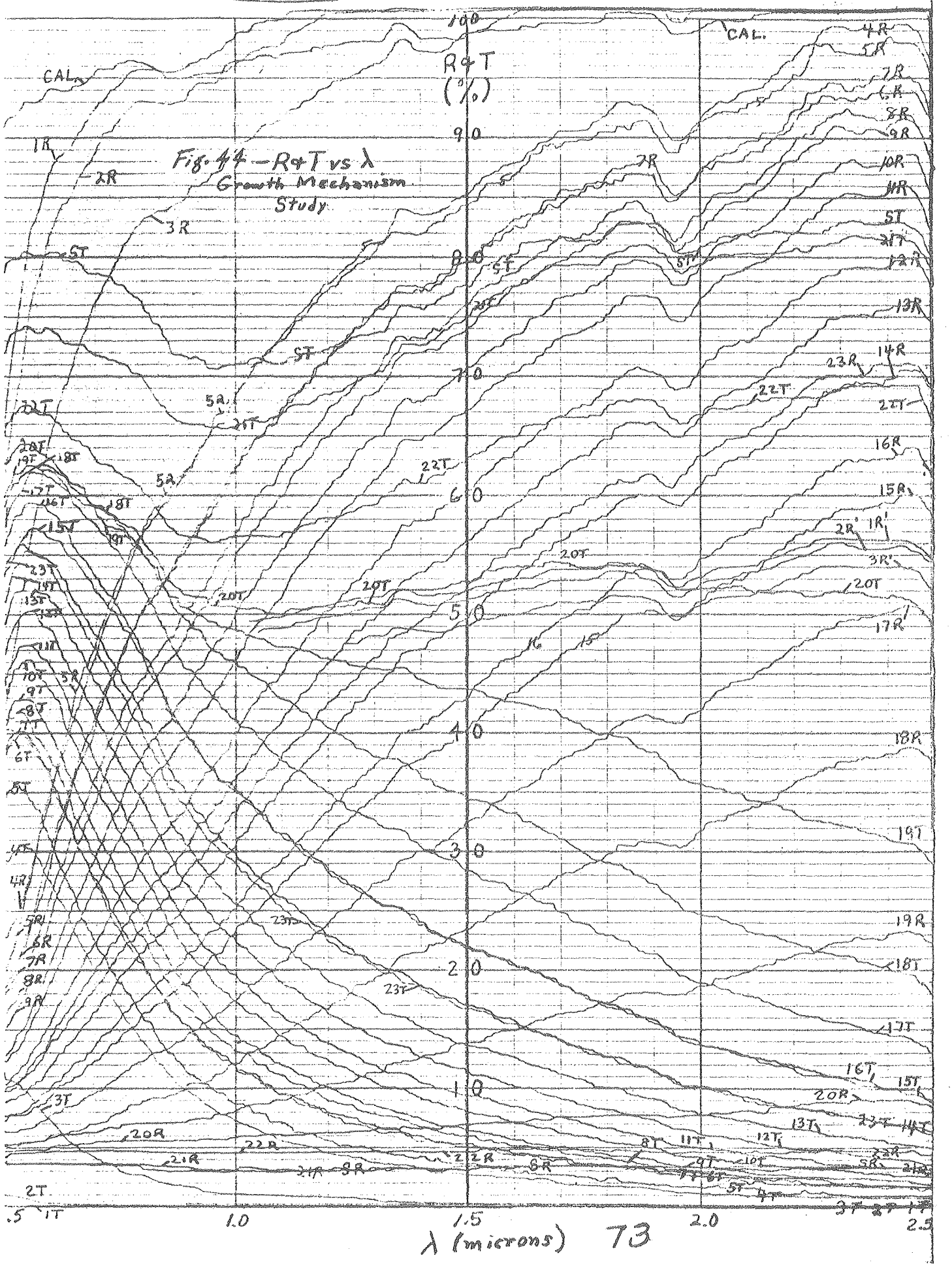


Figure 45 (2 pages) shows SEM photographs of some of the samples including W-54-1, 2, 19, and 21. Figure 45 (a) shows # 1 at magnification of 16K. At the middle bottom there is a slight imperfection in the glass substrate which was used for initial focussing. Essentially the deposit is featureless. Figure 45(b) shows # 2 which is half as thick as 1 and which was photographed over a featureless substrate region. There is no evidence of any structural features. (The lines and graininess are artifacts of the Xerox reproduction. The actual photographs in all cases show no grain size and a very smooth surface.) Sample W-54-19 (1/32 thickness of # 1 and quite transparent in visible is also absolutely featureless with no evidence of agglomeration, large nucleus formation, channeling etc. The large white spot (upper left) and the small spot (upper right) are areas where dust particles on the surface prevented deposition and they are therefore pinhole areas down to the glass substrate. In spite of its thinness, the presence and uniform dispersion of the deposit are obvious. Finally, #21 which is the thinnest sample (2/5 of #19) and which has a reflectance essentially indistinguishable from that of the uncoated glass is also featureless and absolutely uniform. The presence of the film on 21 is evident both from the photograph and from the surface conduction of the deposited film which is necessary in order to take the SEM picture (Because or surface charging, an insulator simply causes beam repulsion and blow-up).

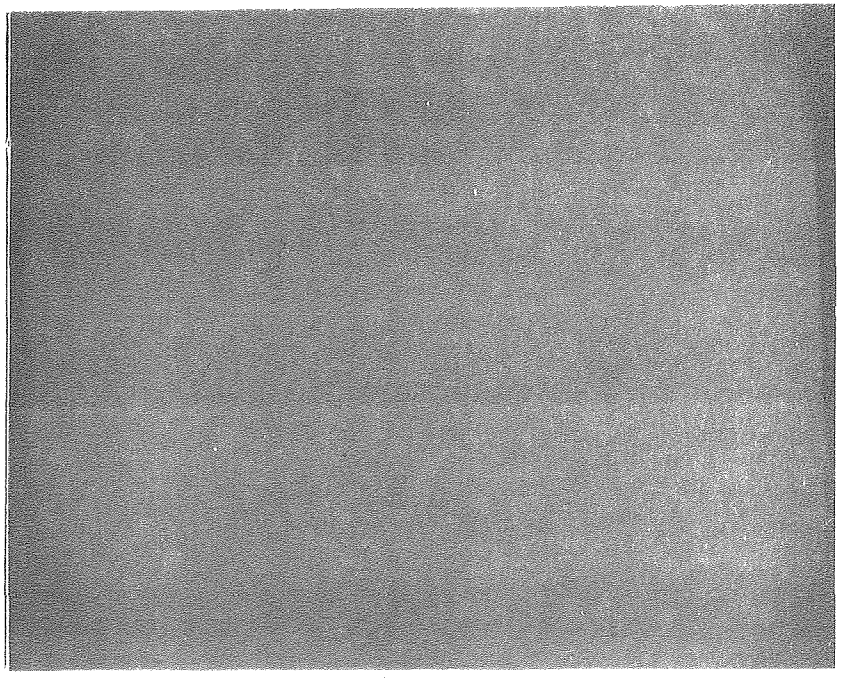
The brass layer on # 21 was calculated to be 24Å based on the calibration standard but is actually believed to be less than 10Å thick. The thickness of the layer however is clearly well below that at which large nuclei are formed and join to raise open and closed areas in films deposited by more conventional methods in which surface mobility of the deposited material is the prime factor. In the present case the penetration feature of the depositing material prevents agglomeration and therefore results in absolutely uniform and densified deposits even in very thin layers.

It is interesting to note that # 21 which has a reflectance essentially the same as the uncoated sample, nevertheless has appreciable absorption in the deposited film as shown by the transmission curves (Figure 44). This would seem to imply that the transition point to high IR reflectance may be associated with a transition from a mixture (due to penetration) of brass and

16K X

1 μ

Figure 45(a) Sample W-54-1; SEM

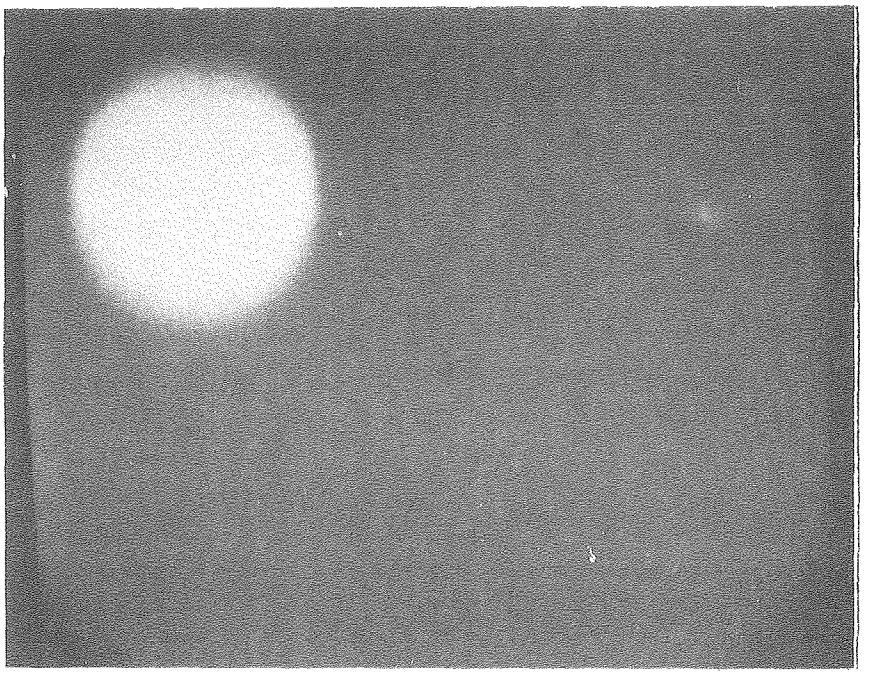


16K X

1 μ

Figure 45(b) - Sample W-54-2; SEM

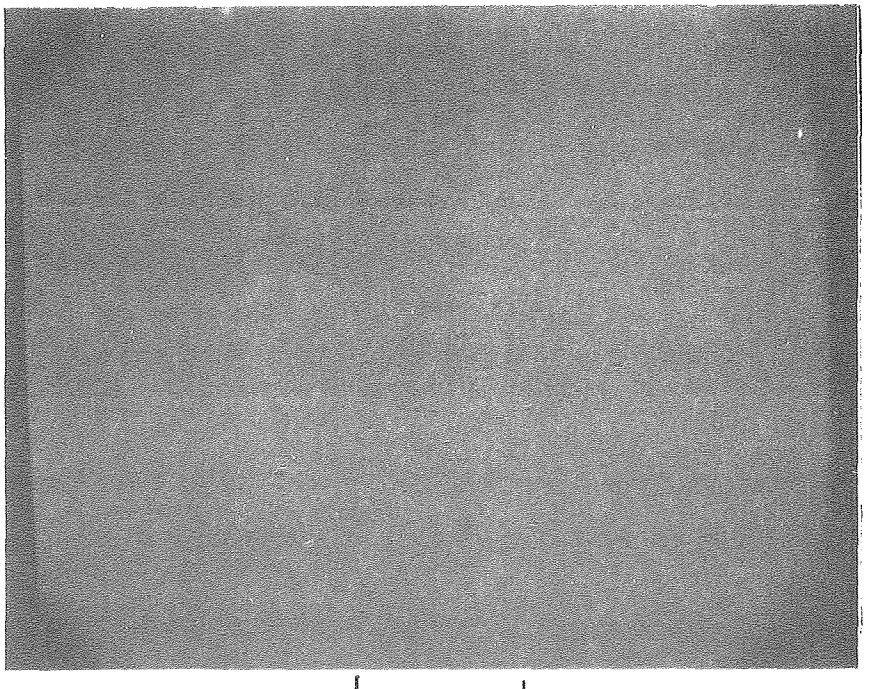
XBB 785-6195



45K X

0.5 μ

Figure 45(c) - Sample W-54-19; SEM



45K X

0.5 μ

Figure 45 (d) - Sample W-54-21; SEM

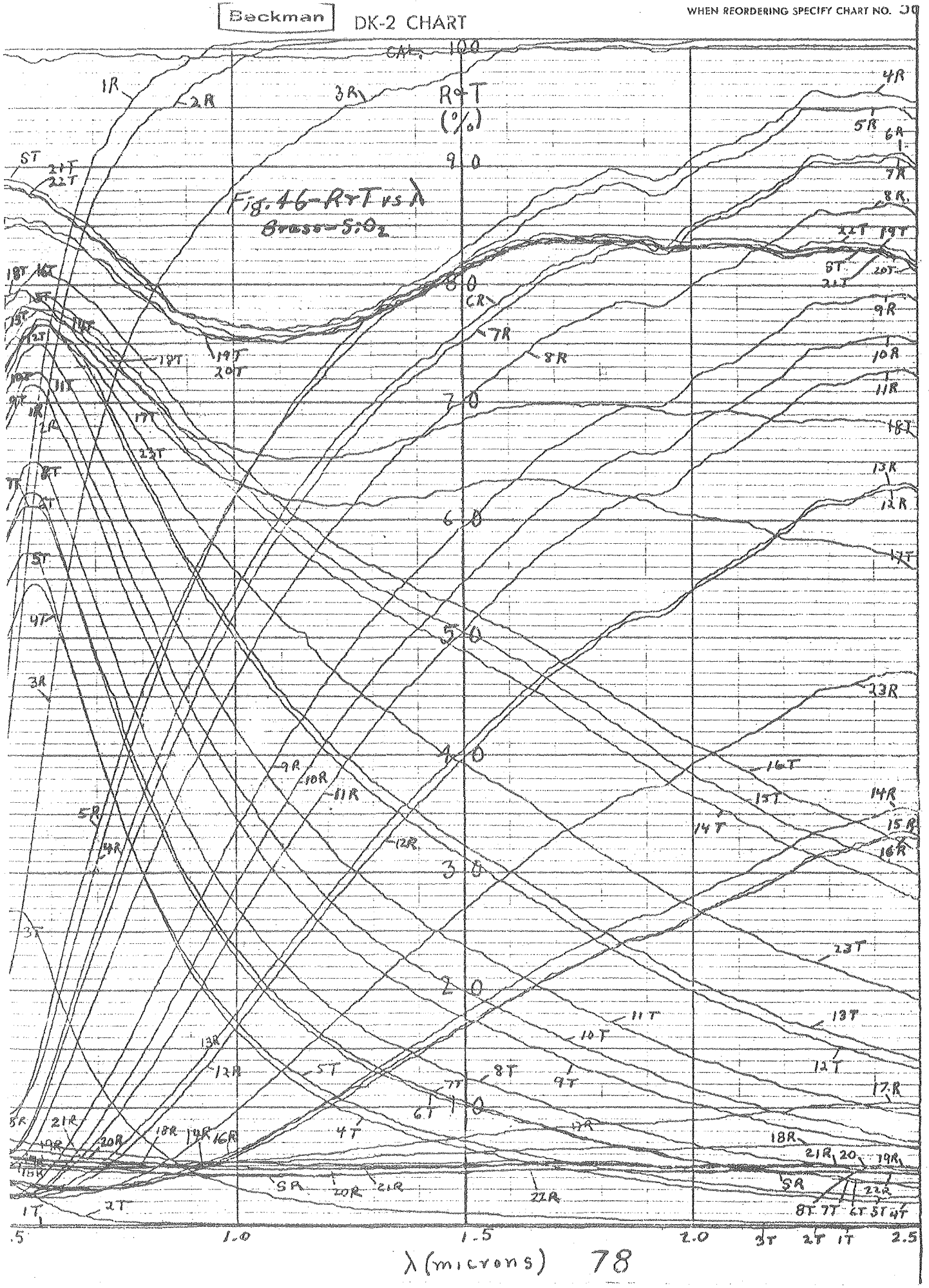
XBB 785-6196

glass for first deposited material, to a pure brass layer as more material is added. This conclusion was drawn when the SEM study was first done. Unfortunately, although it was desirable to overcoat the samples used in the study to protect them and to validate comparisions with previous brass reflecting layer studies (see Section 2.2) other elements of the program forced delay of the overcoating for 3 months. During this period the sample were stored in an evaporator at $\sim 10^{-6}$ Torr. All of them were then overcoated with SiO_2, after cleaning where necessary to remove lead attaching cement and/or fingerprints from the SEM studies. The reflectivity of the uncoated samples, presumably due to oxidation, had gone down apprecialby in the intervening period as shown by Figures 46 and 47 (cp. e. g. sample 19). This decrease is also evident in the reflectivity values in Table 2 which were measured after overcoating. Overall the characteristics are similar to those obtained in the reflectivity study (Figures 32 and 33) with a downward displacement in reflectivity for a given reflecting layer thickness.

The prime conclusion from this study is that the material being deposited under this program does not go down in a conventional manner. This confirms other data from in-house programs which have led to coating materials having other unusual and/or superior characteristics especially for combinations of materials or elements such as brass or SiO_2 etc.

2.4 Weathering

At the beginning of the program, the major failure mode of the windows was expected to be oxidation of the underlying layer through pinholes in the protective layer, accentuated by heat during solar illumination. Although such pinholes could be markedly reduced in number in a clean production environment, they were difficult to avoid in the relatively dirty environment in which the experimental work was performed. It was not believed that there would be any degradation in any of the materials due to UV and the high UV irradiation commonly used in testing plastics, paints etc. was considered unnecessary. High UV lamps also tend to have short lifetimes, making long term testing difficult. Consequently, it was decided to use high intensity


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 30
R+T
(%)
Fig. 46 - R+T vs λ
Brass - SiO2
λ (microns)
.5
1.0
1.5
2.0
2.5
100
90
80
70
60
50
40
30
20
10

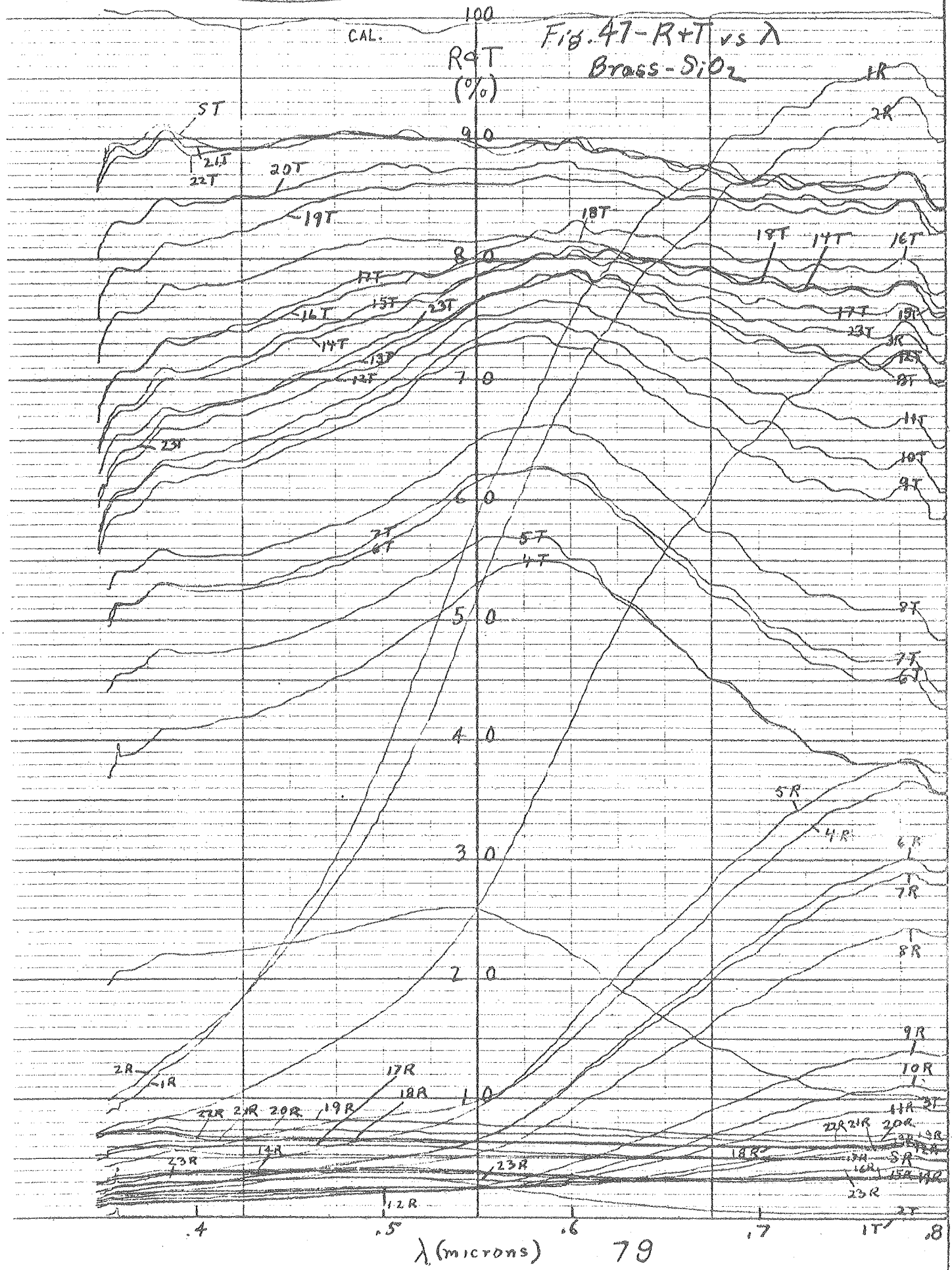


Fig. 47 - R+T vs λ
Brass - SiO2
R&T (%)
CAL.
100
90
80
70
60
50
40
30
20
10
.4
.5
.6
.7
.8
λ (microns)
1R
2R
5T
21T
22T
20T
19T
18T
17T
16T
15T
23T
14T
13T
12T
11T
10T
9T
8T
7T
6T
4T
5R
4R
6R
7R
8R
9R
10R
11R
3T
1T
2T
23R
12R

tungsten-halide floodlamps for solar simulation and possible acceleration of the effects of solar heat, with distilled water spraying for rain and humidity effects. A weatherometer was designed accordingly, primarily using available parts.

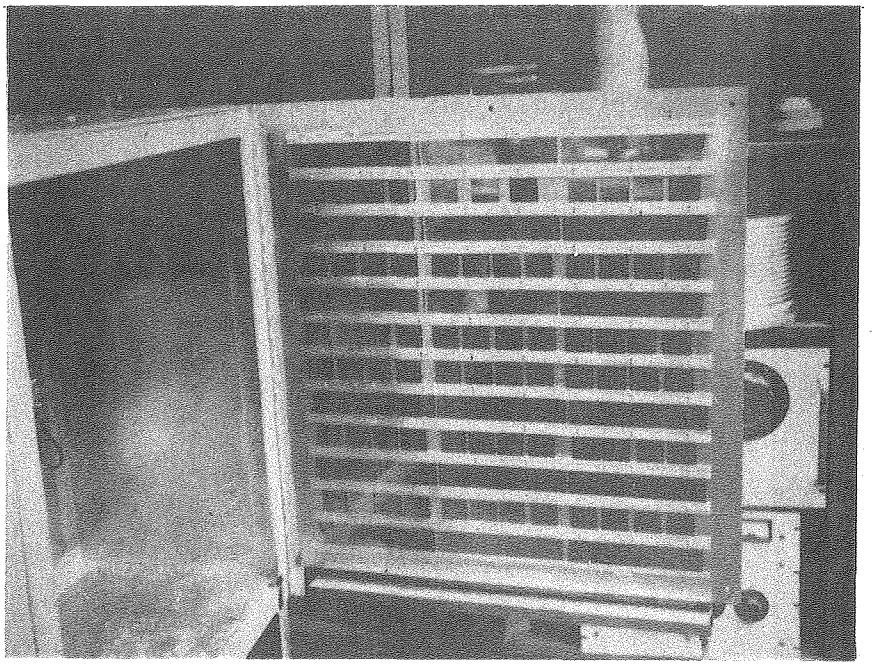
Abrasion testing was to be done by two methods, the first a series of extended washings using industrial methods, and second, the deliberate introduction of scratches into the coated surface with subsequent testing in the weatherometer to look for related accelerated degradation.

Two types of samples were made. For the first few systems (brass-Al_2O_3, brass-SiO_2, Al-A_2O_3 and Al-SiO_2), relatively complex families of 6 samples were made at four different overcoat thicknesses. After problems were encountered with the weatherometer, weathering samples were usually 2 samples at 1-4 different overcoat thicknesses for each system investigated. To date, only preliminary weathering data is available. Since the goal is a 20 year lifetime minimum, more time must be expended, even with accelerated methods, before the best system can be determined.

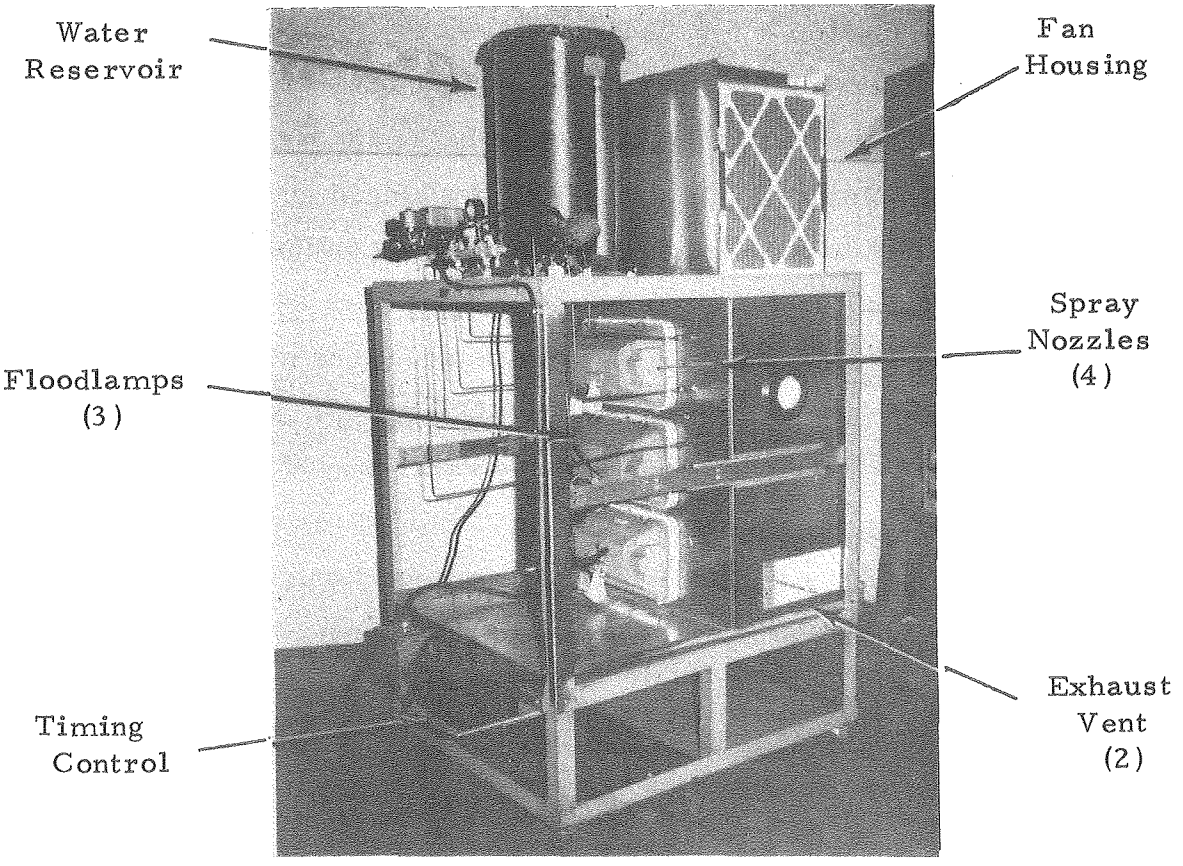
2.4.1 Weatherometer

Closer examination of the weathering problem showed a need for a more complex weatherometer than originally planned. Originally a 3' x 3' x 3 1/2' Al enclosure with a single 1500 watt G. E. tungsten halogen floodlamp (2,000 hour lifetime) on one end was constructed. The average light intensity on the 30" x 32" sample plane (opposite end) was considerably below the desired 1-2 suns (air mass zero-AMO) and the illumination was changed to 3 of the 1500 watt lamps in a bank as shown by Figure 48(b). This increased the light level at the sample plane to 1 1/2 suns at the edges to 2 suns at the center with the extreme corners down to 1.2 suns. Excluding the corners, the light level was therefore equivalent to approximately 2.1-2.8 suns AM2 (terrestrial). Since the weatherometer runs 24 hours a day, each day is therefore equivalent to approximately 6 to 8 conventional days at 1 sun for 8 hours.

On top of the chamber there is a 20 gal. stainless steel tank



Samples in Weatherometer
(a)



Weatherometer
(b)

Figure 48. Weatherometer XBB 785-6194

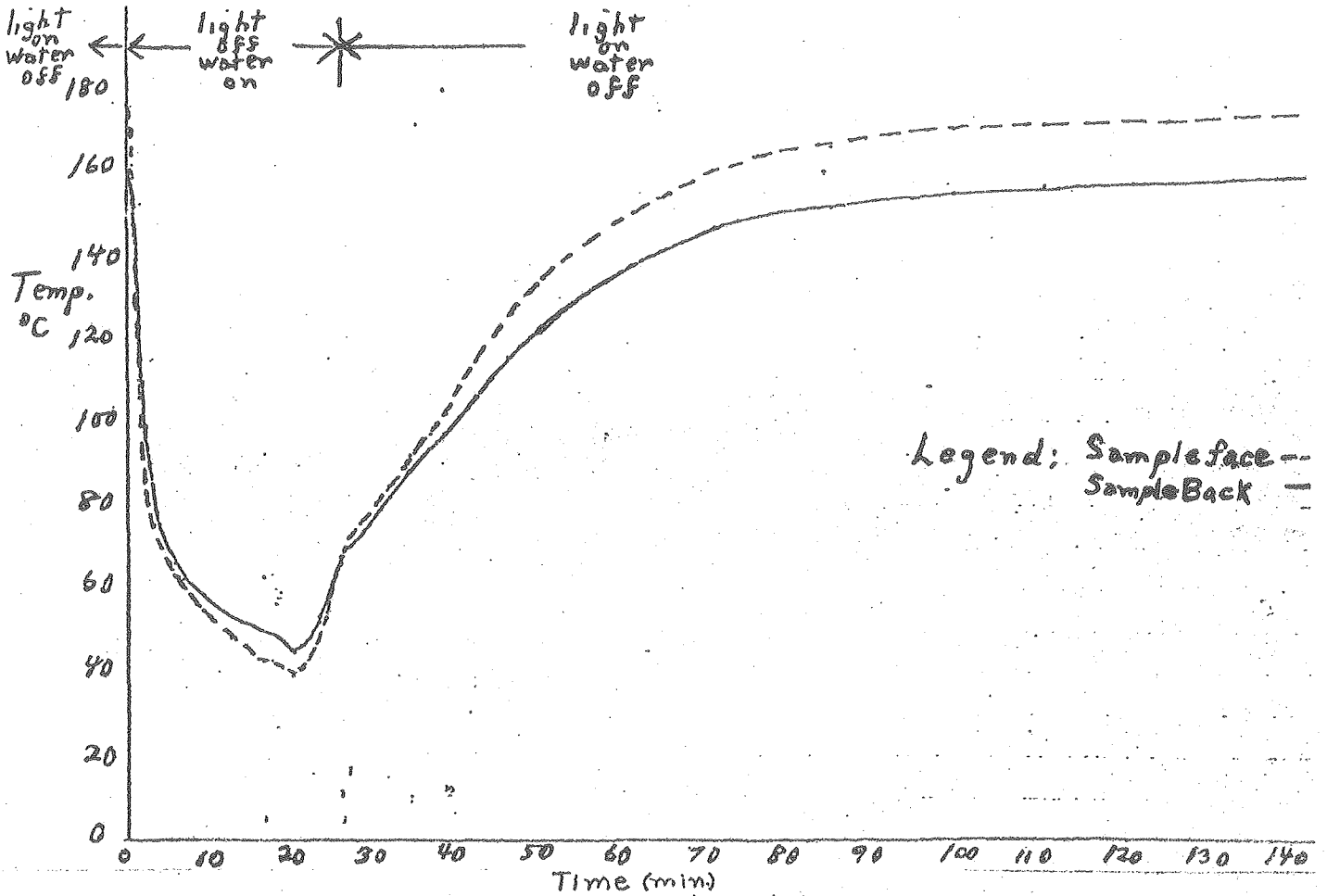
containing distilled water which can be sprayed over the entire sample thorugh 4 spray nozzles using a venturi effect. Pressure to activate the nozzles was originally supplied by the plant air compressor in order to provide aeration of the water. However, severe deficiencies (see below) were encountered due to problems with the compressor and the pressure supply was changed to dry N_2 from the liquid N_2 storage tank. The floor of the chamber is sloped to sample end so that the sprayed water is collected in a trough below the samples for removal.

A spray cycle time was included to control the on-times of the spray and light. The 4 hour cycle was set for 3 hrs. and 20 min. of solar illumination.followed by 20 mins. of spray followed by solar illumination etc. A long spray cycle every 4 hrs.rather than a shorter one every hour was used to accentuate temperature gradient effects and to ensure establishment of the lower temperature equilibrium.

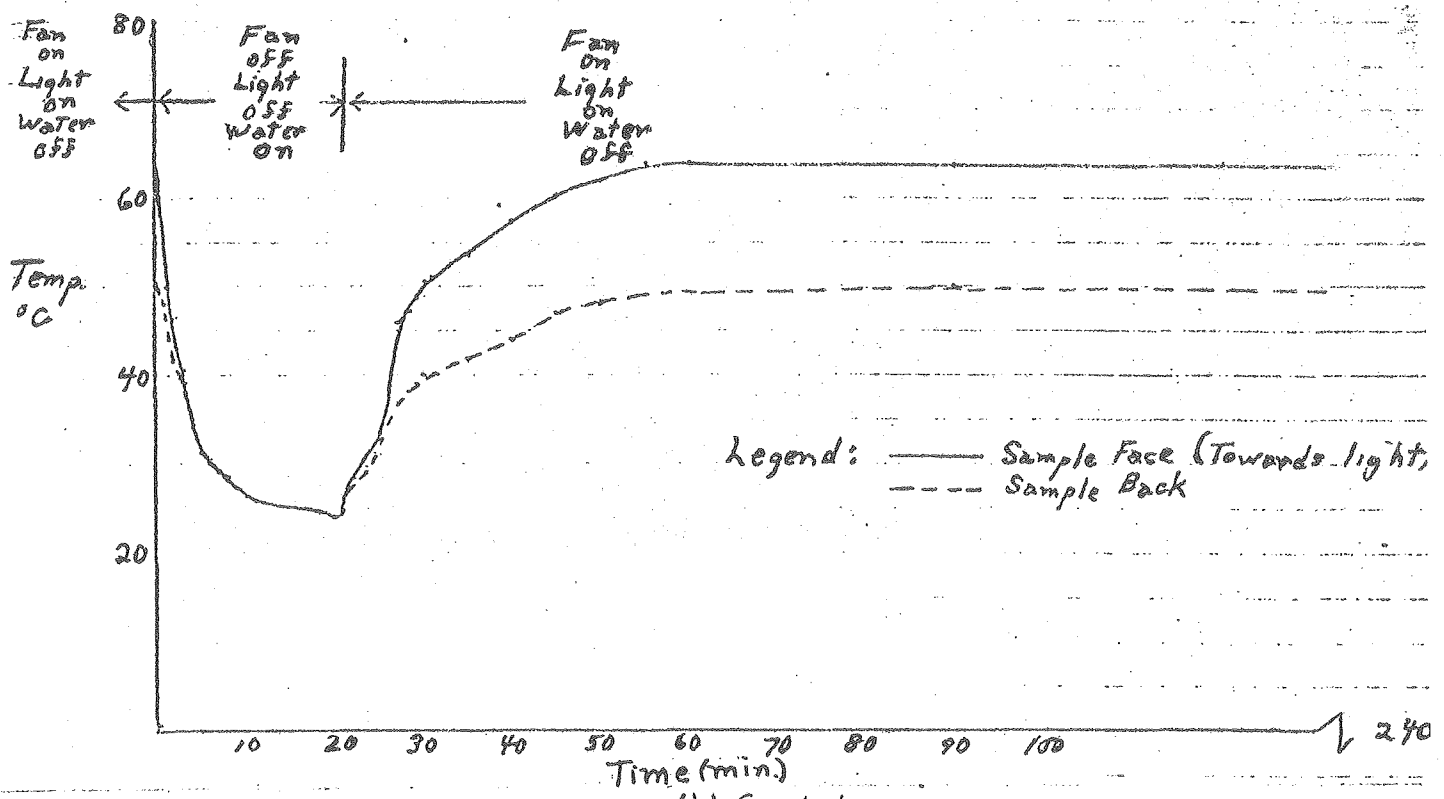
The fan housing and exhaust vents shown in Figure 48(b) did not exist in the original version but were added to reduce sample temperatures after it was established that the samples were being exposed to unrealistic conditions. The samples are held in slots on the hinged end of the chamber opposite from the light bank. Before addition of the cooling fan, the temperature cycles on the front and rear sample surfaces were as shown in Figure 49(a) (Note: Temperature cycles obtained by simultaneous thermocouple measurements). The fan altered the cycles to those shown in Figure 49(b). In the non-fan case, the front of the samples rose to temperatures between 177 and 182°C (depending on ambient) and dropped to approximately 40°C during the spray cycle. For the first few minutes after the water spray was turned on, the entire sample chamber was filled with live steam which was subsequently determined to be a much too severe environment. With the cooling fan, the excursion runs from approximately 64°C to 23°C (1°C below ambient when measured). The fan is turned off during the spray cycle to avoid interference with the spray pattern.

2.4.2 Sample Testing

For the first four systems investigated (brass-SiO_2, brass-Al_2O_3, Al-SiO_2, Al-Al_2O_3), families of samples, consisting of one 2 x10'' and five



(a) Uncooled



(b) Cooled

Figure 49 - Weathenometer Cycles

2" x 2" samples deposited simultaneously, were generated. Different families had different overcoat thickness viz. A (<. 05 μ), B (. 125 - . 175 μ), C (. 275 - . 325 μ) and D (> 2 μ) and different materials (i. e. Al_2O_3 or SiO_2). All combinations were used for both brass and aluminum. In order to maximize information return, the families were coated in a non-uniform distribution so that different samples within a family had somewhat different reflecting and overcoat layer thicknesses. Other factors (discussed below), however, dominated the results and made additional information retrieval from these smaller thickness variations withing larger variations impossible from a practical point-of-view. Five samples (2" x 10" and four 2" x 2") from each family (180 samples in all) were placed in the weatherometer and one was removed for testing in Phoenix, Arizona outdoor testing facility. One sample from each family in the weatherometer was deliberately scratched with a full penetration scratch before insertion.

Initial optical tests on these samples were made on the DK-2A at an outside facility before the instrument was purchased by KCI. Due to the large number of samples, the tests had to be restricted to R and T measurements in the . 35 - 2. 5 μ range and calibration runs were made relatively infrequently. In addition, the instrument was in relatively poor operational condition at that time so that the data are considered inferior in accuracy to those subsequently obtained on the reconditioned machine at KCI. The results are, however, considered valid within better than ± 5% in most cases.

Although specific R and T curves were taken for all samples initially, other overriding factors, in view of time limitations, made it advisable to select one or two samples from each family for subsequent measurements. Usually these were from the center of the distribution where the deposited layer thicknesses corresponded to the calculated values. In general, the other samples from the families behaved similarly, from appearance, to those tested in full detail.

After 500-1000 hours of exposure, two distinct effects which did not obviously correlate with the sample characteristics, were observed on a few samples. These included cracking (two-2" x 10" and one-2" x 2")

and the sudden appearance of what appreared to be run-etch or drip lines at two sample areas. These run-etch lines seemed to initiate on the aluminum backing plate and continued from one sample to the sample directly below as well as on the intervening aluminum. With time the lines spread and eventually covered most of the few samples. The lines appeared on Al_2O_3 coated samples only while the cracked samples occurred in the hottest (i. e. central) regions. Both of these effects occurred over weekends and it was subsequently determined (by checking water reservoir level) that the air compressor must have been off for part of the weekend period. This was confirmed by maintenance which also stated that due to the malfunction, some treated (to prevent bacterial growth) process water had been getting into the compressed air for some time. This process water contains additives (high pH-10.6 in 1% solution) and causes considerable problems (e. g. plugged cooling lines on pumps) in systems in which it is used. Consequently it is used only for non-critical applications.

It was experimentally determined that the sample cracking occurred when the air pressure was off. In this condition, the gravity head in the water tank was sufficient to rouse the water to leave the nozzles as a stream (rather than as a spray) which struck those samples directly in its path. The cracked samples were at, or near, the point of interception and the cracking is believed due solely to differential cooling effects on the hot (180°C) samples.

A literature search also disclosed that alumina in boiling water (or higher temperatures in presence of water) forms various hydrated alumina compounds (e. g. alpha alumina monohydrate). Although chemical analysis for the presence of such compounds could not be performed under the experimental conditions, the probability of their formation in the live steam atmosphere is considered high. In addition, after removal of all samples from the door for washing tests (see below), the door was observed to be highly etched in all areas. The presence of aluminum containing compounds or solutions running down the door and over the samples under the prevailing conditions was therefore obvious. Data on the fused silica (Corning 7940) used for the SiO_2 coated samples also states that the erosion rate in water at 100°C is 125 Å/24 hrs.

Since the steam condition lasted for a few minutes or more per cycle, the erosion rate for SiO_2 coatings in the high temperature condition was probably in the order of a few angstroms or more per day.

The obvious conclusion from the above was that the test conditions were much too severe and unrealistic. The relative results may also have been distorted by the relative rate of compound formation and erosion at the elevated temperatures. After 1, 900 hrs operation, it was therefore decided to reduce the temperature by mounting a cooling fan at the top of the enclosure with vents at the bottom. The temperature extremes were therefore changed as shown in Figure 49(b) where the limits are approximately ambient to +140°F while still maintaining the same light level. After this change no new run-etch lines formed and no additional cracking occurred.

After the initial 664 hrs. of operation, all samples were removed for washing. At this point the cracking and run-etch problems were just beginning. The following procedure was used:

1. All samples were removed from the weatherometer and those samples having the deliberate scratch line were separated out. It was decided not to wash the latter in order to evaluate the effect of scratches as a separate mechanism.

2. The remaining samples were mounted on a large (4' x 4') Al sheet using double backed tape and were washed with a solution of 1 tablespoon of ammonia per gallon of tap water (commercial solution used on Prudential Building in Boston). Frequency of washing was taken as once every two months (the stated frequency for the Prudential Building) yielding 60 washings for a 10 year period equivalent. For each washing, each sample was squirted with the ammonia solution, "scrubbed" at least twice with a foam scrubbing sponge and then squeegeed at least twice to remove the solution.

3. The samples were remounted in the weatherometer.

Close observation of the unwashed scratched samples showed no readily apparent erosion at this point resulting from the full penetration (through deposited layers) scratches. An important point was that there was no apparent erosion on those scratched samples which were in the same area of the weatherometer as the samples that developed run-etch lines. Clearly the latter was a separate phenomenon.

There was no apparent change in any of the washed samples as a result of the washing procedure, with the possible exception that one of the samples (Al-Al_2O_3-B) seemed to show less beading of the washing solution after 30 cycles. The Al plate, however, which was used to hold the samples, showed substantial attack (after 24 washings) by the ammonia solution. The entire surface was attacked with the difference in rates due to the presence of the samples being readily apparent. Measurements of R and T were not carried out at this point because the DK-2A was not yet installed at KCI.

At 1900 hours, the decision was made to change the temperature cycle in the weatherometer. While the physical changes were being made, all of the samples were removed for careful observation, an additional 10 year washing cycle and updated R and T measurements. Some of the samples were found to be severely attacked by the high temperature steam environment, with the Al-Al_2O_3 families having Al_2O_3 thicknesses of <. 05 μ and . 3 μ being most severely attacked. Surprisingly, the Al-Al_2O_3 family with Al_2O_3 only . 15 μ thick showed no evidence of attack except where run-etch lines had initiated. This family was right in the middle of the sample run where the light intensity was the highest, but was not at the center of a spray area as were the other two families. Run-etch lines had therefore started much later on this family.

In general the Al-SiO_2 and brass-SiO_2 families were equivalent and much better than the Al-Al_2O_3. The best resistance to the <u>steam</u> environment occurred with the brass-Al_2O_3 combination. This was not apparent until detailed R and T measurements (see below) were made and the weatherometer was altered, because an early run-etch line on the thinnest overcoat (<. 05 μ) samples indicated rapid attack. (As noted before, no new run-etch lines were

formed after the maximum cycling temperature was reduced.) The conclusion drawn was that the Al_2O_3 was, in general, as resistant, or more resistant than the SiO_2, but the presence of the underlying Al acted to accelerate attack once the attack was initiated.

Visual examination of the samples also showed no evidence of erosion of the reflecting layer from the "deliberate" scratches on selected samples and in general the brass-SiO_2 samples looked the same as originally. However, on some of the samples a number of small scratches were evident which were not visible on reinsertion in the weatherometer after the first 10 year washing test. These scratches were clearly in the direction of the squeegee stroke during washing and are believed due to dragging glass chips, from the unprotected edges, over the surface during the washing tests. Subsequent erosion by the steam made them visible on very close examination. It was decided to conduct all future washing tests on individual samples, rather than on a gross batch as in the original wash test, to avoid transferring glass chips from one sample to another.

In order to directly evaluate the effect of washing, one sample from each set was measured for 0.5 → 2.5 micron reflectivity and was then washed for a 10 years equivalent (60 washes - 1 every two months). At the end of each 10 washes, the sample was remeasured for reflectivity (see Figure 50 for example of results). There was no evidence of any decrease or change in reflectivity for any of the sixteen samples tested in this manner. It is therefore believed that abrasive effects will not be a significant factor in altering the reflectivity charaateristics of the windows.

Comparison of Figure 50, giving the washing results for brass-Al_2O_3 sample 1-4 (Al_2O_3 less than .05 μ), with the original curves in Figure 51 (marked 4) indicates some decrease in R and increase in T. These changes are believed due to shifts caused by the steam erosion and/or spectrometer changes and are not expected to occur at the lower temperatures with measurements made on the overhauled spectrometer. This has already been partly established as shown by Figure 52 which gives the curves for sample 1-4 after an additional 1870 hours under the new conditions. In this case both R and T have increased slightly over the values obtained during the wash

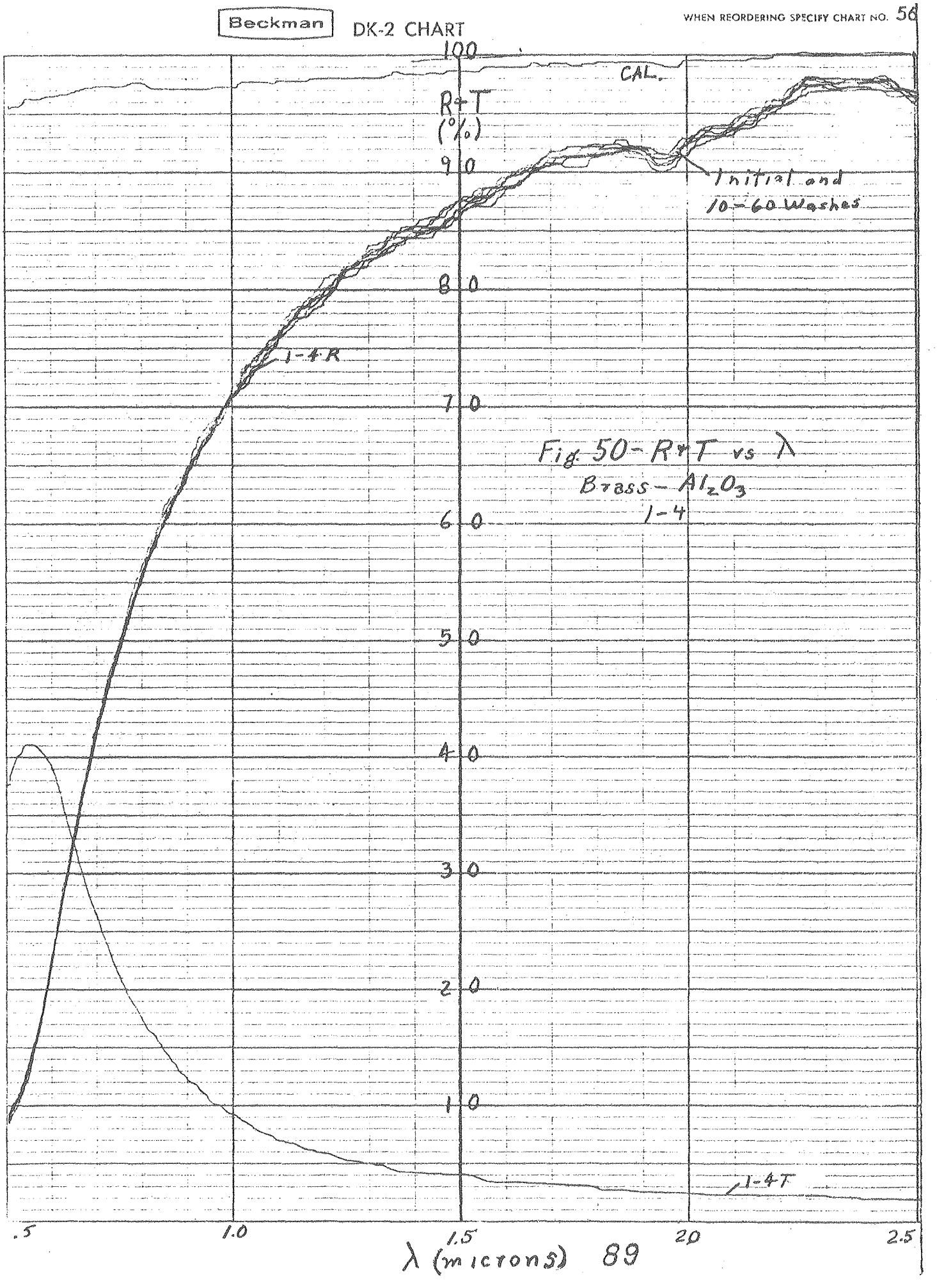


Fig. 50 - R+T vs λ
Brass - Al_2O_3
1-4

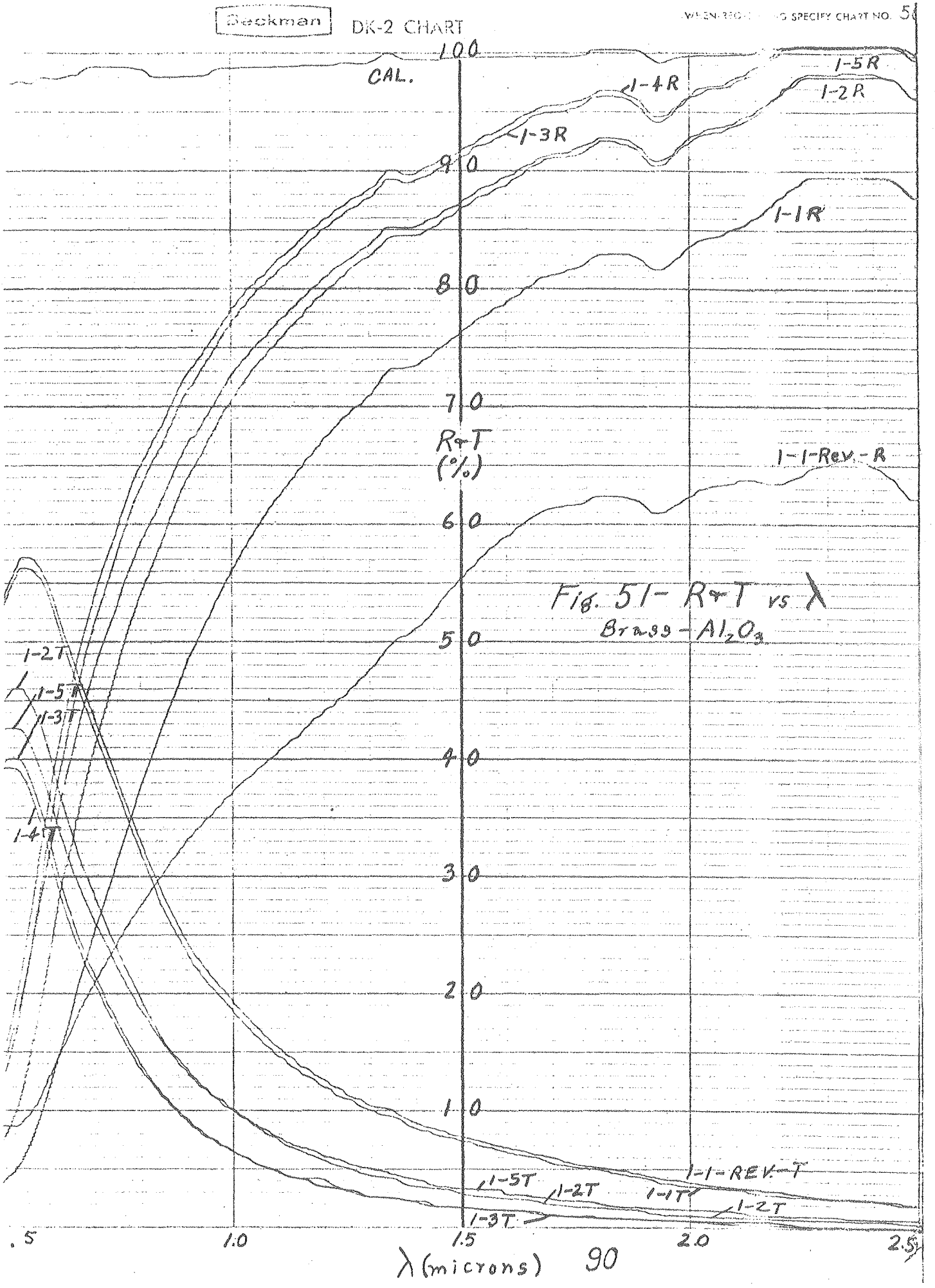


Fig. 51- R+T vs λ
Brass - Al_2O_3

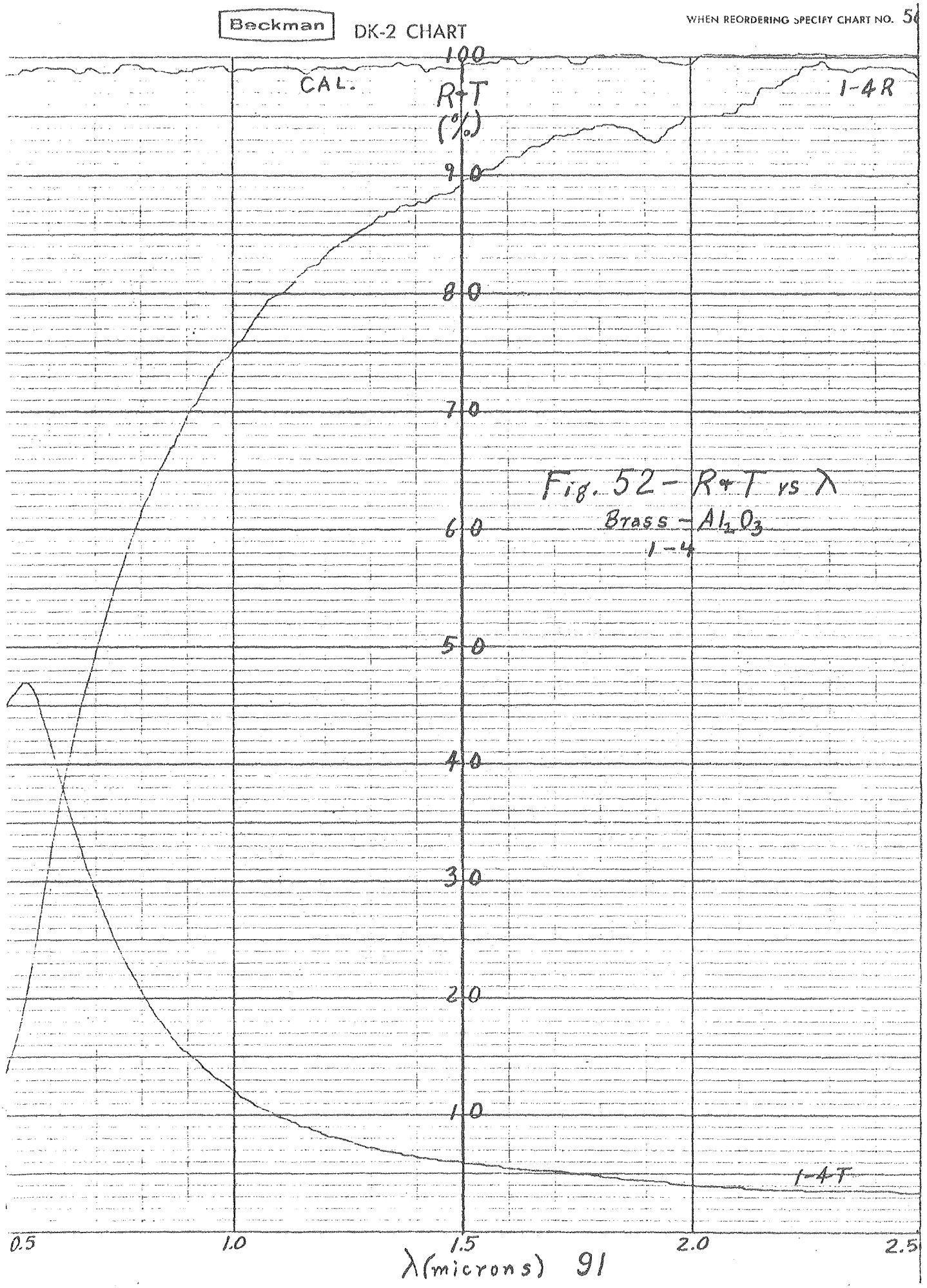


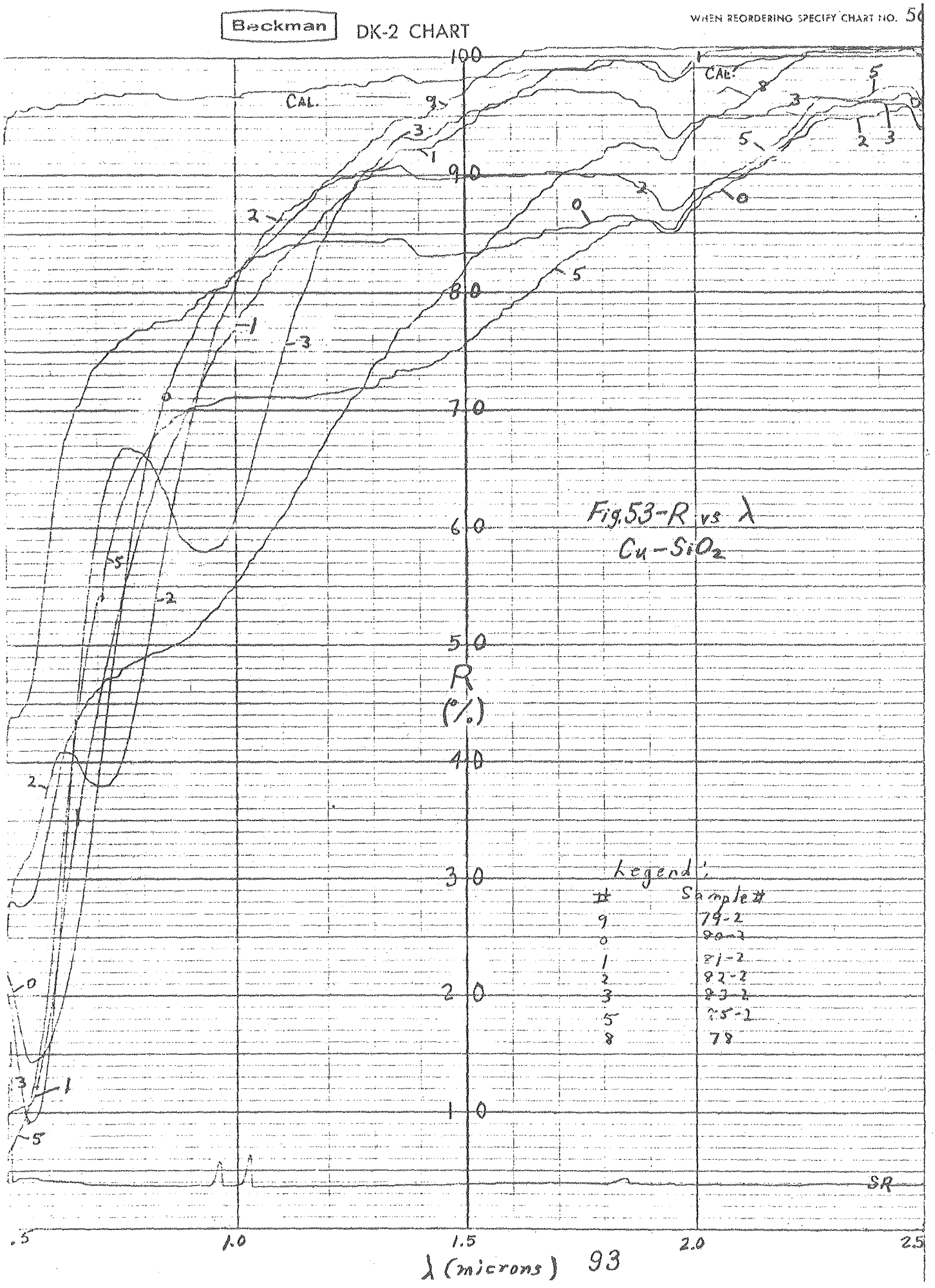
Fig. 52 - R+T vs λ
Brass - Al_2O_3
1-4

cycling (Figure 50). This increase is believed due to changes in the spectrometer during intervening overhaul with small changes in gain and linearity giving improved performance as shown by the calibration line. In any event, there was no additional visual or measurable performance decrease.

All of the other samples to be reinserted in the spectrometer for additional long term weathering tests in Phase 2 of the program were individually given an additional 10 years equivalence wash. The reinserted samples therefore have all been washed for a total of 20 years equivalence. Some samples were removed to make room for the weathering samples from the new material sets (Ni-SiO_2, Cu-SiO_2 etc.). The latter (2-6 different overcoat thicknesses per set) all have SiO_2 overcoatings, a decision based on early weatherometer results. Clearly, based on the brass-Al_2O_3 results, new weathering sets should be made to evaluate Al_2O_3 vs. SiO_2 under the new conditions without prior exposure to the old conditions. However, the SiO_2 is deposited more rapidly and would be preferable, all other factors being equal. No deterioration of any of the new SiO_2 overcoated sets (Ni, Cu, Ti, Inconel 671, Mo, Ag, Au reflecting layers) has been observed after 1870 hrs.

Figures 53 and 54 give R curves for the Cu-SiO_2 weathering set before (Figure 53) and after (Figure 54) the 1870 hrs. exposure. The R values appear to be higher after exposure in all cases, but again this has been attributed to changes in the spectrometer. Some small spots were evident on the samples which did not come off with ammonia, water, or solvents but did come off after brisk rubbing with a kleenex type material. These deposits are apparently mineral in nature and presumably come from interaction of the distilled water with the stainless steel and/or copper feed lines to the spray nozzles. The best solution for this would seem to be more frequent washing to remove the residue before it builds up appreciably.

The samples sent to the outdoor test facility were exposed under concentrated light (8 suns) conditions. Since they are only exposed for part of the day, vs. 24 hours in the weatherometer, the total light exposure per day is roughly equivalent in the two cases. At the end of one month, very little if any change was noted in the samples. At the end of two months, some


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 56
CAL.
CAL.
100
90
80
70
60
50
40
30
20
10
R
(%)
.5
1.0
1.5
2.0
2.5
λ (microns)
SR
Fig. 53-R vs λ
Cu-SiO2
Legend:
Sample
9 79-2
0 80-2
1 81-2
2 82-2
3 83-2
5 85-2
8 78

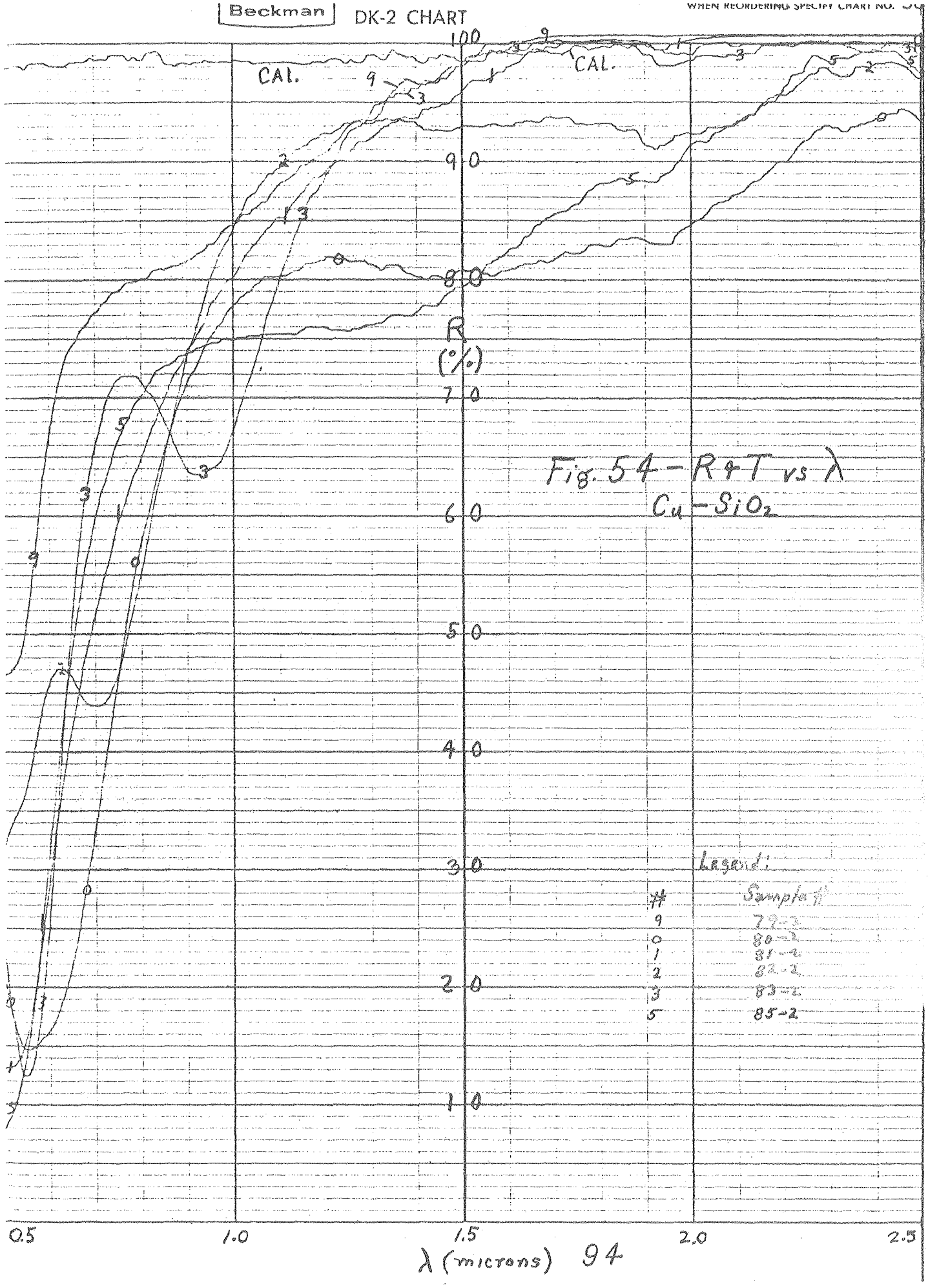


Fig. 54 - R & T vs λ
Cu - SiO_2

of the samples were reported to have darkened and a few spots were reported. Since a different observer reported on the two occasions and since it is believed highly unlikely that the samples could darken (bleaching is much more likely), comments on the results are reserved until the samples are returned at the end of 3 months (scheduled to occur after end of Phase 1 of the program).

In summary, results of the weathering tests, once the conditions were properly established, indicate that the basic method can be used to fabricate high performance durable windows for both office and residential applications. Additional weathering tests are planned as part of any further development effrot.

3 Sample Windows

3.1 Office Windows

At the end of the program, the results of the various investigations on reflecting material-overcoat combinations etc. were combined with coloring requirements to fabricate a family of samples to demonstrate practical window designs. The Cu-SiO_2 system was chosen and PPG solar-bronze plate glass was selected as the substrate. The family is representative of those that could be fabricated with other material combinations and demonstrates general reflectivity and coloring combinations.

Characteristics for those samples are given in Figures 55-58 (cp. Figure 2). The reflectivity curves are as taken using MgO standards throughout the .35-2.5 μ range. As discussed in Section 2.2 they are therefore correct from .35-1.0 μ and then are progressively high from 1-2.5 μ. These curves were all taken before the post-program standards development discussed in Section 3.2.

It is immediately obvious from Figure 55 and 56 that very little energy is transmitted in the 0.7-2.5 μ region with most of the energy reflected at the front surface. Figure 57 shows that the reflectivities in the visible vary considerably such that there is a significant variation in transmitted

81% of the .7-2.5 μ incident energy while the solarcool only reflects 21%. The error in the W-128 value due to the use of MgO is no more than a few percent since the major error in reflectivity occurs where the AM2 spectrum is lowest. A value of 75% would be very safe. On the other hand, there is little error in the solarcool value since the reflectivity for it is low where the measuring error is highest. It is still clear, however, that W-128 has very superior performance to that of the solarcool when compared by the criterion set up in Section 1. In general, the sample set and other similar window designs can be adjusted to provide reflected energy values of 70-80% in the .7-2.5 μ region.

The KCI processing at the current state of development could be used for outside or inside reflecting layers in single glazed applications as well as for enclosed layers in double glazed applications (i.e. on inside of exterior glass). Following the discussion of Section 1, preliminary calculations were made on the effect of these windows on energy consumption in high rise, heavily fenestrated buildings having high indirect heat sources such as lighting, personnel etc. First consideration was given to a building which has been otherwise designed for energy conservation and in which the prime problem in all seasons is clearly one of cooling, or as minimum the avoidance of unnecessary solar loads, e.g. a building in a southern region. Modifications were then made to accommodate buildings in more northerly climates, or buildings in moderate areas which have not been optimally designed for energy conservation. Since the problem of calculating the potential energy savings is quite complex, involving many different types of buildings, other conservation measures used, the total number of buildings involved etc., the calculation was intended only to determine the relative performance of different windows and rough approximations were used where convenient. In particular, normal incidence of all solar related radiation was assumed and factors other than solar load effect were considered the same (all windows unshaded and drapeless) for all structures. Where possible errors in calculating the improved performance of the KCI method were minimized by calculating the energy savings on the conservative side.

Calculations were made for a building having the following characteristics:

Base	: 200 feet on each side - square
Height	: 40 stories
Floor Spacing	: 12 1/2 feet
Fenestration	: 80%
Window Area/Side	: $200 \times 12\text{-}1/2 \times 0.8 \times 40 = 8 \times 10^4$ ft.^{2}
Orientation	: Faces in cardinal directions

Comparisons were made vs. various other single glaze types of glasses including inside reflecting, clear, absorbing only and reflecting only. Using very conservative figures the results were interpreted as indicating a min-max energy saving of 3,000-6,000 barrels of oil/year for a typical single glazed building or 4,500,000 barrels of oil/year per 1,000 typical buildings or equivalent.

Another way to consider the savings is to correlate it with expected new building construction. Daly[(4)] has estimated that if new buildings were designed to achieve 60% energy savings, then savings in new construction could amount to 5.8 million barrels of oil/day by 1990. Commercial buildings which comprise 42% of building stock therefore represent a potential savings of 2.4 million barrels/day. For the windows under consideration here and assuming a rough average energy dissipation factor of approximately 150,000 BTU/ft^2 yr* for buildings currently under construction, the calculated savings (for typical building; 1.6×10^6 ft.^{2} of floor space) of 2.61×10^{10} BTU/year (4,500 barrels of oil/year) represents a 10.9% energy savings. If we further assume that the new windows will be used in only 10% of new commercial buildings the total savings are

$$2.4 \times 10^6 \times \frac{10.9}{60} \times 0.10 = 43,600 \text{ barrels/day or } 16\text{M barrels/year}$$

---

(4) Energy and the Built Environment: A Gap in Current Strategies; The American Institute of Architects, 1735 New York Ave., N. W., Wash. D. C. 20006.

* Weighted average value for 16 office buildings from data given in "Energy Conservation Design Guidelines for New Office Buildings" GSA document, July '75.

by 1990. This does not include any retrofit applications and is calculated using numbers which have been consistently taken on the conservative side.

Operational cost saving for the min-max conditions are \$54-108 K/year and \$81 K/year for the average case. Reference 1 established that for cost effectiveness, the initial fuel savings per year with an improvement must be greater than 6.4% (assumes 20 year life, 10% interest, 6% fuel inflation rate) of the initial "incremental" capital expense. Using this criterion the total incremental cost for the typical building would have to be

$$\frac{\$81,000}{.064 \times 3.2 \times 10^5} = \$3.95/ft^2$$

or less. Obviously this will vary for the different regions because of different actual savings, but since the projected incremental selling price, including manufacturing and administrative O.H., profit and retail profit (if windows go through retailing outlet rather than direct) is less than half of this value, the windows are likely to be attractive for economic as well as energy saving reasons.

The above estimates are concerned only with new buildings of a certain type and do not include retrofit applications. For the latter, it is clear from the program results that the required characteristics may readily be obtained on the plastic substrates. Specific development on choice of substrates, means of application etc. would have to be conducted before reasonable estimates could be made of the associated energy savings.

3.2 Residential Windows

Potential for application of the basic technology to residential uses by shifting the reflectivity threshold to longer wavelengths (see Section 1) was investigated as a secondary goal of the program. The major emphasis was on developing acceptable performance using a manufacturing method with a projected price structure consistent with the residential market.

In most of the reflecting layer-overcoat combinations, it was possible to include sufficient samples with longer wavelength thresholds to define the performance of the combination for residential purposes. The basic objective was to optimize the transmittance (i. e. minimize reflectance) of the combination in the region from .4-2.5 μ (i. e. over AM2 spectrum) while simultaneously providing high reflectivity at 10 μ (i. e. at room temperature radiation).

The Cu-SiO_2 and Ag-SiO_2 sets provided very promising results and were selected for performance analysis. Since only a few samples were involved it was decided to obtain reflectivity curves vs. the best possible standard in all wavelength regions. This was done on an in-house project after the program was completed and the results were not available for feed-back into the reflectivity study results etc., nor could they be in view of the many curves involved. Basically Cu standards were developed and compared with MgO in the .5-2.5 μ region using the DK-2A and with an Al standard in the 2.5 μ - 50 μ region using the Mark IV at Lincoln Labs. The Cu standards have reflectivities of > 97% (absolute) in the .7-50 μ range, and are > 98% for most of the region. For much of the range they exceed the Al standards.

Figure 60 includes a large number of cross-standards. For present purposes, the curve marked MgO-02' gives a comparison of the Cu standards vs. MgO on the double scale (0-200%). The former run 14% higher than the latter at 2.5 μ, gradually decreasing to the same value at .72 μ where MgO is know to be 98% reflective. The curves taken previously for the various reflecting layer-overcoat combinations were not as much in error as the total difference above since the error for any given sample depends on the absolute reflectivity.

For example, the curves are shown for samples 74 (3-74) and 72 (3-72) taken directly against the Cu standards. These can be compared to the curves for the same samples taken vs. MgO (Figure 32). The differences in this case are smaller at the longer wavelengths than the differences between the standards, and the curves are seen to cross over in the

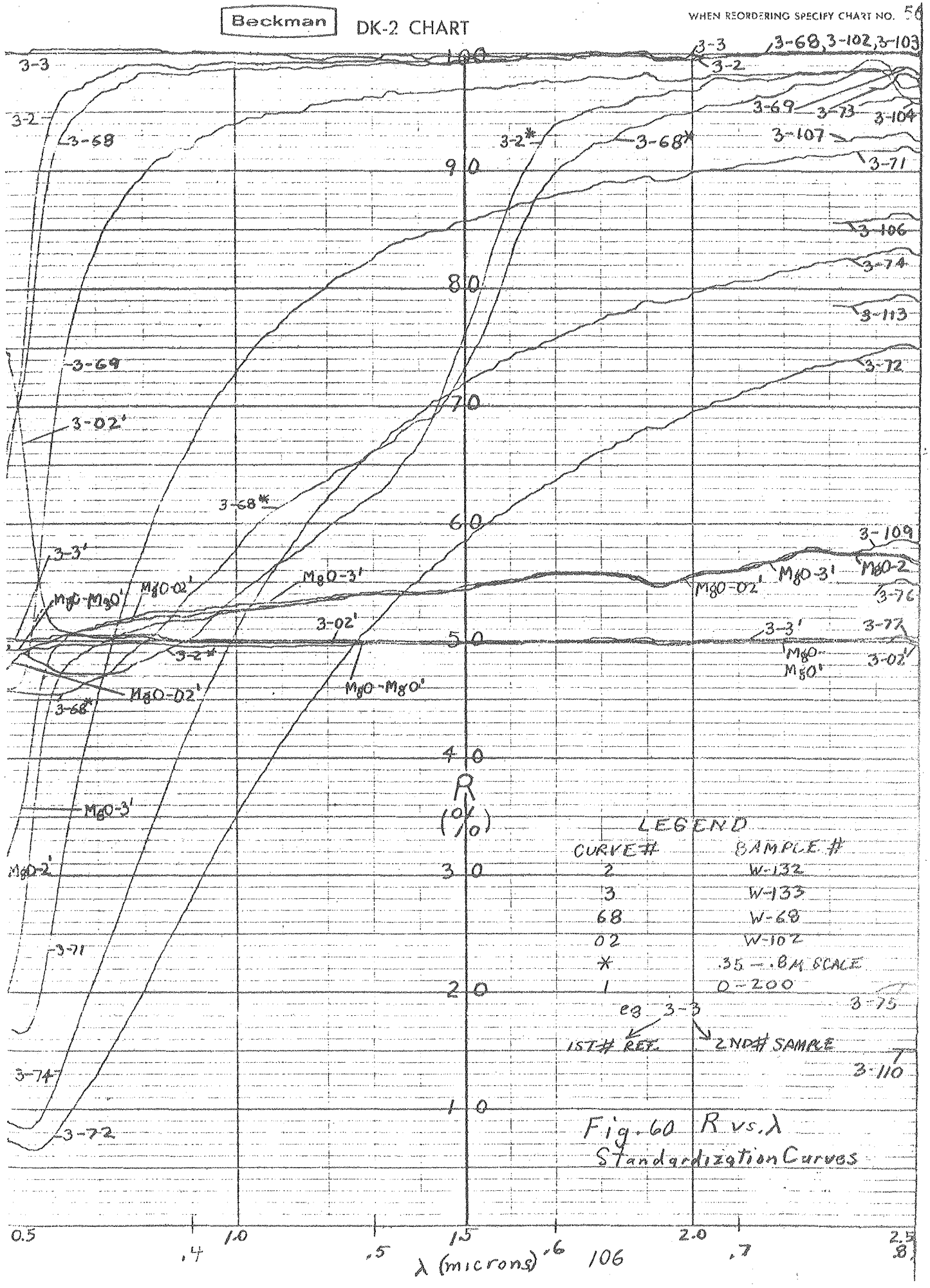


Fig. 60 R vs. λ
Standardization Curves

1.2-1.5 μ region rather than at .7 μ. This shift is primarily due to slight changes in spectrometer function, since the two curves were taken 2-1/2 months apart, and has little effect on the total derived curves shown in Figure 61. The latter show the AM2 spectrum, the reflectivity (132) of the Cu standards vs. the Al standard on the Mark IV in the 2.5-50 μ range, and composite reflectivity curves for samples 72, 74, and 76 (Cu-SiO_2), and 109 and 113 (Ag-SiO_2) over the entire spectrum from .35-50 μ (76 and 109 were not taken above 10 μ). The MgO values were used up to .7 μ, and the Cu standard values from .7-50 μ. These curves are believed to give good absolute values throughout the whole region.

These composite curves have been included to show the total performance of the windows as viewed from the coated or "inside" surface. The requirement for this surface is to reflect the longer wavelength radiation emitted by internal generators (walls, furnishings, people etc.) which are primarily at room temperature (i.e. 10 μ black body), back into the room, thereby containing it. The requirement for transmissivity, however, is for .35-2.5 μ radiation entering from the outside through the uncoated surface. Reflectivity characteristics are quite different from this side because of the much thicker dielectric involved. (Transmission curves are similar from the two sides.) The appropriate response curves, taken from the uncoated sides are shown in Figures 62 and 63 for the samples of interest. Comparison of the curves for samples 72 and 74 with the comparable curves from the coated side (Figure .60 with Cu standards for .5-2.5 μ and Figure 33 with MgO for .35-.8 μ) shows the difference, much of which is due to absorption in the glass substrate.

The values from the reflectivity curve given in Figures 62 and 63 can be multiplied by the AM2 spectrum to yield a reflected energy curve (as was done for the office windows; see Figure 59), which can be integrated and compared with the energy under the AM2 spectrum. This was done for the Cu-SiO_2 samples 72, 74, and 76. For sample 72 this yields a value of 5.9% for the reflected energy from .4-.7 μ and 16.3 from .35-2.5 μ. By comparison, the reflectivity from 4-50 μ on the inside surface runs from 82-90%.

NO. 341-L310 DIETZGEN GRAPH PAPER
SEMI-LOGARITHMIC
3 CYCLES X 10 DIVISIONS PER INCH

EUGENE DIETZGEN CO.
MADE IN U. S. A.

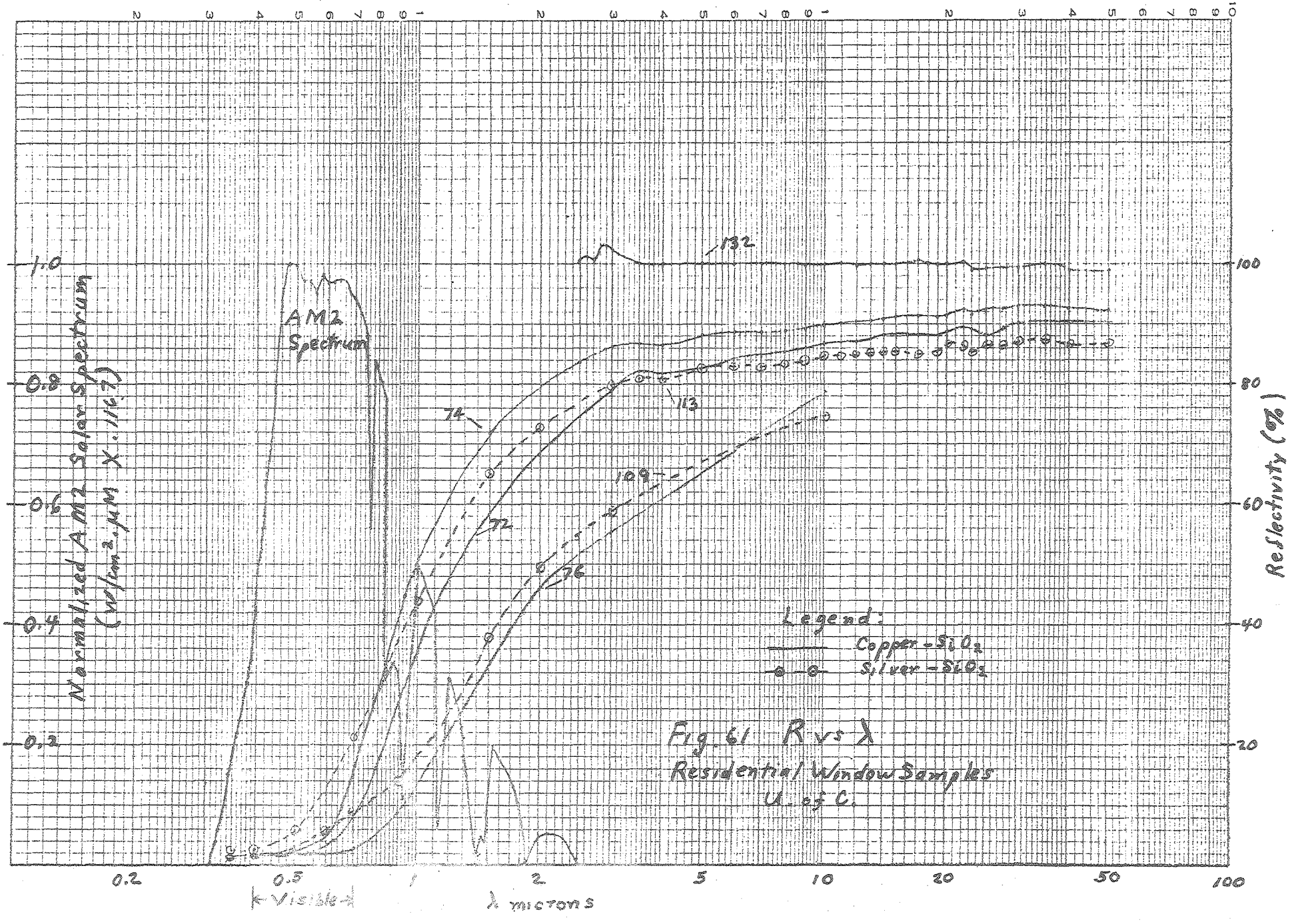
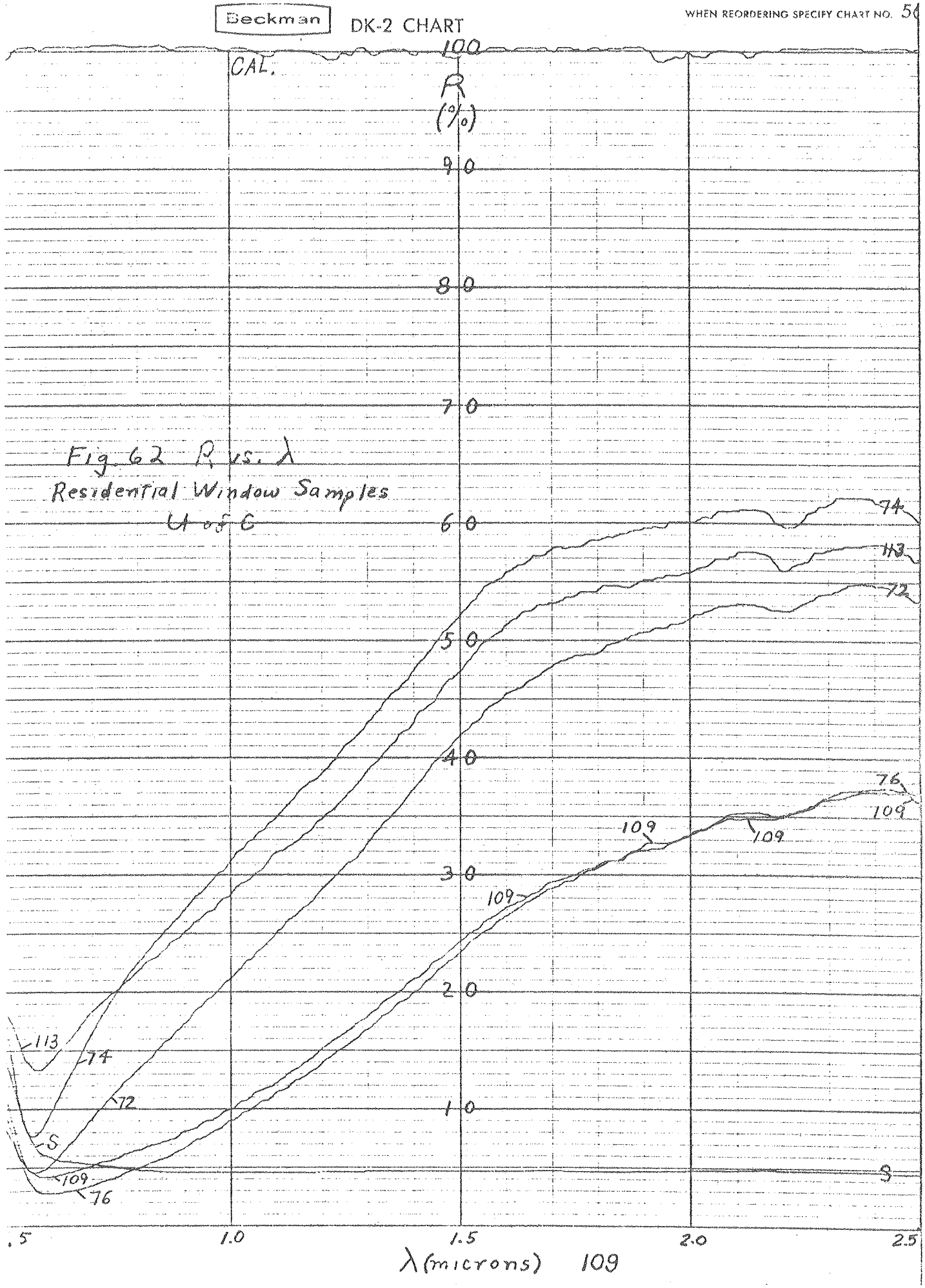
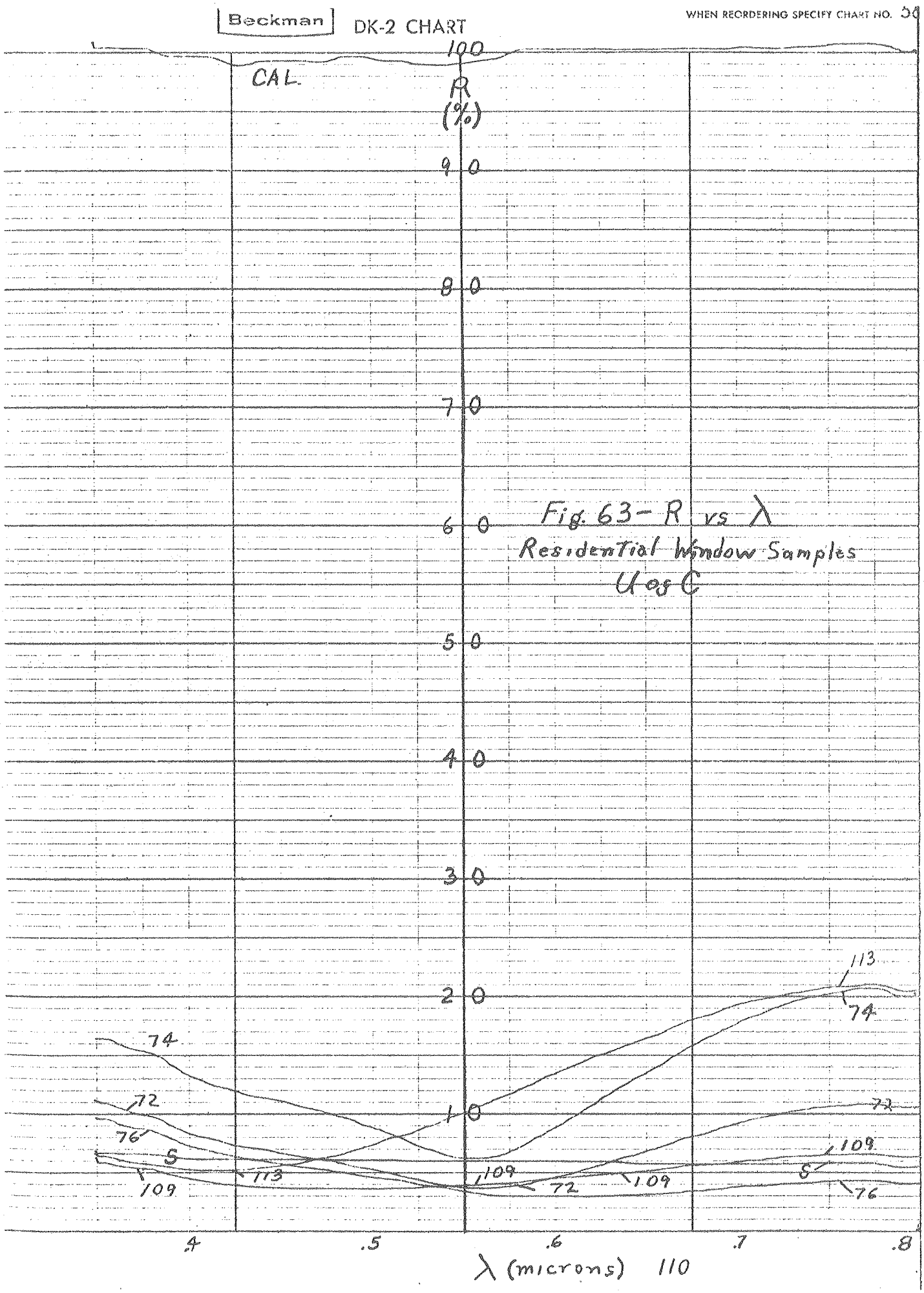


Fig. 61 R vs λ
Residential Window Samples
U. of C.


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 56
CAL.
R
(%)
100
90
80
70
60
50
40
30
20
10
Fig. 62 R vs. λ
Residential Window Samples
U of C
74
113
72
76
109
109
109
109
113
74
72
S
109
76
S
.5
1.0
1.5
2.0
2.5
λ (microns)
109


Beckman
DK-2 CHART
WHEN REORDERING SPECIFY CHART NO. 50
CAL.
100
R
(%)
90
80
70
60
50
40
30
20
10
Fig. 63 - R vs λ
Residential Window Samples
U of C
113
74
74
72
76
5
109
113
109
72
109
72
109
5
76
.4
.5
.6
.7
.8
λ (microns)
110

The comparable numbers for sample 74 are 10. 9%, 23. 2% and 86-93% respectively, while those for sample 76 are 4. 5%, 8. 8% and 79% (at 10 μ - lower below and will be higher above). In all three cases, the visible reflectivities are low with 72 being comparable to uncoated glass and 76 being below. Basically sample 76, which reflects the lowest fraction of incoming radiation, has the lesser ability to contain the internal radiation while 74 has the highest values in each catagory. Actual design choice would depend on may factors, including coloring (see below).

The visible transmissivities for these samples are given in Fig. 33 where the glass substrate (ST) is seen to transmit roughly 88% of the incident light. The difference between the ST curve and the sample curves primarily represents energy absorbed by the deposited layers. It should be noted that the reflecting layer-overcoat will be on the inside of the window so that the extra absorbed energy (both visible and near IR) is absorbed on, and will heat up, the inner surface. A good portion of this absorbed energy will be transferred to the interior by convection. Because of this, the increased reflectivity at 10 μ for a sample such as 76, which has a low reflectivity over the AM2 spectrum for incident radiation, will be almost entirely a positive factor in improving heat retention efficiency. However it may still be desirable, depending on overall design, to increase the long wavelength reflectivity somewhat (e. g. sample 72) while simultaneously reflecting more of the incident radiation.

Although not calculated in detail, the average performances of samples 109 and 113 for the Ag-SiO_2 system will be very similar to those of samples 76 and 74, respectively. Samples 72, 74 and 76 have a pleasant neutral brown coppery cast on both transmitted and reflected light, with 76 having a very minor coloring. Samples 109 and 113 have a light neutral gray appearance as viewed in normal direction from either side and have a light yellow brown reflectance as viewed off normal; the coloring for 109 is very slight.

At the present stage of development, these residential window types are believed to be sufficiently developed, within their performance

limits, to justify preliminary development of production equipment. For large scale manufacturing, the projected cost factors are consistent with the relatively low priced residential window market. The structure of the windows is quite simple and the requirements on production tolerances are relatively loose. Finally, the durability, as demonstrated by the present status of the weathering tests, is almost certainly adequate for extended life under the interior environment in residential structures. Most notable are the lack of deterioration at the deliberately introduced full penetration scratches (see Section 2.4) and the fact that scratches, in general, are not apparent due to the light coloring.

A final point of interest relative to these windows is that they are effective in conserving energy even when installed on the sides of the house not subject to direct solar illumination. The effective 20 BTU/hr/ft^2 (see Section 1.2) reduction in heat loss for single glaze applications (12 BTU/hr/ft^2 for double glaze applications) is still valid. Since many, if not most, new homes will still be built with windows on all sides, this is a very important factor. Also, an inexpensive plastic retrofit becomes quite attractive for energy savings in situations where storm windows are not warranted (intermediate climates) or are economically unfeasible. A quick calculation shows that the energy savings for each house having 200 ft^2 of windows is 1-2 barrels of oil/year, assuming a 150 day heating season.

U.S.GPO:1978-789-158/(F)7

This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

TECHNICAL INFORMATION DEPARTMENT
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720