CPRM

LAMIN

RELATÓRIO DE ESTÁGIO

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Período de Estágio: 10-03-75 - 23-05-75

Local de Estágio: Laboratórios do U.S.G.S.
U.S.A.

1. INTRODUÇÃO

O presente relatório é uma descrição sumária de todas as atividades desenvolvidas durante o meu estágio de treinamento junto aos laboratórios do "United States Geological Survey", através do Convênio MME/USAID.

O período de 01/03 a 23/05, que corresponde a minha permanência no exterior, pode ser dividido da seguinte forma:

- saída do Rio de Janeiro em 28/02;
- chegada a New York, N.Y. em 01/03, partindo para Washington D.C em 02/03;
- chegada a Washington D.C. em 02/03;
- o período de 03/03 a 09/03, foi destinado às apresentações ao Washington International Center, USAID, Washington D.C.;
- entre 10/03 e 12/04 permaneci em treinamento junto aos laboratórios do USGS em Reston, Virginia;
- em 13/04 viajei de Washington D.C. com destino a Denver, Colorado;
- no período de 14/04 e 16/05, estive em treinamento nos laboratórios do USGS em Denver, Colorado;
- entre 17/05 e 23/05, estive em preparativos para a viagem de regresso ao Brasil, chegando ao Rio de Janeiro em 24/05.

2. OBJETIVOS DO ESTÁGIO

A CPRM e mais precisamente, o LAMIN, preocupado em manter-se atualizado em relação a determinados métodos utilizados para realização de análises petrográficas e calcográficas, que são em grande escala requisitadas, não só pelos projetos executados pela CPRM, mas também pelas companhias particulares, achou por bem enviar-me aos Estados Unidos da América, com a finalidade de fazer um treinamento nestes campos.

3. TRABALHOS REALIZADOS NO ESTÁGIO

O programa desenvolvido pode ser dividido em etapas que correspondem a atividades distintas, assim distribuídas:

3.1 - Apresentação à USAID

Como já foi mencionado anteriormente o período de 03/03 a 09/03 foi destinado às apresentações de praxe ao Washington International Center e USAID, em Washington D.C., onde foram ultimadas as providências necessárias para a realização do programa previsto junto ao "United States Geological Survey".

3.2 - Estágio nos Laboratórios do USGS em Reston, Virginia

3.2.1 - Estudo de Minerais Opacos

Durante quatro semanas, tive uma introdução ao estudo de minerais opacos para obter os conhecimentos básicos, uma vez que o aprendizado no verdadeiro sentido da palavra nesta matéria, só poderá ser adquirido após a observação de um maior número de seções padrões, afim de se obter uma caracterização mais completa das propriedades óticas dos minerais e, consequentemente suas identificações. Convém lembrar, que a microscopia dos minérios no seu sentido mais completo, entretanto, depende como se sabe de uma contínua vivência em problemas relativos aos minerais de minérios e respectivos jazimentos. Na primeira etapa, fiz consulta bibliográfica específica afim de que pudesse, após ter aprendido os fundamentos da parte teórica da técnica de microscopia de minérios, passar ao estudo, propriamente dito, das seções polidas ao exame microscópio em luz refletida.

A seguir, tomei conhecimento quanto ao preparo das seções e passei a dar polimento naquelas que iriam ser por mim observadas, pois devido a pertencerem a uma coleção padrão, não de uso frequente, criam consequentemente, uma camada de oxidação, que deve ser retirada. Êste tipo de serviço é sempre executado pelo técnico que necessita utilizar a seção com a finalidade de estudos comparativos. Após as seções devidamente polidas, passei à observação microscópica para o aprendizado da determinação das propriedades óticas de cada tipo de minério, uma vez que, só com a contínua observação das mesmas e um estudo constante, podemos com maior facilidade caracterizá-las.

Após as noções de identificação dos minerais, passei ao estudo do arranjo textural dos mesmos, quando da observação de seções muito bem definidas e que exibiam arranjos texturais diferentes e bem caracterizados.

Convém ressaltar que êste tipo de análise é apoiado paralelamente, sempre que necessário, pelos exames de raios-X correspondentes. Este ponto foi enfatizado pelos técnicos orientadores que sempre se utilizam dos raios-X ou da microsonda-analisadora, quando estão estudando os minerais opacos.

Durante êste período contei com o valioso apoio técnico dos Drs. Robin Brett e Paul Barton.

3.2.2 - Estudo de Inclusões Fluidais nos Minerais

Minha última semana em Reston, Va., foi ocupada estudando noções básicas, de Inclusões Fluidais nos Minerais.

Após ligeira consulta bibliográfica para se ter conhecimento da técnica empregada, tive a oportunidade de observá-la em microscópio de luz transmitida. Utilizei platina de aquecimento, com a finalidade de determinar a temperatura em que a inclusão foi englobada pelo mineral. Fiz também congelamento com Nitrogênio à -195° C, podendo determinar o líquido dentro da inclusão, se era somente água ou esta contendo alguma outra substância. Essa é uma técnica interessante para se determinar principalmente as temperaturas de formação dos depósitos minerais, facilitando tirar maiores conclusões. Evidentemente só é utilizada num trabalho de grande detalhe, afim de se obter resultados mais apurados. Nêste tipo de trabalho tive o apoio técnico do Dr. Harvey Belkin.

3.3 - Estágio nos Laboratórios do USGS em Denver, Colorado

3.3.1 - "Spindle Stage"

Nesta primeira parte do estágio aprendi a medir o índice de refração dos líquidos com os refratômetros de Abbe e Jelly. Como é sabido, isto deve ser feito periodicamente, pois êstes índices são sensíveis a mundanças. Fazendo-se a correção em períodos determinados tem-se o índice de refração o mais aproximado possível, evitando erros de determinação.

A seguir aprendi a manipular a "Spindle Stage", instrumento de pequeno tamanho e de relativo baixo custo, que nos permite identificar com precição os minerais transparentes. A "Spindle Stage" consta basicamente de um conjunto de agulhas, onde são montados os cristais depois de escolhidos ao exame de microscópio estereoscópico e colados, em seguida colocados em uma platina e através de sucessivas trocas de índices podemos determinar os índices de X,Y, e Z. Com êstes dados consultamos gráficos, chegando até a identificação do mineral. Esta técnica não é moderna, mas muito empregada pelos técnicos americanos. Tive também oportunidade de identificar vários minerais, utilizando este método, podendo assim me familiarizar com sua manipulação.

3.3.2 - Análise Normativa pelo Sistema CIPW

Com a análise química dada em óxidos, aprendi a calcular a análise normativa de uma rocha pelo sistema CIPW, onde cada letra representa a inicial de um dos quatro autores que o idealizou (Cross, Iddings, Pirsson, Washington).

Através de vários calculos utilizando regras e fórmulas preestabelecidas, obtemos este tipo de análise, que é mais utilizado em rochas efusivas, devido a dificuldade apresentada, para a realização das análises modais e subsequente obtenção da porcentagem de cada um dos componentes, nêste tipo de rocha.

3.3.3 - Análises Petrográficas

Tive a oportunidade de estudar ao microscópio de luz transmitida, seções delgadas de tipos variados de rochas, para sua caracterização mineralógica, textural e classificação.

3.3.4 - Petrografia Aplicada aos Agregados de Concreto

Visitei os laboratórios onde são efetuadas as análises nos agregados de concreto, não somente através do estudo petrográfico de seções delgadas, como também com o apoio paralelo de análises de raios-X e de espectrografia. Não tive oportunidade de trabalhar neste laboratório, pois não estavam sendo desenvolvidos trabalhos desse tipo, mas, obtive bibliografia de grande valia para nós.

Durante êste período de quatro semanas, tive valioso apoio técnico do Dr. Ray Wilcox.

4 - CONTATOS MANTIDOS

Além dos contatos mantidos com os orientadores durante o período de estágio, que considero de grande valia, não só do ponto de vista do aprimoramento técnico recebido, mas também pelo fato de serem pessoas de relacionamento nos Estados Unidos capazes de comigo manterem contatos sôbre a existência de novos métodos de trabalho bem como bibliografia atualizada, pois foram unânimes em se prontificarem a me auxiliar sempre que precisasse, tive também a oportunidade de visitar uma das maiores minas de zinco do mundo, em Franklin, New Jersey, podendo assim conhecer o tipo de ocorrência, sua mineralogia, os trabalhos desenvolvidos de exploração e sua provável gênese.

Assisti palestras e seminários, no mínimo um por semana, onde os geólogos do U.S.G.S. ou de outros países, mostram os diferentes trabalhos por êles realizados ou em andamento, no vasto campo da Geologia. Uma dessas palestras foi ministrada pelo único astronauta-geólogo, que esteve na Lua, e nos expôs os trabalhos executados pela NASA.

5 - ANÁLISE CRÍTICA

5.1 - Washington Internacional Center, U.S.G.S., Washington D.C.

Fase introdutória bastante interessante, pois objetiva esclarecer o programa e os órgãos a serem visitados, além de nos propiciar contatos com pessoas de outras nações, nos permitindo assim saber um pouco mais sôbre hábitos, costumes, tradições, economia etc., de diferentes povos. Nêste primeiro contato também foi-nos apresentada uma síntese de como vive o povo americano, dando-nos uma idéia geral do seu comportamento.

5.2 - U.S.G.S., Reston, Virginia

O treinamento principal desenvolvido nestes laboratórios, referiu-se aos estudos de minerais opacos, tendo esse parecido em minha opinião ter um bom rendimento, levando-se em conta o espaço de tempo transcorrido para o aprendizado básico tanto da parte teórica como da prática em si. Apesar de não ser uma matéria nova, para mim o foi, pois não tinha tido ainda oportunidade de desenvolver um estudo, mesmo que de base, nêste ramo, que é de grande importancia no campo da Geologia Economica.

Quanto ao estudo de Inclusões fluidais nos minerais sei que não é de utilização imediata em virtude do tipo de trabalho executado no momento, mas é sabido que é uma técnica já bem desenvolvida e que nos possibilita obter maiores dados sôbre a formação dos minerais, permitindo-nos tirar conclusões mais precisas, principalmente no estudo de depósitos minerais. Acredito que, num futuro não muito longínquo, quando realizarmos um trabalho mais apurado, tenhamos então a chance de utilizar esta técnica aprendida, claro que, com a aparelhagem necessária.

Assim considero o aprendido nesta fase do programa, de grande interesse no ramo da Geologia Econômica.

5.3 - U.S.G.S. Denver, Colorado

Considerei o aprendizado da manipulação da "Spindle Stage", importante como método auxiliar na identificação de minerais transparentes. Em certos casos, quando êstes pertecem a uma série isomórfica, mesmo com análise de difração de raios-X, torna-se difícil muitas vêzes, sua identificação; entretanto, com este método e sem perda de material, podemos identificar os minerais transparentes.

Os resultados obtidos através de análises normativas das rochas são importantes, como por exemplo quando se deseja definir a gênese de um determinado grupo de rochas principalmente quando se tratam de rochas efusivas. Desta forma, tanto o aprendizado da manipulação da "Spindle Stage", do cálculo da análise normativa pelo sistema CIPW e das seções petrográficas estudadas constituem elementos importantes no estudo mineralógico , petrográfico e petrogenético.

No que se refere a Petrografia Aplicada aos Agregados de Concreto, apesar de não ter tido treinamento neste setor, pude ver o caminhamento de uma amostra ao entrar no laboratório para ser submetida às análises necessárias. Além disso, obtive alguma bibliografia, que nos será muito útil, visto que, contávamos somente com poucos exemplares nesse assunto , em nosso acêrvo.

6 - CONCLUSÕES

Do ponto de vista dos objetivos a serem atingidos, apesar do programa não poder ter sido cumprido na íntegra, conforme previamente estabelecido, por motivos alheios à vontade dos organizadores deste, bem como da bolsista, considerei meu estágio plenamente satisfatório, para o que muito contribuiu a acolhida que tive pelos técnicos do USGS.

Das técnicas por mim aprendidas, algumas serão de utilidade imediata no trabalho corrente do nosso laboratório, outras de emprego quase que imediato, dependendo ainda de um aprimoramento que só pode ser adquirido com maior prática, e outras, num futuro não distante, quando efetuarmos trabalhos mais detalhados e estivermos munidos da aparelhagem necessária.

7 - RECOMENDAÇÕES

Em função dos trabalhos realizados, das observações feitas e das condições atuais do Laboratório da CPRM, sugiro:

- a vinda de especialistas ao Brasil com a finalidade de ministrar cursos e palestras, o que é muito benéfica, uma vez que, ao invés de ser somente um técnico a se beneficiar com os novos conhecimentos, daremos a chance para o aperfeiçoamento de um maior número de brasileiros. Evidente, técnicos esses, capacitados e atualizados, e que estejam dispostos a nos transmitir as novas técnicas e métodos;

- quanto ao material técnico empregado, o equipamento usado em nossos laboratórios é bem atualizado, no que se refere ao tipo de trabalho que está sendo executado. Poderia sugerir de imediato a compra da"Spindle Stage", que é de baixo custo e de manipulação que pode ser facilmente esplanada aos demais técnicos, com relativa aplicação rápida e que é importante na identificação precisa dos minerais transparentes. Quanto a aparelhagem utilizada no estudo de Inclusões fluidais nos minerais, tenho todas as especificações necessárias, que serão fornecidas ao chefe do LAMIN, para quando sua utilização for considerada de interêsse;

- quanto a atualização do pessoal, apesar do técnico de laboratório ser específico, deveria ser dada a êle a chance de se transferir para participar de outros setores dentro da companhia, em campos correlatos, quando assim sentisse necessidade, possibilitando desta forma uma ampliação de conhecimentos que viria a trazer benefícios tanto para o técnico como para a própria emprêsa;

- afim de manter um bom nível de produtividade, é necessário manter os técnicos atualizados em seus respectivos e correlatos campos de trabalho, através da participação em cursos, seminários, palestras e em congresso tanto de âmbito nacional como internacional. E que esta chance deva ser dada a todos os técnicos de um modo geral, podendo assim todos terem oportunidade de se beneficiar, com os novos conhecimentos;

- também julgo necessário, até onde me foi permitido observar, seja dada a atenção devida a todos os técnicos, no que se refere a sua capacidade profissional, afim de serem evitadas insatisfações , geradas pelo não reconhecimento dessa referida capacidade.

8 - AGRADECIMENTOS

Apresento meus agradecimentos a todo o pessoal do U.S.G.S. que me assistiu durante minha permanência nos Estados Unidos e cujas atenções me proporcionaram um estágio eficiente e uma estada agradável. Em especial gostaria de mencionar os técnicos Dr. Ray Wilcox,Dr. Robin Brett, Dr. Paul Barton e Dr. Harvey Belkin, que não só me orientaram mostrando grande interesse em me transmitir seus conhecimentos, mas também me facilitaram a obtenção de toda a bibliografia necessária.

Gostaria também de agradecer à Presidência da CPRM, à Diretoria da Área de Pesquisa, à chefia do LAMIN bem como à Coordenadoria do Convenio MME/USAID, pela oportunidade de estabelecer contato com Laboratórios de gabarito internacional.

Rio de Janeiro, 17 de junho de 1975.

Jane da Silva Araujo

Jane da Silva Araujo
Geólogo

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A N E X O

Notes on

SPINDLE STAGE ORIENTATION PROCEDURES

By

Ray E. Wilcox
U. S. Geological Survey
Denver, Colorado
January 1965

[For microscopes with
polarizer north-south]

Notes on
SPINDLE STAGE ORIENTATION PROCEDURES

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Ray E. Wilcox

The optical element of immediate interest for a uniaxial crystal is the optic axis, and those for a biaxial crystal are the principal vibration directions X, Y, and Z, and the two optic axes. Stripped to the essentials, the positions of the two optic axes in space define the biaxial indicatrix completely, since X and Z bisect their angles and Y is perpendicular to their common plane (the optic plane). In uniaxial crystals the two optic axes coincide, and the plane of the optic axes becomes indeterminant. The positions of these optical elements may be obtained either under orthoscopic or conoscopic illumination (Wilcox, 1959). The conoscopic procedure is generally more advantageous, not only because it is faster, but also because it furnishes a "directions image" and thereby a more complete visualization of the optical behavior of a crystal. Crystallographic planes, such as cleavage or crystal faces, likewise may be oriented and usually are handled orthoscopically for plotting with reference to the optical directions.

ADJUSTMENT OF THE MICROSCOPE AND SPINDLE STAGE

In orientation of crystals for quantitative optical measurements, whether for measurements of principal refractive indices or for plotting of geometric relations of the optical or crystallographic elements of the crystal, it is essential that the microscope be in adjustment, that is, all the elements of the illuminating and imaging lens systems must be centered in respect to the microscope axis. These adjustments are normally done as a matter of good practice in routine orthoscopic work. Since conoscopic illumination here is going to be used for precise orientation, it will be necessary to pay more than usual attention to centering of the Bertrand lens.

Instructions for most of these adjustments may be found in the operating manual of the particular model of microscope. Unfortunately these manuals seldom provide for more than approximate centering of the Bertrand. Precise centering may be obtained by the following procedures after centering of the objective and substage system:

1. With a small opaque grain or other easily recognizable point placed at the crosshair intersection, insert the Bertrand lens (but do not cross nicols) and raise the tube of the microscope until the telescopic view of the object is in focus. If the Bertrand is centered the opaque grain should still be at the crosshair intersection and should remain there upon rotation of the microscope stage.

2. If it does not remain centered, adjust Bertrand centering screws and recheck by rotation of stage.

An alternative procedure of general application is as follows:

1. Start with an interference figure showing a sharp isogyre (preferably containing an optic axis) exactly along the EW crosshair.

2. Rotate microscope stage in steps of exactly 90°. If the Bertrand is centered, the isogyre should lie exactly along a crosshair at each setting, first NS, then EW, then NS again. Adjust the Bertrand centering screws if necessary to achieve this.

The "Reference Azimuth" is needed if the optic or crystal elements are to be plotted stereographically. Throughout this discussion the Reference Azimuth is the reading of the microscope stage when the spindle points exactly south (toward the observer). It may be established as follows: Push the spindle far forward in its slot until the parallel-sided shank can be seen in profile under low magnification. With the spindle tip pointing towards the observer, the shank is made parallel to the NS crosshair, whereupon the reading of the microscope stage is recorded as the Reference Azimuth.

The Reference Azimuth is to be subtracted from each reading of the microscope stage before stereographic plotting (see following section). If the spindle stage can be attached on the microscope stage in such a position that the Reference Azimuth is exactly 0°, then microscope stage readings can be plotted directly. This can be done on most microscopes by appropriate shifting of the spindle stage beneath the stage clamps or by proper placement in the mechanical stage. Should it be necessary in subsequent manipulations to shift the spindle stage on the microscope stage (for instance, to bring a different part of the grain to the crosshairs), the shifting must be done without rotation, else the Reference Azimuth will be changed.

For determination of principal refractive indices only, it is not necessary to establish the Reference Azimuth of the spindle on the microscope stage, since the readings at the oriented positions will not be used except perhaps for quickly resetting the orientation. To prevent confusion due to duplication of directions exactly 180° apart, readings are taken over only the first 180° of rotation of the microscope stage from the Reference Azimuth.

STEREOGRAPHIC PLOTTING

Where it is desired to relate the several geometric elements to each other, the stereographic projection is of great utility. In the following the "meridianal" stereographic net will be used, that is, a net of N-S great circles (meridians) and E-W small circles (latitudes).[1] Either the true stereographic net (Wulff net) or the purposely distorted "equal area" net (Schmidt net) is suitable. The plotting is carried out so that the final plot shows the crystal-optical directions as they lie in space when the microscope stage has been returned to the Reference Azimuth setting and the spindle arm flat at zero on the spindle scale (at the extreme clockwise rotation as viewed from the spindle tip).

Plotting of a geometric element is done as follows:

1. Attach a transparent overlay to the net, mark the North point with a hook representing the spindle arm, as in figure 1, mark the center with a dot and the South point with a tick mark.

2. Subtract the Reference Azimuth from each reading of the microscope stage.[2]

Table 1.--Data for orientation (figures 1, 2, and 5) and index determination

Mineral: Anorthite

(Reference Az.=0°)

Refractive index	Spindle arm reading	Microscope stage reading
Nx 1.575	122°	104°
Ny 1.584	21°	126°
Nz 1.588	51°	40°
Cleavage: 001	150°	155°

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[1] An alternate stereographic net is used by Tocher (1962), Fisher (1961), and Garaycochea and Wittke (1964), who place the spindle axis at the center of the base circle. Microscope stage readings are plotted radially from the center, spindle arm readings counterclockwise from a zero point on the base circle.

[2] Alternatively, a ring may be cut out of a sheet of paper with its inside diameter equal to the net diameter. Tick marks corresponding to the graduations of the base circle are placed on the ring and numbered at 10-degree intervals. The ring is attached to the net, placing the Reference Azimuth of the ring at the N pole of the net. All microscope stage readings then plot directly according to the numbering on the ring.

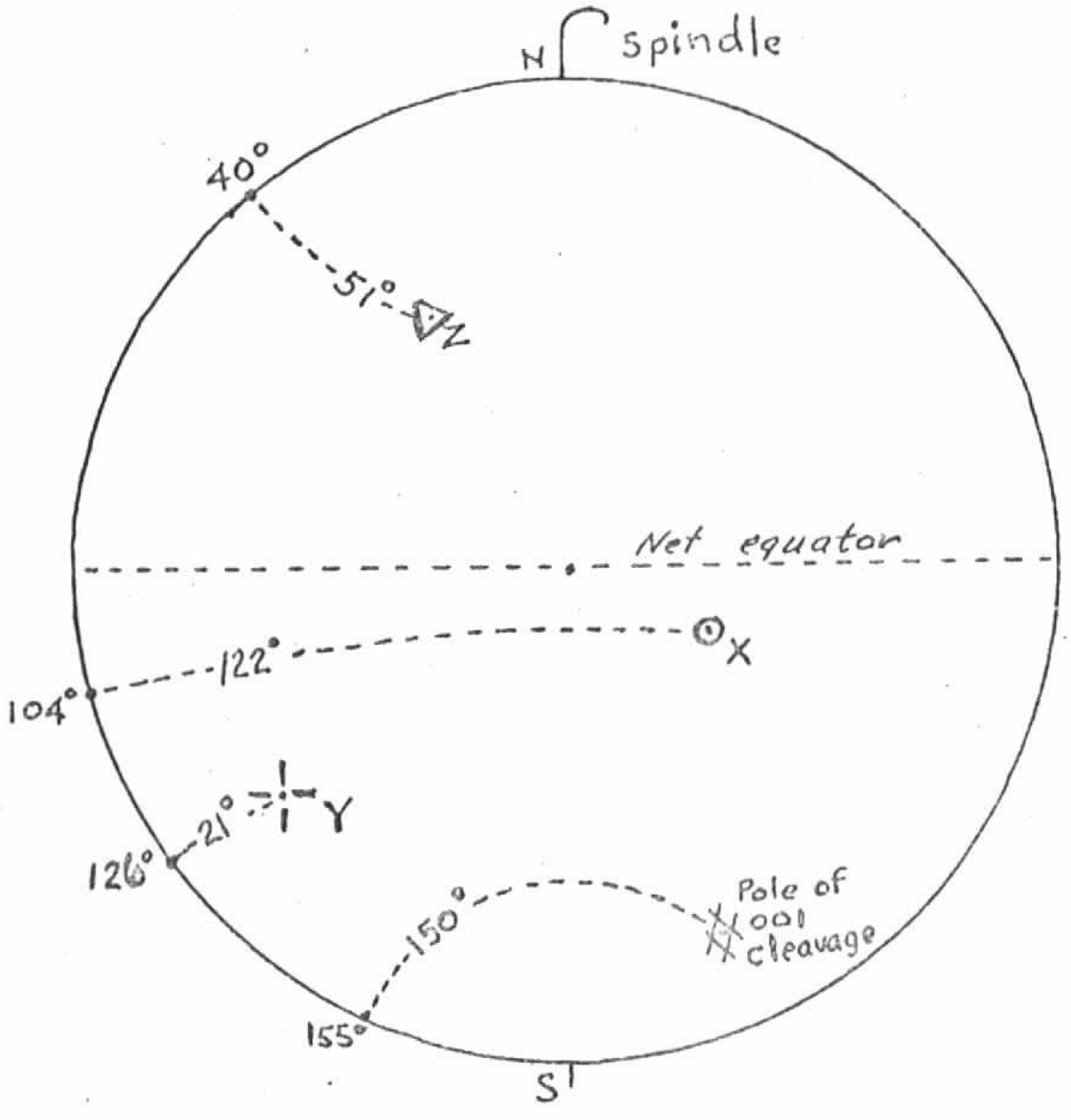


Figure 1.--Principal directions X, Y, and Z plotted on marked overlay on Wulff net with spindle returned to reference position (data of table 1).

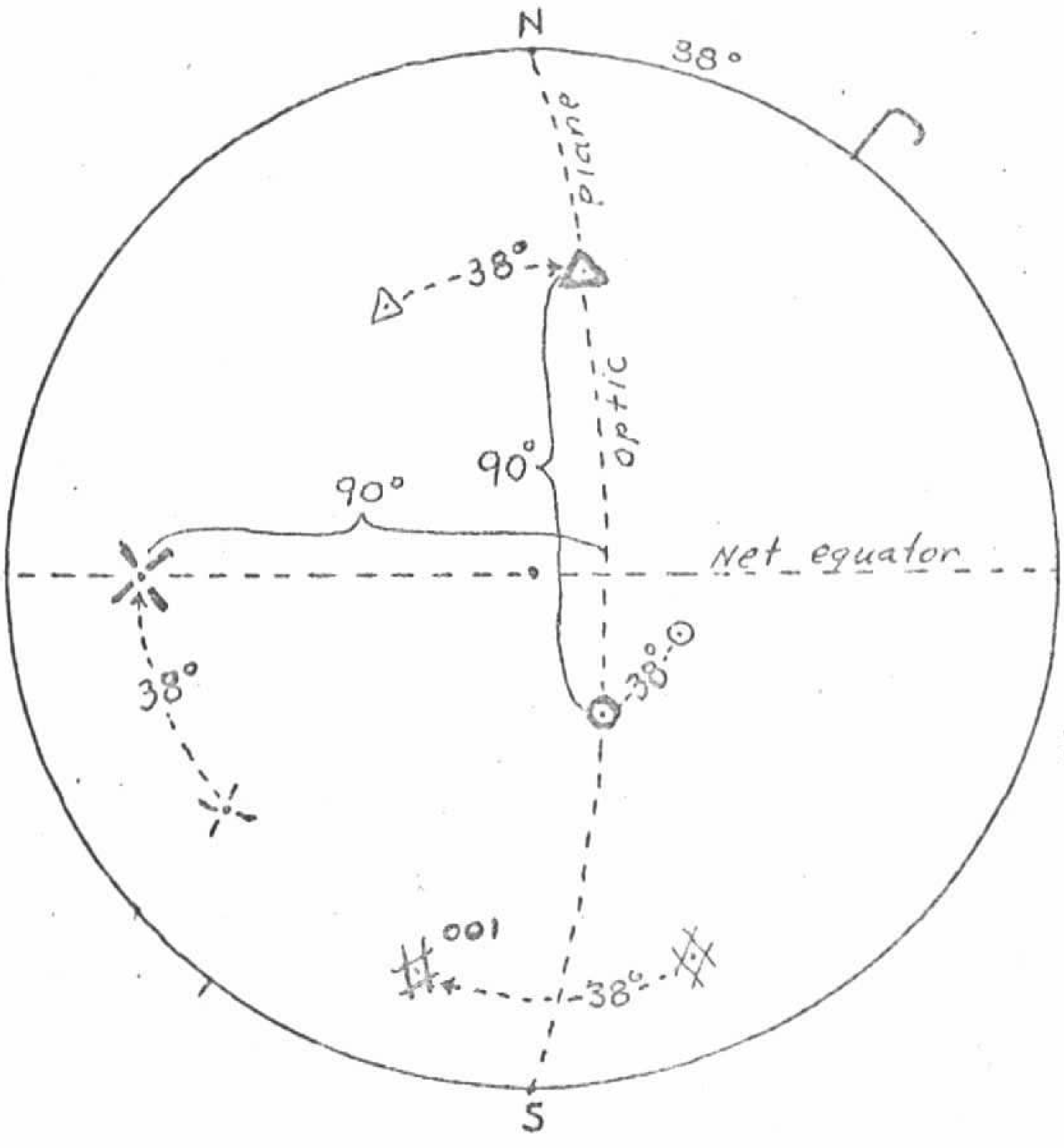


Figure 2.--Overlay of figure 1 rotated to place Y on equator, X and Z on net meridian.

3. Taking the example of the data sheet of table 1, plot X by counting off 104° (the reading of the microscope stage) counterclockwise around the base circle from the N point then eastward along the small circle for 122° (the reading of the spindle arm) and mark the point, as shown in the figure. Plot Y similarly 126° counterclockwise around the base circle and 21° eastward along the small circle, and Z similarly 40° counterclockwise around the base circle and 51° eastward along the small circle. (Note that the conventional symbols of circle, cross and triangle are used to designate X, Y, and Z, respectively.)

4. By definition, the directions X, Y, and Z of the optical indicatrix are mutually 90° from each other. With the overlay in the reference position on the net these angles cannot be read directly, but by rotating the overlay to place two of the points, say X and Z, on the same meridianal great circle, the true angular distance between them may be counted off, as in figure 2. Since the plotted position of Y likewise is 90° from both X and Z, the point Y must fall on the equator 90° from the XZ meridian, as counted off along the equator, which itself is a great circle.

5. As the plot is now placed, the optic plane can be drawn on the overlay by tracing the XZ meridian. Again by definition, the two optic axes will lie on this meridian, symmetrically disposed about the two bisectrices, X and Z.

6. A vibration direction is horizontal NS when the microscope stage has been rotated to bring the crystal to extinction. This vibration direction may be plotted in the same manner as was done for X, Y, and Z, using the readings of microscope stage (less the Reference Azimuth) and spindle arm. At exactly 90° on the microscope stage from this setting is another extinction position representing another vibration direction. On the plot this point falls just 90° from the first extinction position on the same meridian.

7. If a recognizable cleavage plane or crystal face (such as the 001 cleavage of table 1) is rotated into a vertical EW position, the pole of this plane is horizontal NS. Using the readings of microscope stage (less the Reference Azimuth) and the spindle arm at this position, the pole may be plotted as a point on the stereographic net, as done for the cleavage in figure 1. If desired, the plane itself may be shown by rotating the overlay to bring the pole to the equator, and tracing the meridian that lies 90° away.

8. In the foregoing the element has been plotted as it appears after having been rotated from the horizontal NS position, in which it was recognized, back to the primary reference position of the spindle. The same basic operations may be followed to plot an optic element that has been recognized in a position other than horizontal NS. Such is the case in plotting optic axes for determination of optic angle.

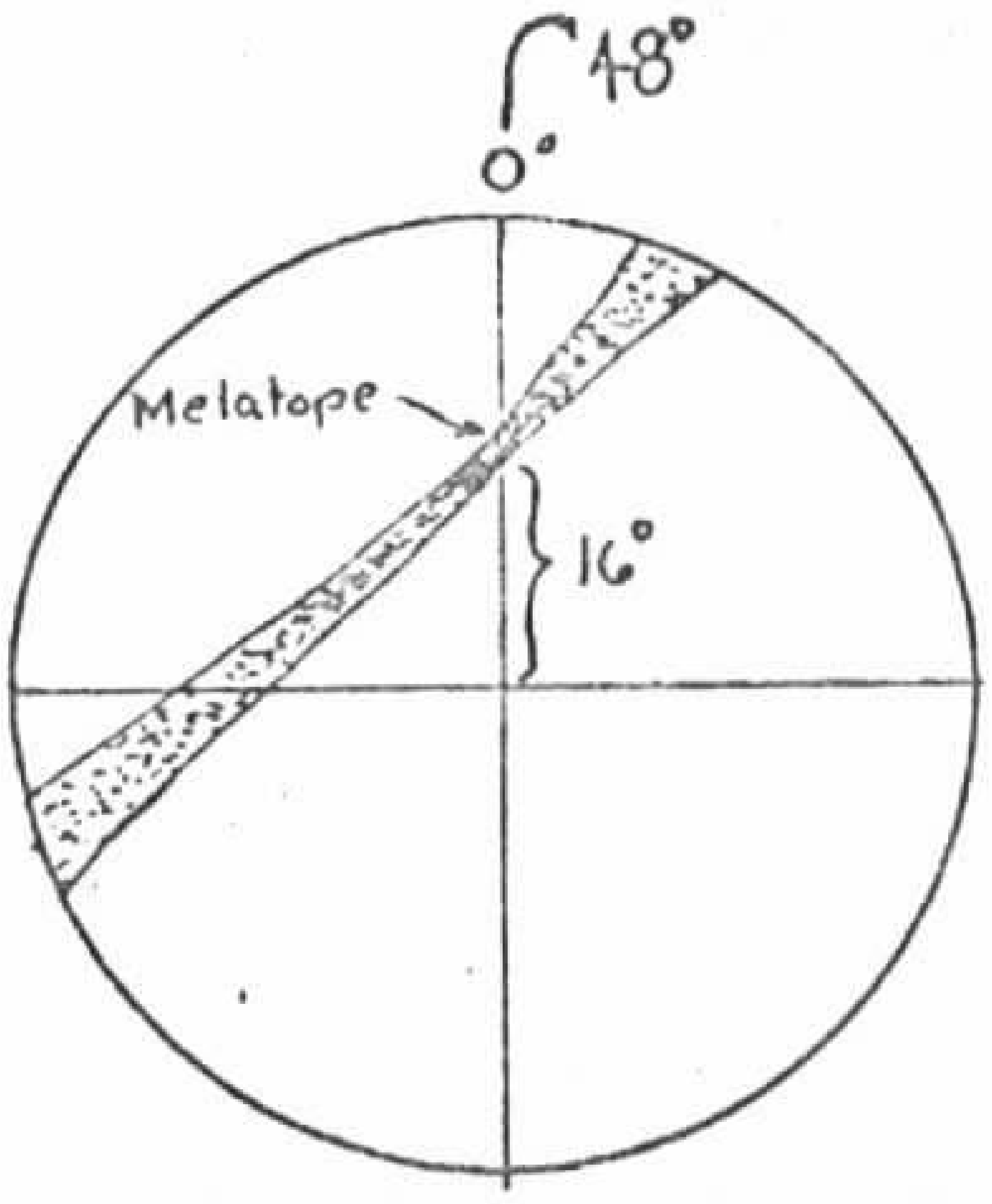


Figure 3.--Conoscopic image of optic axis on N-S crosshair, spindle arm setting at 48°, microscope stage at 0°, Bertrand lens inserted.

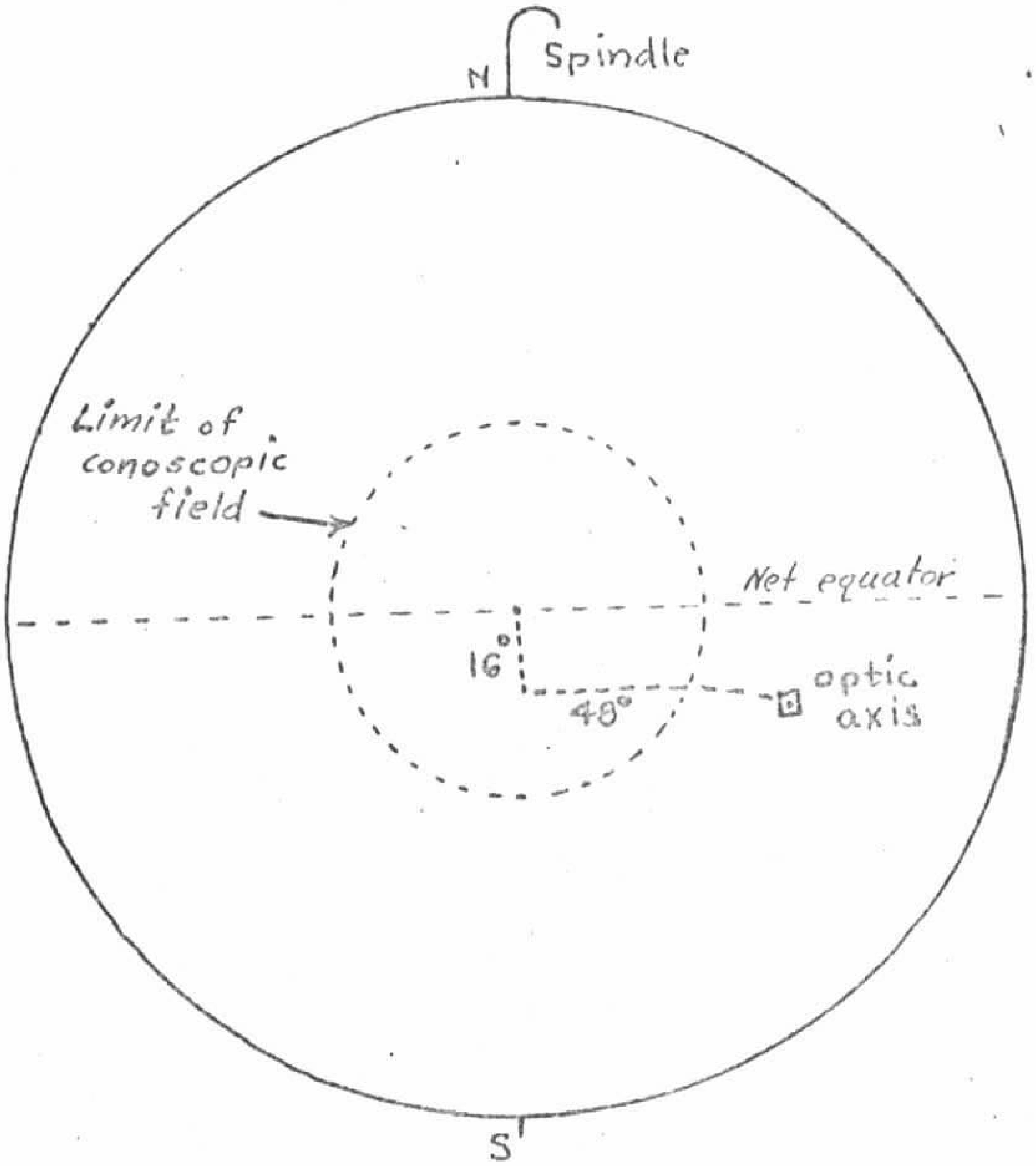


Figure 4.--Optic axis of figure 3 plotted on stereogram with spindle returned to reference position.

In figure 3 for instance, the melatope (optic axis) has been placed on the NS crosshair with the microscope stage at 0° and the spindle arm at 48°. With the ocular micrometer and Mallard's constant, the angular distance from the microscope axis was found to be 16°. (For this the approximate value of Ny should be known.) In plotting, start at the center of the net, as in figure 4, proceed 16° <u>south</u> (because the Bertrand lens inverts the conoscopic image), thence 48° eastward to mark the point of the optic axis.

9. Transposing the various crystal optical elements to a position different than that of the reference position is done in the usual manner, as outlined for the universal stage of Haff (1940, 1942). Thus a plot of a plagioclase X, Y, and Z, and (001) cleavage may be rotated to bring Y to the center and X and Z to the base circle, as in figure 5, for comparison of (001) with the conventional plagioclase migration curves (Troger 1956 Plate I; Emmons 1943, Plate 12).

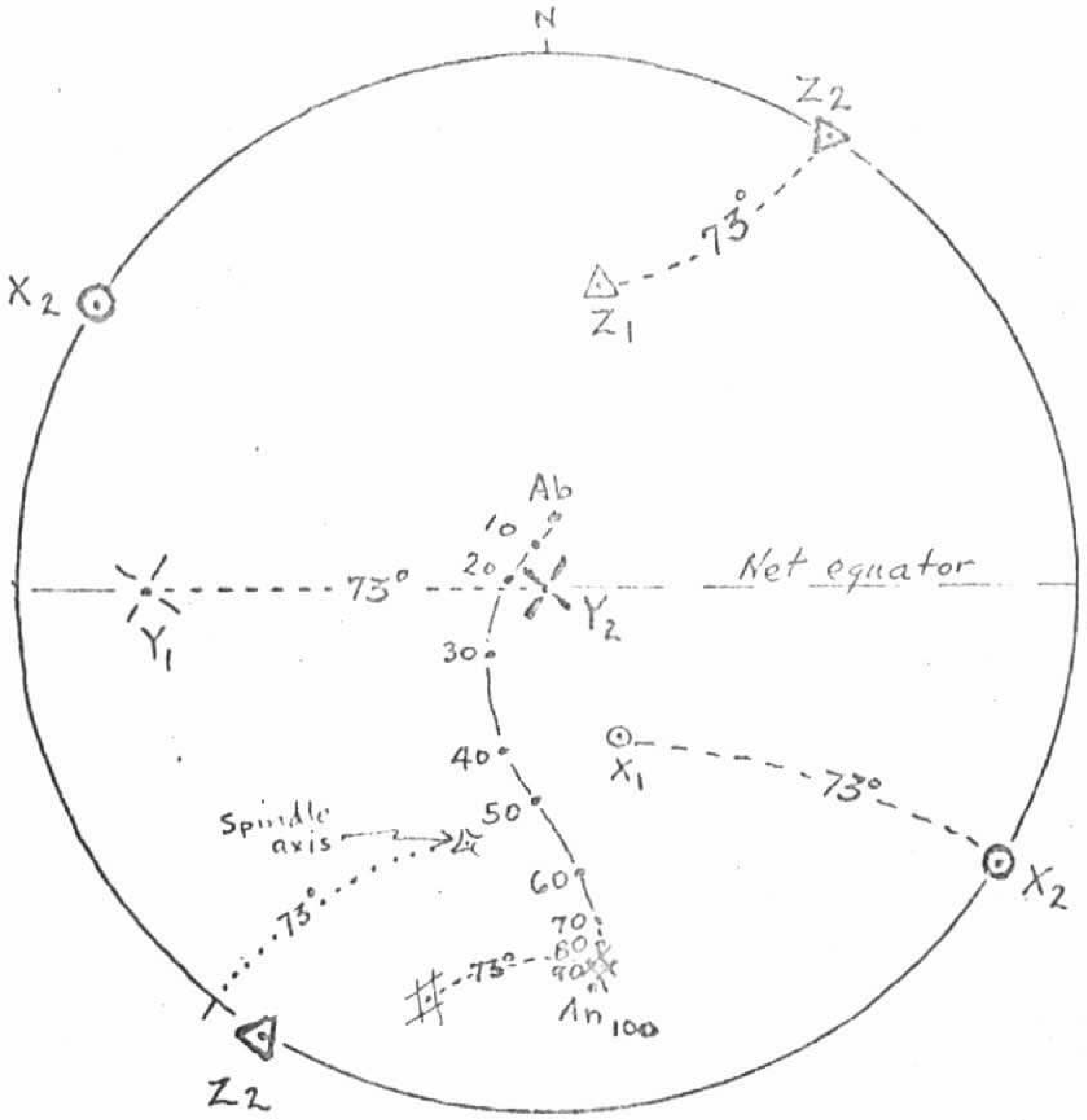


Figure 5.--(Plot of anorthite crystal (table 1) transposed to place Y at center for comparison of position of (001) with plagioclase migration curve.

CONOSCOPIC PROCEDURE

For a measurement of a principal refractive index, the corresponding symmetry axis of the indicatrix is placed horizontal and in the plane of the lower nicol (here assumed to be NS). This situation is expressed in conoscopic illumination as an interference figure bilaterally symmetrical about the EW crosshair. As illustrated in figure 6, the figure itself may take various forms, thin and well defined in the vicinity of the melatope (optic axis) and broader at a distance therefrom. In the latter case the final symmetrical setting is most easily recognized by the symmetrical movement of the isogyre in respect to the EW crosshair as the microscope is rotated a few degrees back and forth. The "flash figure" represents an extreme example of this behavior.

Interference figures are generally of better quality when there is at least an approximate match in index between liquid and crystal, say within 0.03. The best interference figure is not usually found at the setting of the microscope tube for best focus of the orthoscopic image. Rather, starting at this setting and inserting Bertrand lens and substage condenser, it will be found that by raising the tube slightly the interference figure smooths out and fills the conoscopic field without distortion.[1]

See footnote

Uniaxial crystals

~~A position of general utility for a uniaxial crystal is with its optic axis horizontal. This position is recognized conoscopically by the so-called "flash figure." On a good microscope, the setting at which the "flash figure" is centered can be made to within a degree or two of rotation about the spindle arm. The following procedure is suggested:~~

1. ~~The crystal under conoscopic illumination between crossed nicols is rotated on the microscope stage to bring one isogyre approximately along the N-S crosshair.~~

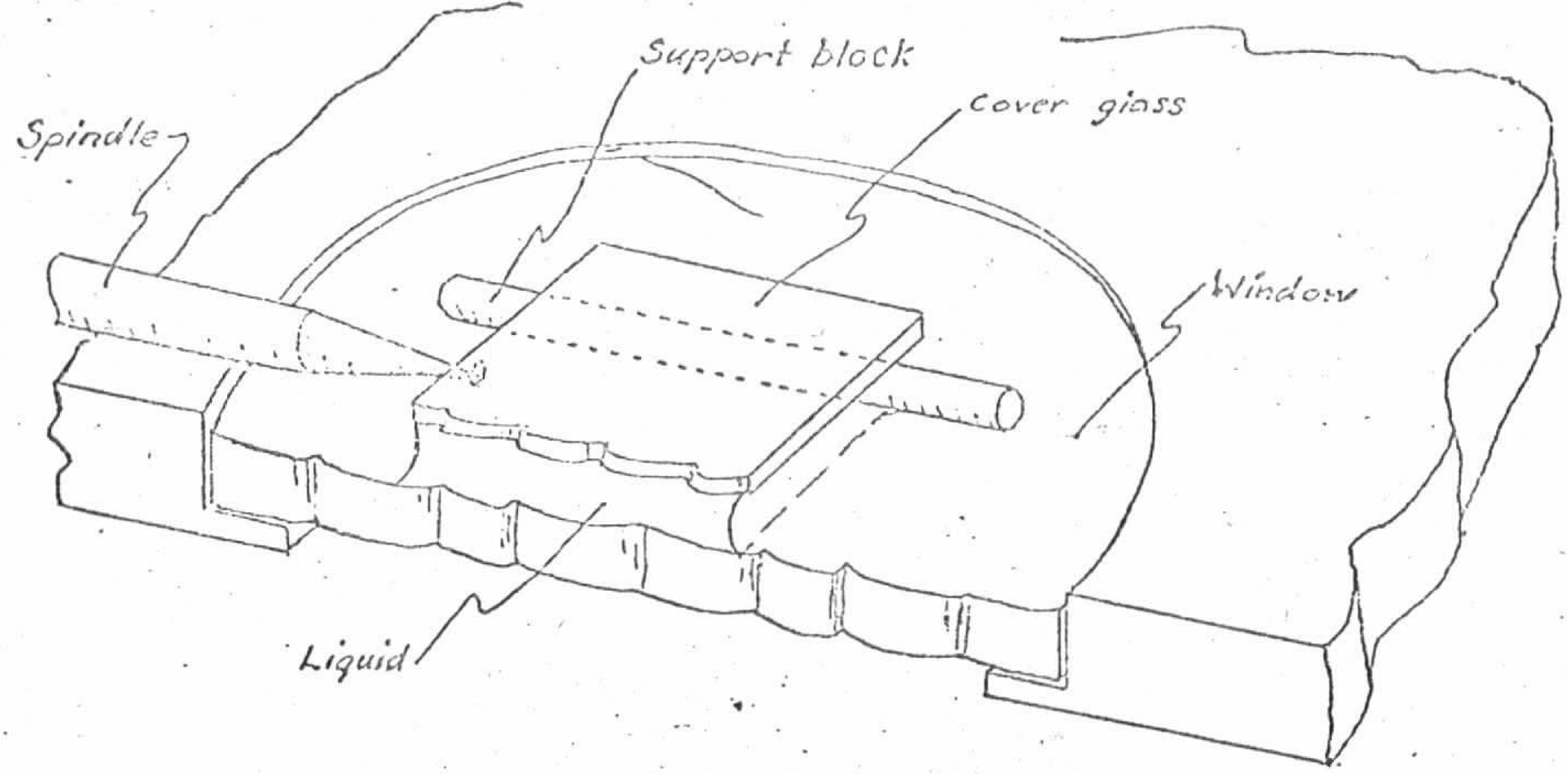
2. ~~Then by stepwise rotations about the spindle axis with required adjustment of the microscope stage to maintain the arm of the isogyre approximately along the N-S crosshair a point is reached at which the isogyre becomes noticeably broader and "fans" across the field as the microscope stage is rotated back-and-forth. With a small rotation about the spindle axis beyond this point, the isogyre is seen to broaden to nearly fill the whole field and, upon rotation back-and-forth of the microscope stage, it flashes out of field first in one opposed pair of quadrants, then in the other. In this position the optic axis is horizontal.~~

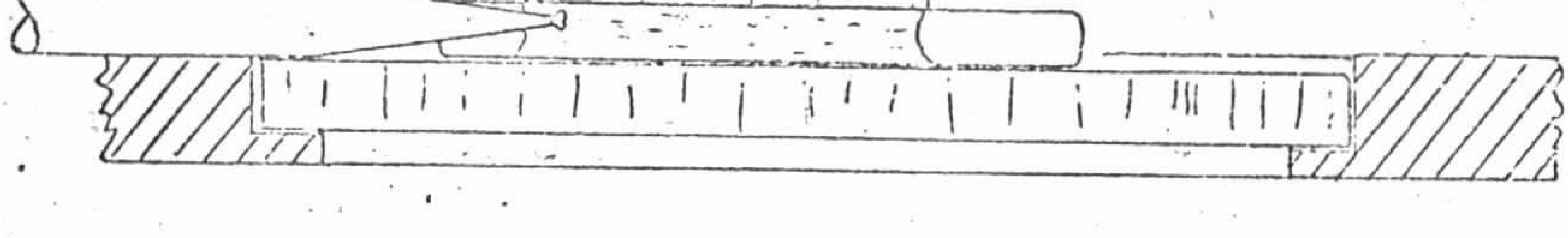
3. ~~It is good practice to establish this setting definitely by rotating about the spindle axis a bit further, until the "fan" action of the isogyre is again seen but on the other side. The crystal is then returned to the best setting for the flash figure.~~

(footnote, p. 8)
-end of paragr. 2.-

// Note that for the high-N.A. objective used here an orthoscopic image of the crystal fragment is not actually required, only the conoscopic interference figure, which in many cases may still be obtained even though the objective's working distance is too small to permit an orthoscopic image. The tests with the auxiliary plate for Fast and Slow directions in the conoscopically oriented fragment may be made by slightly raising the tube and refocussing the Bertrand lens, thereby providing a telescopic image of the whole fragment.

Should the working distance of the objective be too small to permit a full conoscopic figure, it may be necessary to modify the spindle stage. One expedient is to substitute support blocks of a smaller diameter than the spindle, as shown in the sketch, permitting a closer approach of the objective to the crystal fragment. It is to be noted that the advantage gained thereby is greater than would be the case if *both* the spindle diameter and the support diameter were reduced by this amount.





insert 11/15/71
(Substitute for
p. 8 - bottom
p. 9 - top)

Uniaxial crystals

The most direct way to determine whether a crystal is uniaxial is to orient it so that the optic axis is within the conoscopic field of view. The interference figure then is a dark cross, figure 5a, with arms that remain parallel to the crosshairs as the stage is rotated. Here the optic sign may be determined by noting the change of interference colors in the quadrants upon insertion of the accessory plate (cf. Bloss 1960, p. 127; Wahlstrom 1969, p. 260). Here also the ordinary principal refractive index could be compared with the index of the liquid by rotating the stage to place the intersection of the dark cross on the E-W crosshair, converting to orthoscopic illumination, and withdrawing the upper nicol. It is just as well, however, to go directly to the flash figure, as outlined in the following, where both the ordinary and extraordinary principal indices may be determined.

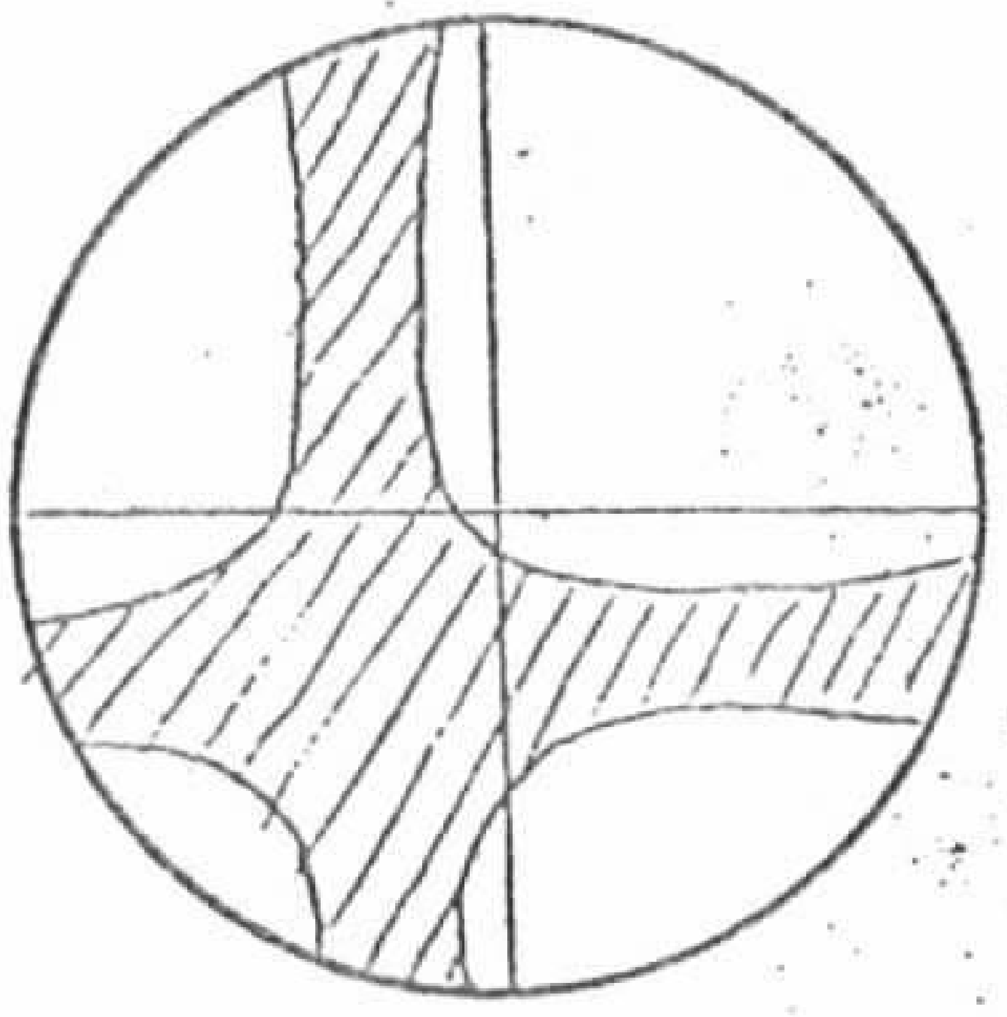


Figure 5a.

1. Rotate the microscope stage to place the E-W isogyre along the E-W crosshair.

2. By alternate small rotations about the spindle axis and adjustments of the microscope stage to keep the isogyre approximately along the E-W crosshair, proceed through the position in which the isogyre noticeably broadens and "fans" rapidly across ~~the field~~ with small rotation of the microscope stage. With a small spindle rotation beyond this position the isogyre is seen to broaden to nearly fill the whole field, and, upon rotation of the microscope stage back and forth, it flashes out of the field first from one opposed pair of quadrants, then from the other. This is the so-called "flash figure" and indicates that the optic axis is horizontal.

3. It is good practice to establish this setting firmly by rotating a bit further about the spindle axis until a reversed "fan" action of the isogyre is observed. The crystal is then returned to the setting that gives the best and most symmetrically-behaved flash figure.

4. Having followed the E-W isogyre to the flash figure, we know that the optic axis is not only horizontal, but it is E-W. Thus the vibration direction of the ordinary ray is N-S parallel to the lower nicol, and its index may be compared to that of the liquid by converting to orthoscopic illumination and withdrawing the upper nicol.

5. Rotation of 90° on the microscope stage (to the other extinction position) places the optic axis, N-S, parallel to the lower nicol, and, with the upper nicol withdrawn, the crystal is in position for comparison of the extraordinary principal index with the immersion liquid.

~~4. Knowing now that the optic axis is horizontal, we proceed to place it NS with the aid of Lommel's Rule:~~

~~A. From an extinction position, rotate the crystal a few degrees clockwise. If in doing so the isogyres depart from the NE and SW quadrants, the optic axis has been rotated into the NE quadrant. It can now be rotated the few degrees back to the NS position for determination of the extraordinary index. Rotation to the other extinction position enables determination of the ordinary index.~~

~~B. If on the other hand the clockwise rotation causes the isogyre to flash out of the field in the NW and SE quadrants, the optic axis has been rotated a few degrees into the NW quadrant. It now can be placed NS by appropriate rotation on the microscope stage.~~

Biaxial crystals

A principal vibration direction is horizontal NS when an isogyre lies EW across the center of the conoscopic field, so that the EW crosshair divides the isogyre into two mirror-image halves, (for instance, as in figure 6 a, b, and c). Such a bilateral symmetry of the isogyre results from the fact that a symmetry plane of the indicatrix is exactly vertical and EW. With the principal vibration direction now horizontal NS, the corresponding principal index may be measured (see section on refractive indices) or the readings taken of the microscope stage and spindle arm scales for plotting on the stereographic net (see sections on plotting, optic angle, etc.)

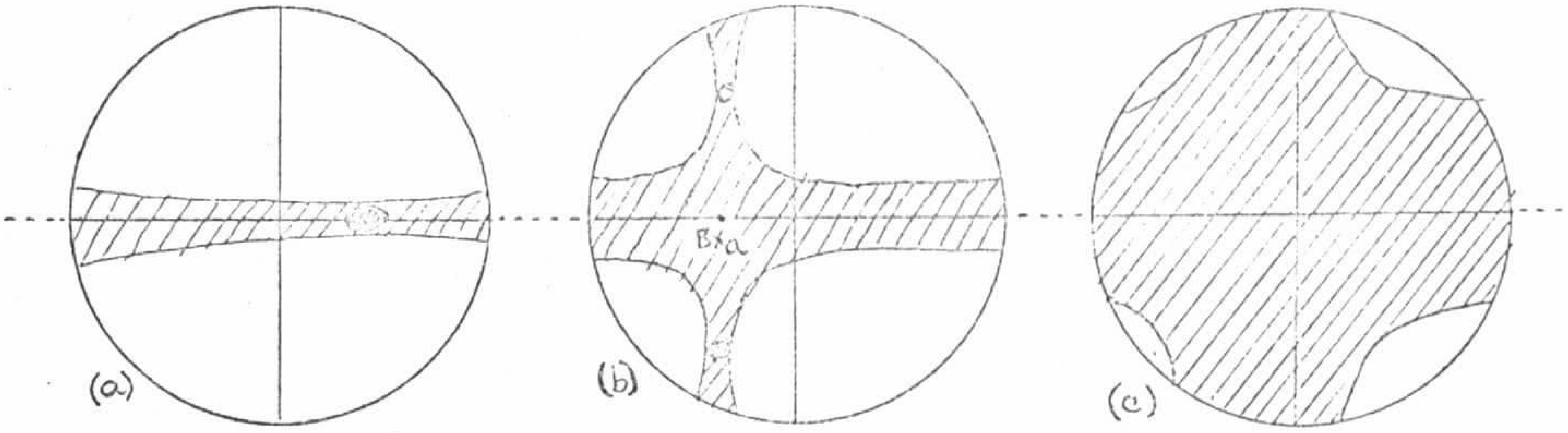


Figure 6.--Examples of interference figures of crystals oriented with one principal vibration direction horizontal NS:
(a) Y horizontal NS (optic plane EW)
(b) A bisectrix horizontal NS, other bisectrix near vertical.
(c) A bisectrix horizontal NS, other bisectrix near horizontal EW.

While any of the three principal directions X, Y, or Z may be oriented first, there are certain advantages in starting with Y, the optic normal, as follows:

1. Make sure that the microscope system, including the Bertrand lens is centered. (Determine the Reference Azimuth if data is to be plotted.)

2. With the Bertrand lens, substage condenser and upper nicol inserted, rotate through the sweep of the spindle arm to see if an optic axis passes through or near enough to the conoscopic field to recognize its position. (The isogyre pivots about the optic axis as the microscope stage is rotated.)

3. Place the isogyre containing the optic axis symmetrically along the EW crosshair, for instance as in figure 7a. The symmetry plane of the indicatrix thus placed vertical and EW must be the optic plane, since it contains the optic axis. The optic normal Y is now in the desired horizontal NS position for determination of N_y.

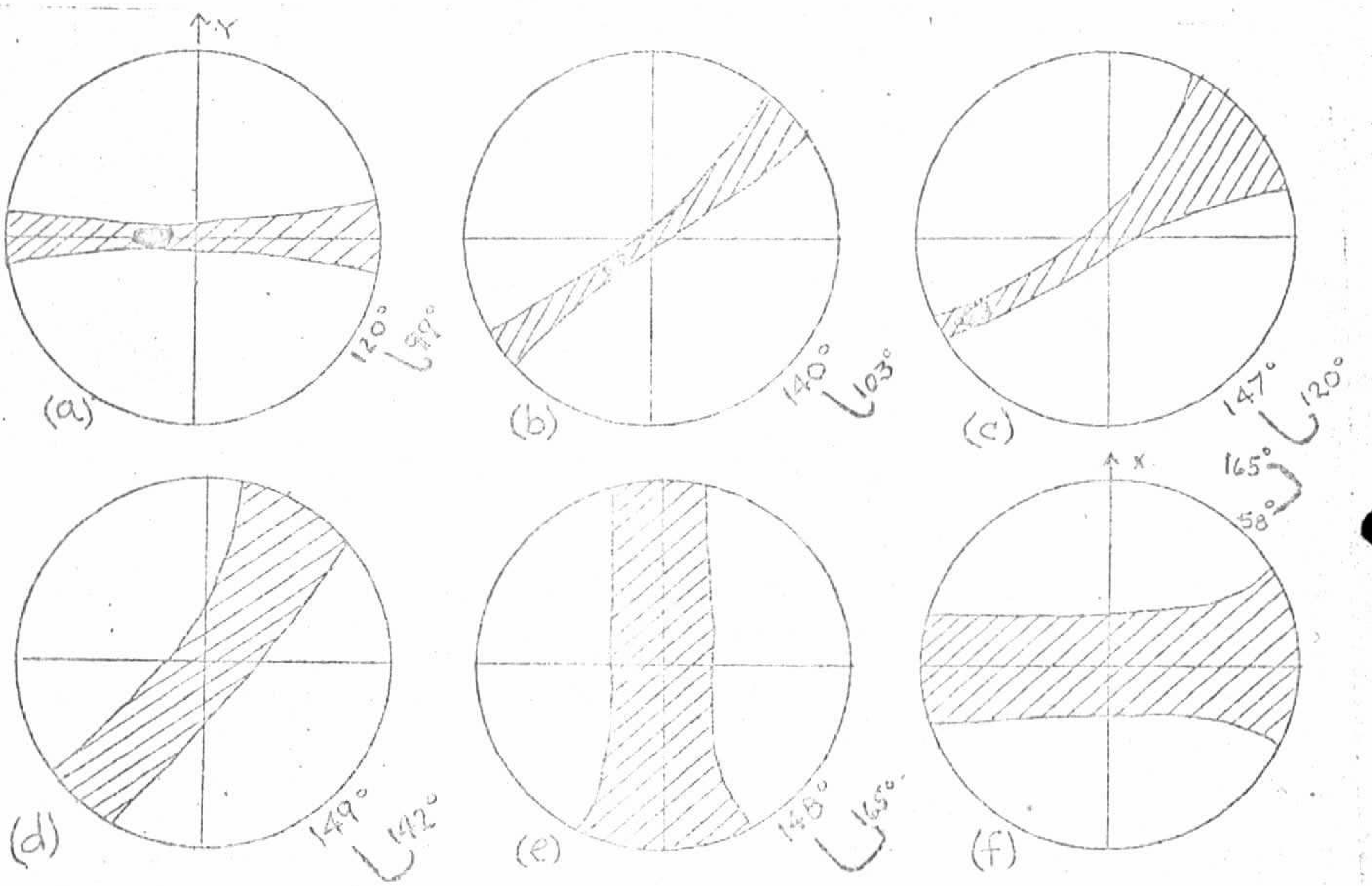


Figure 7.--Example of sequence of interference figures (with Bertrand lens) seen as isogyre is followed from setting for Y to that of X of an anorthite crystal. $2V_x$=78°.

4. If the optic axis lies well within the conoscopic field, rotate microscope stage to the 45-degree position and determine optic sign and approximate optic angle by the curvature of the isogyre. Likewise note dispersion of optic axes, if present. Return to the EW setting of the isogyre. (Detection of curvature of the isogyre becomes less reliable the further the optic axis from a central position in the conoscopic field.)

5. Follow the isogyre by small increments of rotation on the spindle axis and necessary rotations of the microscope stage to swing the isogyre (no longer EW) back to cut through the center of the field, for instance as done in figures 7, a to f. Eventually a new position is reached where the isogyre lies symmetrically along either the EW or NS crosshair.

   A. If the isogyre now lies along the EW crosshair, a symmetry plane is vertical EW and another principal vibration direction has been placed horizontal NS, in position for determination of the principal refractive index. The direction cannot be Y, since that was already found in a different position, so it must be X or Z. Determine which by the usual test with the accessory plate, and record readings of the microscope stage and spindle arm.

   B. If the isogyre lies along the NS crosshair, rotate the microscope stage 90°, to make the isogyre lie along the EW corsshair, and refine the settings of spindle arm and microscope stage to make the EW crosshair truly bisect the isogyre. Determine by the usual test with the accessory plate whether X or Z is the vibration direction now horizontal NS. Record readings.

   C. Since we now have two of the principal vibration directions, we could find the third at exactly 90° from both by plotting on the net. It is generally good practice, however, to proceed to find the third principal direction independently, as in Step 6, below.

6. By additional incremental rotations on spindle and microscope axes, follow the isogyre further until still another setting is found where it is either EW or NS. If EW, treat as in Step 3A, if NS treat as in Step 3B. This is the third principal vibration direction.

   If upon reaching the end of swing of the spindle arm (either 0° or 180°) no further EW or NS isogyre has been encountered, return to the setting for Y and follow the arm of the isogyre by incremental rotations about the spindle axis in the opposite direction until the sought-for EW or NS isogyre is found.

In the majority of cases the procedure outlined above will lead more rapidly to the settings for the three principal vibration directions than a random search, and will involve least equivocation in the identification of each direction as X, Y, or Z. When by chance a centered bisectrix figure is encountered, one extinction position furnishes Y and rotation of 90° on the microscope stage to the other extinction position furnishes X or Z without changing the setting of the spindle arm. When a truly centered optic normal figure is encountered, one extinction position furnishes X and the other Z.

Special attention should be given, however, to the apparently centered optic normal (flash) figure. Precautions are necessary in the case of a crystal of small optic angle, say less than 40°, because the behavior of such a crystal may approach that of a uniaxial crystal ($2V = 0°$), and the apparent flash figure indicates only that the acute bisectrix is horizontal; the optic plane itself may be inclined. Fortunately the position of the acute bisectrix may be established by applying Lommel's Rule as for uniaxial crystals (see above for uniaxial crystals) and the optic sign may then be determined with the aid of the accessory plate. Should the crystal happen to be mounted with the acute bisectrix sub-parallel to the spindle axis, the correct horizontal NS settings for the obtuse bisectrix and the optic normal may not be ~~as~~ apparent to the untrained eye, and for determination of their respective principal indices it may be more effective to revert to the orthoscopic procedure outlined in another section. No difficulty in choice of proper setting occurs when the acute bisectrix is at a large angle to the spindle axis, for it is seen in the conoscopic field at appropriate settings of the spindle arm.

Conoscopic "extinction curves" (cf. also "extinction curves" in "Orthoscopic Procedures" below).

Since the interference figure is a "directions image" the center of the conoscopic field displays the behavior of the light passing through the crystal parallel to the microscope axis. Thus a crystal at extinction under orthoscopic illumination will furnish an interference figure in which an isogyre passes through the intersection of the crosshairs. Successive extinction positions at different settings of the spindle arm may be followed conoscopically as follows:

1. Make sure that the microscope system, including the Bertrand, is centered. Determine Reference Azimuth.

2. Rotate on the microscope stage to place the spindle axis EW. Advance the spindle arm until the darkest medial portion of an isogyre lies at the intersection of the crosshairs. (The isogyre may be found to lie in any direction across the field.) Record spindle arm and microscope stage reading.

3. Rotate spindle arm to the nearest 10° graduation on scale and rotate the microscope stage to bring this same isogyre back to the intersection of the crosshairs. Record readings.

4. Similarly record microscope stage readings for the isogyre passing through center of field at successive settings of the spindle arm at 10° intervals through the full 180°. (For those zones where the isogyre is swinging rapidly--where the optic axis is near the center of the field--advance the spindle arm by smaller amounts, say 5° or less.) This defines the so-called "equatorial" extinction curve, described in detail in the section on orthoscopic procedures.

5. Plot these points on an overlay on the stereonet (see section on "Stereographic Plotting").

6. Another extinction curve is defined by the series of "conoscopic" extinction positions 90° away on the microscope stage from each of the settings determined in Steps 1-3 above. This is the so-called "polar" extinction curve, and readings may be plotted similarly.

It is instructive and often helpful to compare the extinction positions taken alternately under conoscopic and orthoscopic illumination. The conoscopic generally provides the more precise setting of extinction when an optic axis is near the center of the field. The orthoscopic setting may be the more sharply defined when a bisectrix is near horizontal. The fundamental fact that the principal vibration directions X, Y, and Z, lie on the continuous extinction curves is simply demonstrated by tracing the extinction curve conoscopically through each of the three settings at which a principal direction is horizontal NS, as indicated by the symmetrical disposal of the isogyre along the EW crosshair.

ORTHOSCOPIC PROCEDURES

The orthoscopic procedures outlined here are useful as a supplement to the above described conoscopic procedures. They are not recommended for use to the exclusion of the conoscopic procedures, except where the microscope at hand is not capable of producing a satisfactory interference figure. Orthoscopic procedures in some applications are capable of high precision, but generally their usefulness is limited because at any one setting the optical behavior of light is seen for only one direction of transmission in the crystal. (The conoscopic procedure, on the other hand, presents to the observer the behavior of light in a large cone of directions, and he is better able, for instance, to bring a desired element into orientation, or to make a quick estimate of optic angle, optic sign, etc.)

The methods described in this section are based on extinction behavior between crossed nicols under orthoscopic illumination. For determination of the spacial relations of principal optical directions and the optic angle of the crystal, readings will be taken of the settings of microscope stage and spindle arm at selected extinction positions, and these will be plotted according to the conventions described in the section on "Stereographic Plotting" above.

Extinction positions

Between crossed nicols with a given setting of the spindle arm, rotation on the microscope stage brings the crystal to a position of extinction. Here one vibration direction of light passing through the crystal is horizontal NS, and this directional element can be plotted stereographically in the same manner as described in the previous section using the readings of microscope stage and spindle arm. Rotation of exactly 90° on the microscope stage brings the other vibration direction NS, and its position may similarly be plotted.

Although there are four such extinction positions in a full 360° rotation of the microscope stage, we will limit ourselves here to the two which fall in the range 0° to 180° on the microscope stage. The other two simply duplicate the first two for our purposes, and to prevent confusion we avoid them.[1]

For purpose of plotting the extinction curves described below and for their further use in determination of optic angle, the extinction positions should be determined as exactly as feasible. The microscope lens system first of all should be in adjustment and the vibration directions of the nicols strictly parallel to the crosshairs. Because the spindle arm cannot be set much more precisely than ± 1° using the crude plastic scale

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[1] Because of the construction of the spindle arm, we automatically avoid the four additional replicate extinction positions for a setting of the spindle arm just 180° from the first setting.

on current models (Wilcox 1959), it will not be necessary to determine its setting at extinction closer than say ± 0.5°. This amount of tolerance is permitted also by the reliability of the stereographic net--most currently available nets are no better than ± 1°. Where justified by the requirements of the problem and the precision of equipment and plotting net, methods are available for more exact determinations of extinction position (cf. Wright, 1911, p. 115-146).

Under routine orthoscopic illumination the crystal fragment may appear to remain at extinction, as far as the eye can discern, over a rotation of several degrees on the microscope stage. This is because the minimum of light intensity is not sharp as one approaches and passes the position of true extinction. It is good practice to constrict somewhat the field diaphragm (the lower of the two substage diaphragms), whereby the illuminating rays are made more nearly parallel and a sharper setting for extinction is obtained, in spite of the lower intensity of total illumination.

White light suffices for most routine determinations of extinction position. With crystals of strong dispersion of the optic axes or the indicatrix, however, definite extinction in some positions is only to be attained in monochromatic light. As a source of monochromatic light a sodium lamp (5893Å) may be used or, a corresponding interference filter (Schott-Jena, Baird-Atomic) may be placed in the path of white light.

If an optic axis is near parallelism with the microscope axis, the position of true extinction, that is of minimum light, is even less sharp. The crystal may appear to be dark through a wide rotation of the microscope stage, and worse yet, it may not attain total extinction at any position. Acceptable results may sometimes be obtained by still further restriction of the field diaphragm, but in these circumstances it may be helpful to revert to conoscopic illumination, where, with the aid of the isogyre, there is no doubt of the true position of extinction (see section above on "conoscopic extinction curves").

As the rotation about the spindle axis proceeds the crystal fragment may depart from the center (crosshair intersection), because the fragment has not been mounted exactly at the rotational axis. Small departures of this type are not harmful in orthoscopic work. Large departures require recentering of the grain without disturbing the Reference Azimuth.

Extinction curves

Having established one position of extinction, a small change in the setting of the spindle arm will normally cause the crystal to depart from extinction, and a small rotation of the microscope stage will be required to restore extinction. By successive incremental advances of the spindle arm and restoration of extinction by rotation of the microscope stage a continuous series of extinction positions may be followed, corresponding to a progressive change of vibration direction as the crystal is rotated about the spindle as a zone axis. Plotted on the stereographic diagram, these define an "extinction curve." Since for each setting of the spindle arm there is another extinction position at just 90° rotation on the microscope stage, there is a second extinction curve representing a second series of vibration directions. One curve crosses the equator of the net and is called the equatorial curve; the other passes through the pole of the net -- the spindle axis -- and is called the polar curve (see figures 8 and 9). The properties and mathematical relations of extinction curves are described in some detail by Joel and Garaycochea (1957), and their use in the determination of optic angle are discussed by Wilcox (1960), Tocher (1962, 1963), and Garaycochea and Wittke (1964).

Uniaxial crystals

The equatorial extinction curve of an uniaxial crystal mounted with its optic axis oblique to the spindle axis is a great circle. The normal to the plane of the great circle of the equatorial extinction curve is the optic axis and lies on the polar extinction curve. The following simple means of locating the optic axis is available and is illustrated by figure 8, based on the data of table 3:

1. Check adjustment of microscope and determine Reference Azimuth of spindle. Immerse crystal in liquid somewhere near its average refractive index.

2. Rotate microscope stage to place spindle axis EW, then rotate about spindle axis to bring crystal to extinction. Record this as one point on the equatorial curve.

3. Follow this extinction curve by stepwise rotations of spindle axis and restorations of extinction on microscope stage, until spindle arm is at 0° (flat). Carefully set extinction on microscope stage and record.

4. Return to first setting (Step 2) and follow extinction curve similarly in opposite direction until spindle arm reaches 180°. Carefully set extinction on microscope stage and record.

5. Theoretically these three points should establish the great circle of the equatorial curve. In practice it is desirable to establish several more points along the curve, especially in the middle portions, and record the readings.

6. Plot the points stereographically on an overlay taking into account the Reference Azimuth. (See section above on "Stereographic Plotting").

7. Rotate overlay to bring the points to that meridianal great circle on the net which best fits the plotted points. Trace the great circle.

Table 3.--Data for orthoscopic orientation of a uniaxial crystal

Mineral: Quartz

Ref. Azimuth 0°
Liquid 1.550

Spindle arm reading	Microscope stage reading	
	Equatorial	Polar
0°	62°	152°
40°	90°	180°
80°	119°	29°
140°	129 1/2°	39 1/2°
180°	118°	28°

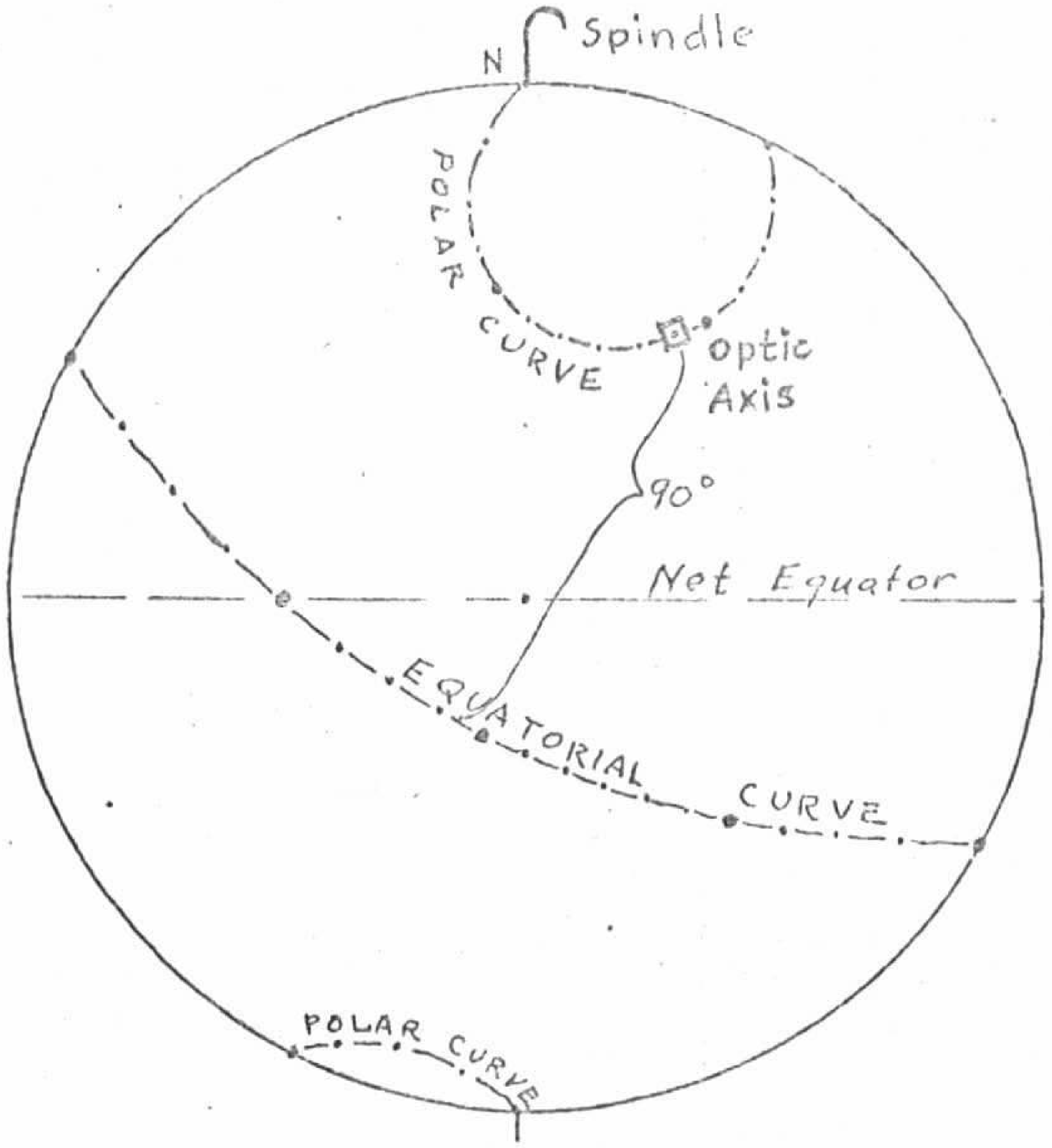


Figure 8.--Extinction curves of a uniaxial crystal mounted with its optic axis at 40° to spindle axis (table 2).

8. From the intersection of this great circle with the equator, count off 90° along the equator and mark a point. This is the optic axis.

9. Rotate the overlay to place the spindle axis again at the north point. If it is desired to set the optic axis horizontal NS, the readings of microscope stage and spindle arm may be taken from the net.

Biaxial crystals

The equatorial extinction curve of a biaxial crystal departs from a great circle by an amount depending on the size of the optic angle and the orientation of the crystal indicatrix in respect to the spindle axis. The optic normal, Y, and one of the bisectrices, X or Z, lie on the equatorial curve; the other bisectrix lies on the polar curve. These relations, plus the fact that X, Y, and Z are 90° from one another, make it possible to find the actual positions of X, Y, and Z on the stereographic plot as follows:

1. Check adjustment of microscope. Determine Reference Azimuth of stage. Immerse crystal in liquid somewhere near its average refractive index.

2. Between crossed nicols with orthoscopic illumination, rotate on microscope stage to place spindle axis EW. Rotate about spindle axis to extinction. This is a point on the equatorial curve.

3. Trace out this curve in both directions, setting the spindle arm carefully at even 10° intervals and the microscope stage carefully at the best extinction of the crystal for each setting. Record readings as in table 3.

4. After subtracting the Reference Azimuth from each reading of the microscope stage, plot the readings on an overlay on a net (see "Stereographic Plotting" above), and draw a smooth curve through the points. This is the equatorial curve.

5. Rather than physically determining the corresponding polar curve, it is usually sufficient to mark a point at exactly 90° along the meridian from each plotted point and draw a smooth curve.

6. Plot approximate positions of Y and the two bisectrices, (estimated conoscopically or by other means). If Y does not fall exactly on the equatorial curve, shift it to the curve, as in figure 9.

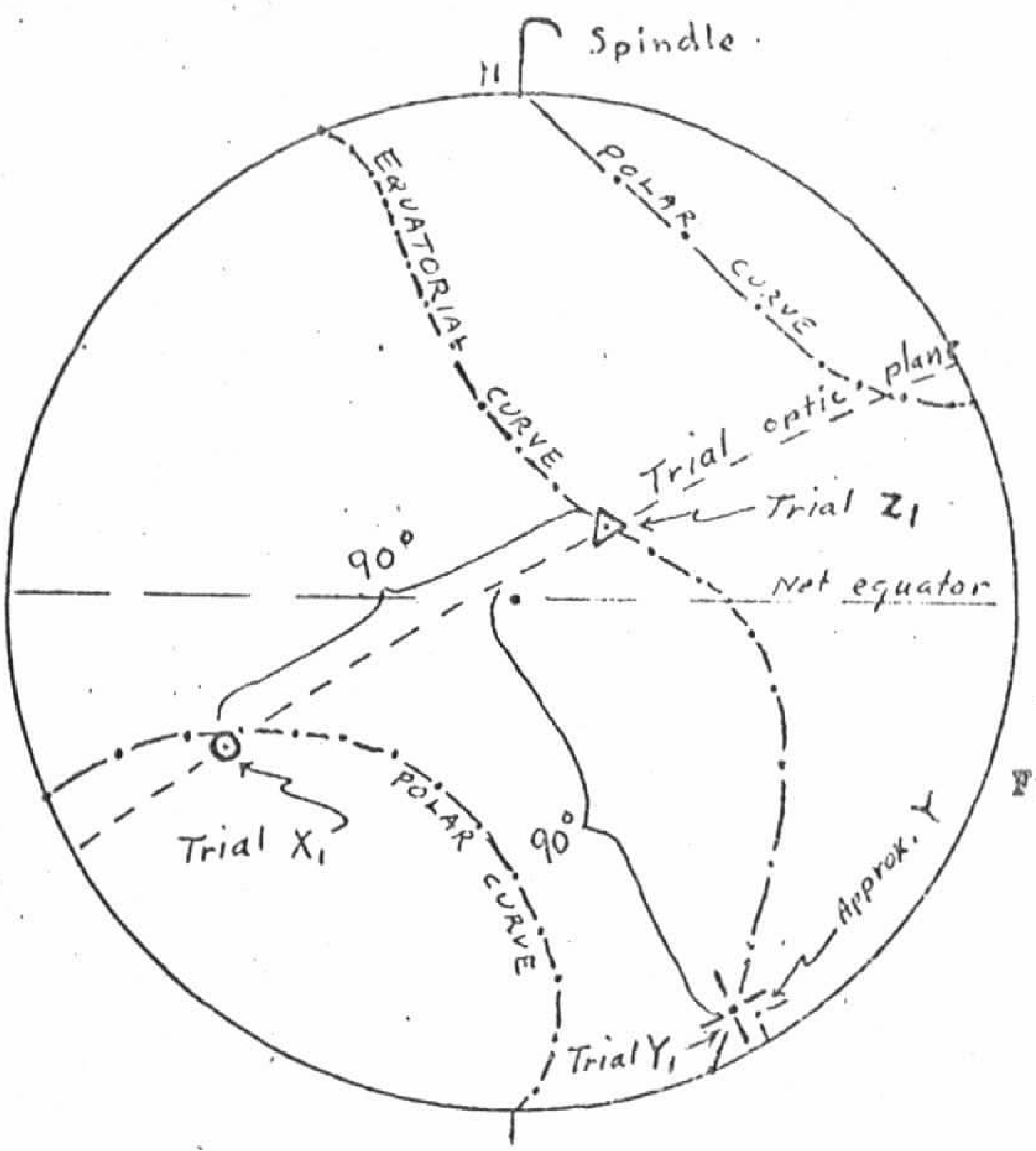


Figure 9.--Trial position of 90° spherical triangle to fit extinction curves (data of table 3).

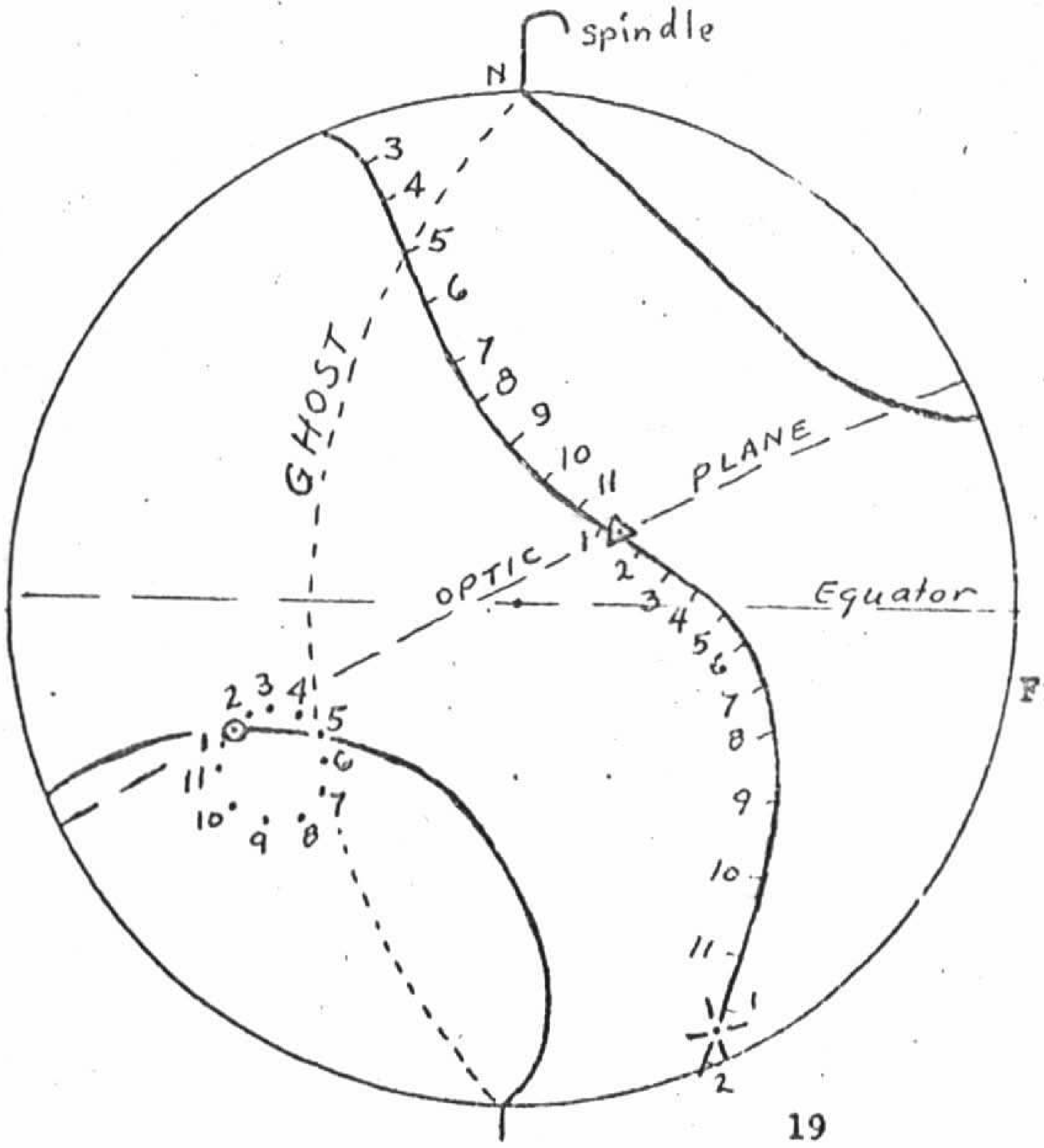


Figure 10.--Complete sequence of trial positions of 90° spherical triangle. True XYZ triangle lies between trial sets 1 and 2.

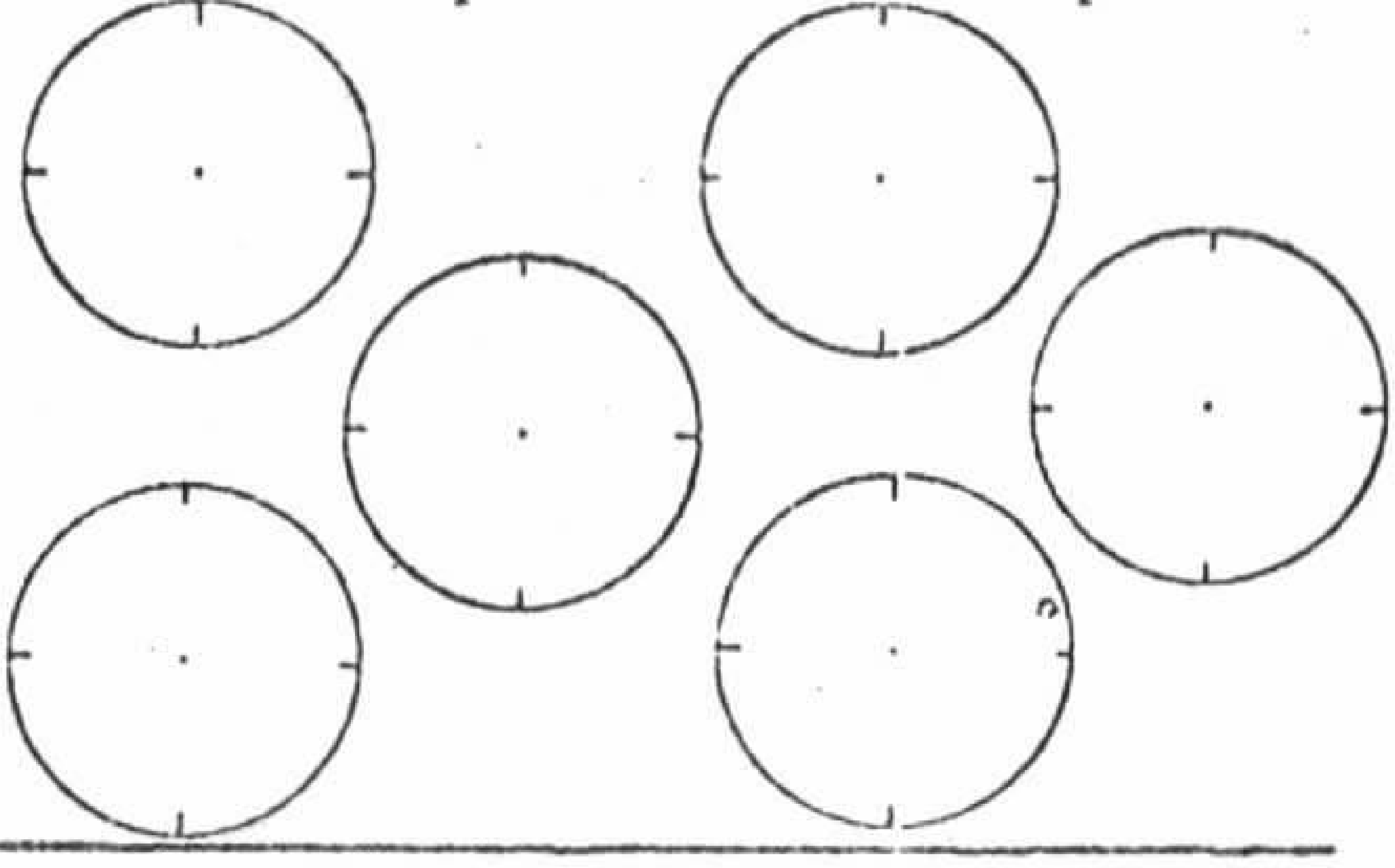
TABLE 3 Data sheet for SPINDLE STAGE

MINERAL Orthopyroxene LOCALITY Bow Lake, Wash. SPEC. No. Cr-1-60

REFR. INDICES & COLOR:

		spin.arm	mic.stage
N_x	1.685-1.687 (range)	30° * (30)	110° (112)
N_y	1.692-1.694 (ranges)	172 (171)	149 (153)
N_z	1.701	112 (113)	76 (75)

orthoscopic conoscopic



FACES & CLEAVAGES:

* numbers in parentheses are positions of X, Y & Z adjusted on basis of extinction curves.

OPTIC ANGLE:

2Vdirect

2V indirect (-) 66° ± 2° (extinction curves)

2V calc

EXTINCTIONS: Liquid n= 1.69
Ref. Az. = 0°

spin.arm	mic.stage Equat.	Polar
0°	23°	113°
10°	21	111
20°	21	111
30°	22	112
40°	24	114
50°	27	117
60°	32	122
70°	38	128
75	43	133
80°	47	137
85	52	142
90°	56	146
95	61	151
100°	66	156
110°	72	162
120°	79	169
130°	85	175
135	89	179
140°	95	5
145	105	15
150°	117	27
155	132	42
160°	143	53
165	148	58
170°	152	62
180°	[illegible]	[illegible]

REW
10/22/60

7. Rotate the overlay to place the adjusted position of Y on the net equator, count off 90° along equator and follow meridian to its intersection with the equatorial curve. This is a trial point for one bisectrix (point Z_1 in figure 9).

8. Count off 90° along the meridian and mark the trial point of the other bisectrix. If it falls on the polar curve, the requirement that X, Y, and Z be exactly 90° from each other is satisfied.

9. In only two positions of the 90° spherical triangle can its apices all lie on the extinction curves. One position is that of the true XYZ triangle. The other is that of a "ghost" triangle that may be identified by the fact that one of its sides extended passes through the spindle axis on the overlay (cf. Joel and Caraycochea, 1957).

10. If, as for point X, of figure 9, the trial point does not fall on the polar curve (or if it does but is an apex of the "ghost" triangle) it will be necessary to try another position of the 90° spherical triangle. Mark a new trial position of Y on the equatorial curve a few degrees away from the first and repeat Steps 8 to 10.

11. If the third apex is now closer to the polar curve than before, try yet other positions for Y in the same direction until all three apices lie on the extinction curves and the triangle is not the "ghost", as above defined. This then is the true XYZ triangle.

12. If the third apex is farther from the polar curve, try other positions for Y in the opposite direction along the equatorial curve to finally reach the true position of the XYZ triangle. The complete locus of trial positions is shown in figure 10, where the true positions of X, Y, and Z fall between trial sets 1 and 2.

13. The choice of which apex is X, Y, or Z is more readily made conoscopically. If conoscopic facilities are not available, it will be necessary to identify the principal directions orthoscopically as follows:

    A. The principal direction that has been located on the polar curve is either X or Z. Taking the stage and spindle arm readings as indicated by its plotted position, set this bisectrix horizontal NS. If by use of the accessory plate it turns out to be the faster ray, it is X; if the slower it is Z. Record.

B. The two principal directions on the equatorial curve are Y and the remaining bisectrix, and the most direct means of distinguishing them orthoscopically is by actually determining their indices of refraction:

Set one of the principal directions in question horizontal NS and change liquids until an index match is obtained. Now follow the equatorial extinction curve by appropriate rotations toward the position of other principal direction and observe whether the index increases or decreases. If, for instance, the bisectrix on the polar curve had been found to be X and now upon following along the equatorial curve the index is seen to increase, we must have started at Y on the equatorial curve and be proceeding towards Z. Label as such.

CHANGING IMMERSION LIQUIDS in the spindle stage

It is not necessary to disturb the orientation of the crystal during changing of liquids, but it is essential to take appropriate care to prevent contamination of either the new liquid in the cell or the supply bottle of new liquid. The following procedure is recommended:

(1) The old liquid may be largely removed by placing the end of a small strip of blotter or coarse filter paper against the spindle immediately behind the immersion cell. The removal may be hastened by another strip on the opposite side of the spindle, as in fig. 1. In a similar manner any stray liquid on the plate in front of the cell is similarly removed.

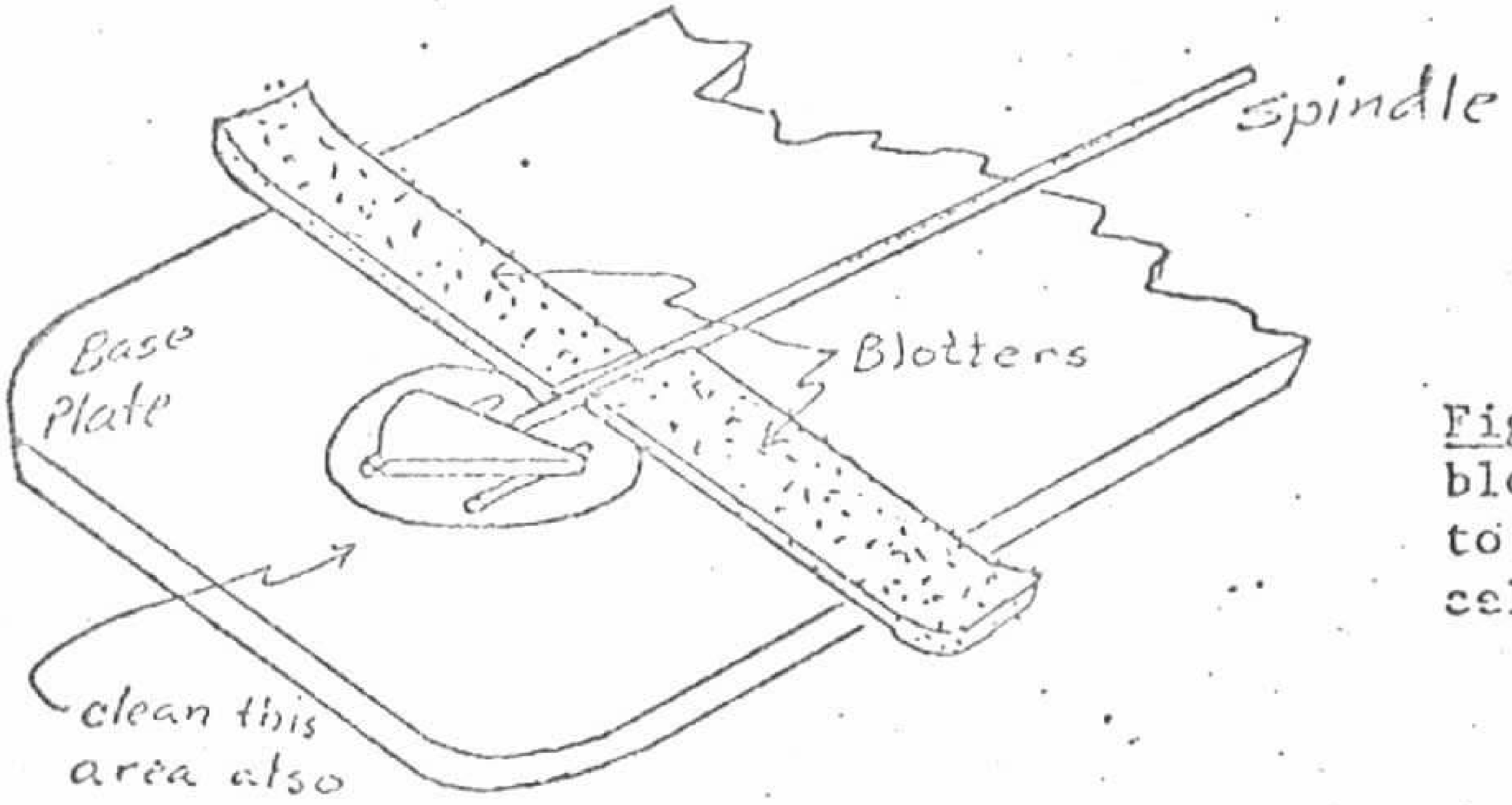


Fig. 1. Placement of blotter or filter strips to remove liquid from cell.

(2) A drop of the new liquid is then introduced into the front of the cell by barely touching the applicator rod to the plate and cell front, whereupon capillarity draws the liquid into the cell. To guard against contamination of the supply bottle of new liquid by any small amount of the old liquid picked up by the applicator, excess liquid on the tip is removed by touching it to a blotter before returning it to the supply bottle.

(3) The liquid now in the cell is slightly contaminated by the remnants of the old liquid and, unless only a rough index comparison is desired, an additional rinsing will be required. The liquid is again removed from the cell and from the plate in front of the cell with blotters, then a fresh drop of the new liquid added (and the applicator again touched to the blotter before returning to the bottle).

(4) If the index interval between the two liquids is not large, say not more than .01, contamination by this time has been reduced to a level too small to be detectable by routine techniques (sensitivity not better than about ± .001). Further rinsing therefore is not usually required, and one may proceed to compare the index of the crystal with that of the new liquid. If the index change has been large, or if very sensitive techniques of index comparison are to be used, additional rinses are advisable.