

Vol. III, No. 8

December, 1970


Seasons Greetings

Heroin as a fine yellow powder was encountered by the Chicago Police Department Laboratory. The yellow color was apparently added on purpose.

"Purple Rock" (Microgram Vol. III, No. 7 [Nov. 1970], p. 190) has been confirmed in two exhibits submitted from Thailand to the BNDD Special Testing and Research Laboratory. One exhibit consisted of 17 grams of "Green Lion" brand, #3 heroin, and sold for \$4.32. Analysis showed 48% barbital, 32% caffeine, 2.5% heroin and 1.5% strychnine. The other exhibit, 16.5 grams of "Lucky Strike" brand, #3 heroin, purchased for \$3.60, showed on analysis 39% barbital, 44% caffeine, 1.0% heroin, and 0.9% strychnine.

"Blue Cheer", a LSD tablet containing strychnine and methylamphetamine has appeared on the black market in Australia. The tablets were reported in the Australian press to contain 10 milligrams of strychnine, 350 micrograms of LSD, and 35 milligrams of methylamphetamine.

"The One", a black resinous substance purported to be natural tetrahydrocannabinol and resin extracted from marihuana, has been seen in northeastern United States. The material is believed to be smeared on bread and eaten, and is reportedly also smoked in a pipe.

Heart Shaped tablet, tan in color, has been examined in our Chicago Regional Laboratory as an exhibit from the State of Illinois, Bureau of Identification. The perfectly heart shaped tablet, measuring 9.6 mm. (maximum height), 8.9 mm. (maximum width) and 2.9 mm thick, is single scored between the two vertices with short score marks forming a peace symbol.  The tablet has been appearing in the Illinois area frequently. Analysis revealed no narcotics nor dangerous drugs. Lactose and starch were present.

Analytical methods in **Microgram** do not have official status. Use of funds for printing this publication approved by the Bureau of the Budget, April 8, 1969. **CAUTION:** Use of this publication is restricted to forensic scientists serving law enforcement agencies.

MDA (3,4-methylenedioxyamphetamine) tablets (Microgram Vol. III, No. 7 [Nov. 1970], p. 189) have been examined in the BNDD Special Testing and Research Laboratory. The tablets were generally round, convexo-concave, mottled grayish brown with white, reddish-brown or yellowish-brown. They were uneven in diameter and thickness, poorly made, and appeared to have been made in triturate mold. Buckling on drying made them convexo-concave. The tablets had an average weight of 119.2 milligrams and measured 5.45 - 5.70 millimeters (about 7/32 inch) diameter and were 3.98 - 4.94 millimeters thick.

Phencyclidine and marihuana in plant material was recently examined in an exhibit submitted by a Pennsylvania Police Department to the BNDD New York Regional Laboratory. Laboratory analysis showed 17.9 milligrams of phencyclidine hydrochloride per gram of plant material. Subsequently, the Washington Regional Laboratory found a mixture of marihuana and phencyclidine hydrochloride residue in a pipe received from a local police department.

Symposium on Drug Identification for Forensic Laboratories jointly sponsored by the National Association of Police Laboratories and BNDD was well received by all participants. Over eighty forensic scientists attended the three day symposium held in New York during November 18 - 20, 1970. Representatives from as far away as California, Canada, and Bermuda were present.

SELECTED REFERENCES:

Nakamura, George R. and Parker, Brian P., Assay Of Cocaine In The Presence Of Procaine and Quinine By Column Chromatography, Journal of Chromatography, Vol. 52, No. I, (October, 1970), pp. 107-110.

Davis, Larry E.; Kalousek, George; Rubinstein, Edward; Hepatitis Associated With Illicit Use of Intravenous Methamphetamine Public Health Reports, Vol. 85, No. 9, (September, 1970), pp. 809-813.

The International Reference Organization In Forensic Science (INFORM), The Forensic Sciences And The Drug Problem, Part-II-The Hallucinogenic Drugs, Vol. 3, No. 1, (January, 1971), pp. 1-8.

NEW EDITIONS:

Claus, E. P.; Tyler, V. E.; and Brady, L. R.; Pharmacognosy, Sixth Edition, 1970, Lea & Febiger, Philadelphia, Pennsylvania.

Remington's Pharmaceutical Sciences, (RPS XIV), Fourteenth Edition, 1970, Mack Publishing Company, Easton, Pennsylvania 18012

BNDD Forensic Chemist Seminars are planned for

February 8 - 12, 1971

April 12 - 16, 1971

June 14 - 18, 1971

These seminars are free of charge, but participants must provide their own transportation, lodging, meals, and other expenses. The sessions are held at the BNDD Headquarters, Washington, D. C.

The seminar is intended for forensic chemists engaged in law enforcement activities. For applications and additional information contact:

Special Training Division
National Training Institute
Bureau of Narcotics and Dangerous Drugs
Washington, D. C. 20537

MEETINGS

Sixth International Meeting of Forensic Sciences, Belfast, 1972

This meeting will be held from September 21st to 26th at the Queen's University, Belfast, Northern Ireland. There will be sections on:

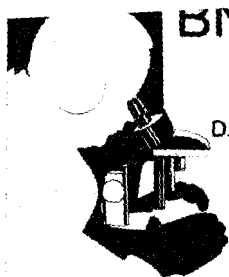
Biology	Pathology
Chemistry	Psychiatry
Toxicology	Dentistry
Jurisprudence	Questioned Documents

A social program will include receptions, entertainments and visit to the countryside. A special program will be arranged for ladies accompanying delegates. Inexpensive, but comfortable mixed accommodation for both individuals and families can be provided by the University and there are also hotels in the locality for those who prefer them.

A mailing list is now being prepared and those who would like to receive further information when published should write to:

The Secretariat
Sixth International Meeting of Forensic Sciences
Institute of Pathology
Grosvenor Road
Belfast, BT12 6BL
Northern Ireland

American Academy of Forensic Sciences annual meeting, Phoenix, Arizona, February 21-26, 1971. Registration forms are available from Arthur H. Schatz, J. D., Secretary-Treasurer, American Academy of Forensic Sciences, 750 Main Street, Suite 1000, Hartford, Connecticut 06103. Advanced registration is \$50.00, registration after January 20, 1971 or at the door is \$60.00. (See also Microgram, Vol. III, No. 7, November, 1970.)



BNUD LABORATORY NOTES

DATE 6/15/70

-203-

NO. 10

DRUG TYPE

METHODOLOGY X-ray diffraction

SCALE FOR THE CONVERSION OF ANGLES OF DIFFRACTION TO INTERPLANAR SPACINGS

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The procedure ordinarily followed in X-ray diffraction qualitative analyses involves the use of tables for the conversion of angles of diffraction to interplanar spacing. This is necessary because the Powder Diffraction File (1) lists the interplanar spacings (d-spacings) for the patterns on each substance, rather than the angles of diffraction. The reason is that the diffraction angles will vary with the X-ray wavelength being used, whereas the d-spacings are fundamental values which do not change.

The most suitable goniometer speed for organic compounds and mixtures (organic and/or inorganic) has been found to be $1^\circ 20'/\text{min.}$, which usually results in adequate resolution of the numerous peaks of the resulting patterns. Lesser speeds consume unwarranted time, and twice the speed ($2^\circ 20'/\text{min.}$) results in poor resolution of the peaks of the pattern. The X-ray diffraction goniometer is synchronized with the strip chart recorder so that each degree traversed by the goniometer is equal to a length of one inch on the chart. The usual procedure is to determine the angle of diffraction for each peak in the pattern from the ruled chart paper and write it down in the vicinity of the peak. Knowing the angle (2θ) and the wavelength (λ), the d-spacing (d) for the set of planes in the crystal responsible for the diffracted beam can be calculated using the Bragg equation

$$\lambda = 2d \sin \theta$$

where d is expressed in Angstrom units. Ordinarily the d-spacings are obtained from conversion tables (2) which are

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commercially available, and these are also written on the chart paper in the vicinities of the relevant peaks. Since there are many peaks in the patterns for organic and most inorganic substances, obtaining and notating d-spacings usually consume the major part of the operator time required for the analysis.

Scales for the conversion of angles to d-spacings for chart speeds of one inch/min., and goniometer speeds of $1^\circ/\text{min.}$, are not commercially available. Scales for goniometer speeds of $2^\circ 20'/\text{min.}$ are commercially available,* but these are of limited value because of reasons noted above. Therefore, a scale has been constructed from a plastic strip sixty inches in length and about three inches in width. Initially a strip taken from the chart paper used on the instrument was marked with the d-spacings for all angle fractions from 3° to 55° , a scan which is adequate for the identification of practically all of the compounds analyzed in this laboratory. Then the plastic strip was placed over the strip of chart paper and, with the aid of a T-square and triangle, lines were engraved with the sharp point of a compass. Along the two horizontal edges of the plastic strip, the angles were inscribed, one inch apart, to correspond to the chart paper. The main body of the scale was inscribed with lines representing the d-values for the angle fractions. The engraved lines were then filled with red or black wax.

It can be seen from the Bragg equation that for angles evenly spaced at one inch intervals, the d-portion of the scale will be a continuously expanding one from lower to higher angles. The angles will increase in one direction while the d-spacings will increase in the opposite direction. The accompanying drawings illustrate portions of the scale.

In using the scale, it is placed over a strip chart recording and lined up with respect to the diffraction angles shown on the chart and the scale. Thus the numbered d-spacings are superimposed on the peaks of the pattern and can be read directly. In most cases identification can be made without writing down any of the angles or d-spacings, since the d-spacings for the peaks of the pattern are there in full view by merely positioning the scale.

* N. P. Nies, 969 Skyline Drive, Laguna Beach, Calif. 92651

It has been found that the scale equals the accuracy of the conversion tables, and, in addition, reduces the chances of error.

It is well known that for some of the data in the Powder Diffraction File, especially data taken from film, because of experimental error or slight errors in measurement, the d-spacings may vary from their true values. Experience in interpreting X-ray diffraction patterns dictates what usually are reasonable maximum errors in routine X-ray work. Because the d-values are continually expanding, it is difficult to visualize how divergent the d-spacing of a certain peak in a pattern is from that found in the file. With the scale in position, however, the extent of the divergence is obvious.

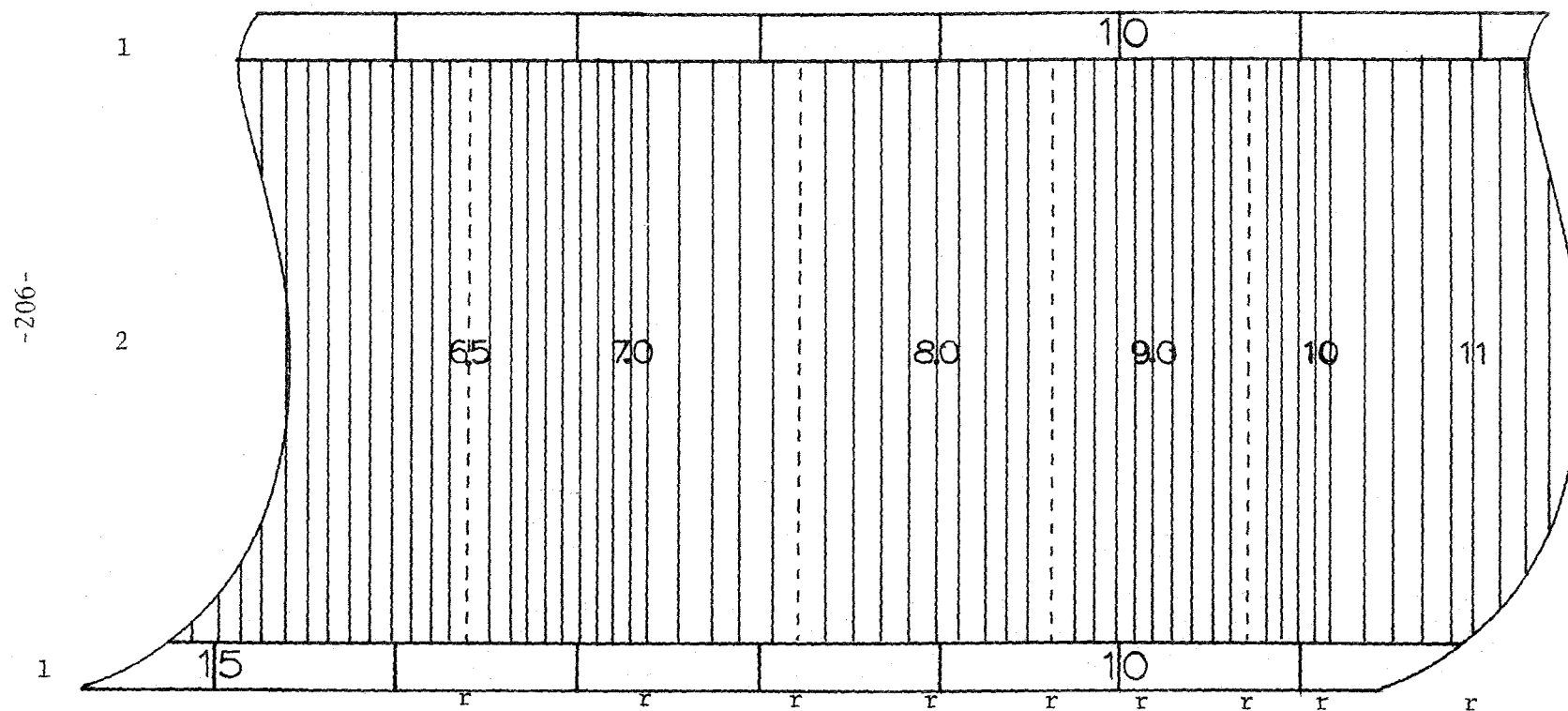
In addition to the above, the scale is useful as a ready reference when angles for certain d-spacings (or the reverse) are required in the manipulation of the diffraction equipment, making it unnecessary to consult the tables for such information.

Since the construction of the scale, a considerable amount of time has been saved in routine analyses and the accumulation of data on authentic reference materials.

REFERENCES

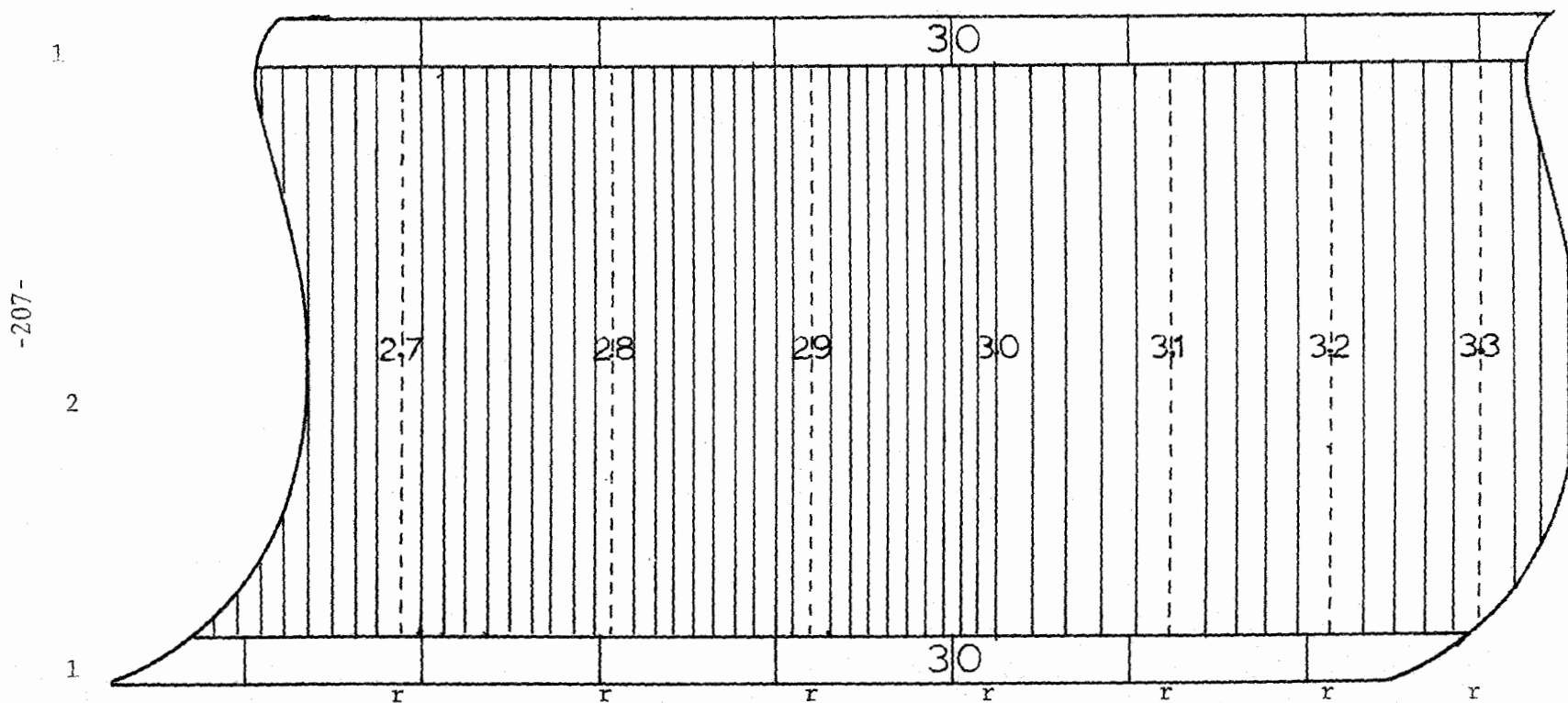
1. Powder Diffraction File and Index Volume (Organic and Inorganic), American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa.
2. Tables for the Conversion of X-ray Diffraction Angles to Interplanar Spacing, Applied Math. Series 10, U. S. Dept. of Commerce, National Bureau of Standards, 1950.

SEGMENT OF SCALE FOR THE CONVERSION OF X-RAY
 DIFFRACTION ANGLES TO INTERPLANAR SPACING
 (8° to $15^\circ 2\theta$)



- r: red lines
 1. X-ray diffraction angles (2θ)
 2. Interplanar spacing ($d\text{\AA}$)

SEGMENT OF SCALE FOR THE CONVERSION OF X-RAY
 DIFFRACTION ANGLES TO INTERPLANAR SPACING
 (27° to $34^\circ 2\theta$)



r: red lines
 1. X-ray diffraction angles (2θ)
 2. Interplanar spacing (dA)