

TABLE 2—Continued

<i>h</i>	<i>k</i>	<i>l</i>	$d_{\text{calc.}}^2$	$d_{\text{meas.}}$	<i>I</i>	<i>h</i>	<i>k</i>	<i>l</i>	$d_{\text{calc.}}^2$	$d_{\text{meas.}}$	<i>I</i>
-3	2	1	2.211	2.214	60	1	5	1	1.977	1.973	4
3	0	1	2.197	2.193	2	-2	-5	1	1.948	1.945	5
2	5	0	2.196			-1	8	0	1.926	1.925	6
-4	1	1	2.116			3	-6	1	1.914	1.911	15
1	-6	1	2.109	-4	7	0	1.908				
-4	3	1	2.092	2.089	30	4	-1	1	1.903	1.902	7
3	1	1	2.091			4	3	0	1.899		
-3	7	0	2.089							1.881	3 <i>b</i>
3	4	0	2.080	2.084	30				1.848	3 <i>b</i>	
0	-6	1	2.065	2.063	5				1.809	4	
-4	0	1	2.064							1.801	4
4	2	0	2.061							1.786	5
-5	3	0	1.994	2.000	15				1.770	6 <i>b</i>	
-5	2	0	1.992	1.986	4 <i>b</i>				1.757	5	
-4	-1	1	1.979							1.742	7
									1.628	42	

in Table 2. A film taken with  $\text{CrK}\alpha$  radiation was selected for publication, since it gave the best resolution of the many doublets and triplets which occur on the diffraction pattern; however, films taken with  $\text{FeK}\alpha$  and  $\text{CuK}\alpha$  radiations and diffractometer patterns taken with  $\text{CuK}\alpha$  radiation were also measured and gave similar values. The results are in general agreement with the pattern given in the ASTM X-ray Powder Data File under Sodium Carbonate Hydrate,  $5\text{Na}_2\text{O}\cdot 8\text{CO}_2\cdot 3\text{H}_2\text{O}$ , Wegscheider's Salt (Card No. 2-0696).

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A RAPID CHANGE ROTATING SAMPLE HOLDER FOR  
X-RAY DIFFRACTION OF POWDERS

JERRY M. WAITE, *Socony Mobil Oil Company, Inc., Field  
Research Laboratory, Dallas, Texas.*

Deviations in the  $x$ -ray diffraction intensity from a flat powdered sample are due principally to inhomogeneities from orientation of crystallites and segregation of crystalline mixtures (Klug and Alexander, 1954; Parrish, 1956; de Wolff *et al.*, 1959). Rotation of the powder sample about an axis perpendicular to its face reduces these deviations by a

factor of 4 to 5 (deWolff *et al.*, 1959). Devices giving this rotation are then particularly useful for quantitative analysis. A rotating sample holder that allows rapid interchange of samples and requires no adjustment of sample position relative to the  $x$ -ray beam has been designed as an accessory for the General Electric XRD-3D Spectrogoniometer. It is interchangeable with the flat sample holder normally used for diffraction from powder samples. Although other sample rotating devices have been made for the Norelco Wide Range Goniometer (Buhler, 1954; Cham-

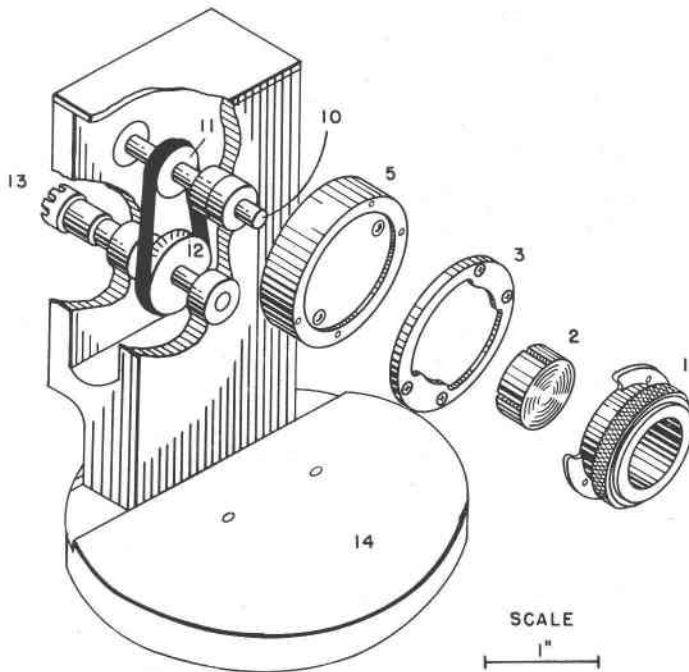


FIG. 1. Rotating sample holder for G.E. XRD-3D spectrogoniometer.

payne, 1946), the convenience of this rapid change sample holder stimulated design modification to permit its use on the Norelco unit.

The rotating sample holder consists of three separable units: the quick change sample holder, the mount and rotational drive assembly for the sample holder, and the drive motor.

Less than a minute is required to load the sample holder and put it in place for  $x$ -ray analysis. The quick change sample holder (1) is clamped with its face on a flat plate (Fig. 1). The powder sample is poured in the back of the holder and the piston shaped plug (2) inserted. Thumb pressure applied to the plug is usually sufficient to pack the sample and hold

it in place. The holder is then removed from the flat plate and the surface of the sample shaved with a razor blade. Upon inserting this flanged sample holder into its mount and turning it  $90^\circ$  to lock in place, the sample is ready for  $x$ -ray analysis. No further adjustment is required.

The mount for the sample holder consists of a flanged plate (3) which is fastened to the recessed plate (5). The sample holder is positioned in its mount by spring loaded ball bearings in the recess of plate (5) which keep the flanges of the sample holder tight against plate (3). Detents for the ball bearings in the sample holder flanges denote the locking position. A protective retaining cup (4) which fits into (5) was found desirable for the Norelco accessory to catch any sample material which might fall from the sample holder. The sample holders for this accessory unit were recessed to fit over the retaining cup.

In the G. E. XRD-3D accessory, the sample holder mount is attached to shaft (10) of the rotational drive assembly (cut-away section of 14). Shaft (10) has no end play, so the sample holder mount is always kept in the correct position relative to the  $x$ -ray beam. The pulleys (11, 12) give this unit a mechanical advantage or velocity ratio of about 1.33. The assembly is driven through the coupling gear (13) by a 60-RPM motor mounted on top of the goniometer behind the  $x$ -ray shield. The assembly is mounted in stand (14) which is interchangeable with the flat sample holder and may be easily removed after disengaging the coupling gear. The position of the sample holder is displaced from the center of the spectrogoniometer toward the detector to compensate for the divergence of the beam at low angles. By so doing, the full beam is diffracted off a  $\frac{3}{4}$ " diameter sample down to at least  $15^\circ$  ( $2\theta$ ). At  $11^\circ$  ( $2\theta$ ) only a small part of the beam does not strike the sample. Hence for Cu  $K\alpha$  radiation, the principal diffraction peaks of gypsum and kaolin can be utilized.

For the Norelco accessory (Fig. 2), the sample holder mount is fastened to the top of the vertical shaft (20) which is positioned by the threaded stop (21) and rotated by the rotational drive assembly (22, 23, 24, 25, 26, 28). A synchronous motor attached to the gear end of the mount (27) turns a driving rod (26) through gear (28). On the other end of the driving rod (26) is pulley (25) which rotates the vertical shaft (20) by means of pulley (22) and belt (not shown) through the idler pulleys (24). The shaft (20) is held vertical and allowed to turn by ball bearings; the upper bearing is mounted in the upper leaf of the yoke-like support (23) and the lower bearing is mounted separately but fastened to the lower leaf of the yoke (23). The mount (27) also supports a radiation shield (not shown).

Although the advantage of this rotating sample holder over the commercially available Norelco rotating sample device (Buhler, 1954) is mainly one of convenience, the possibility of sample material sifting into

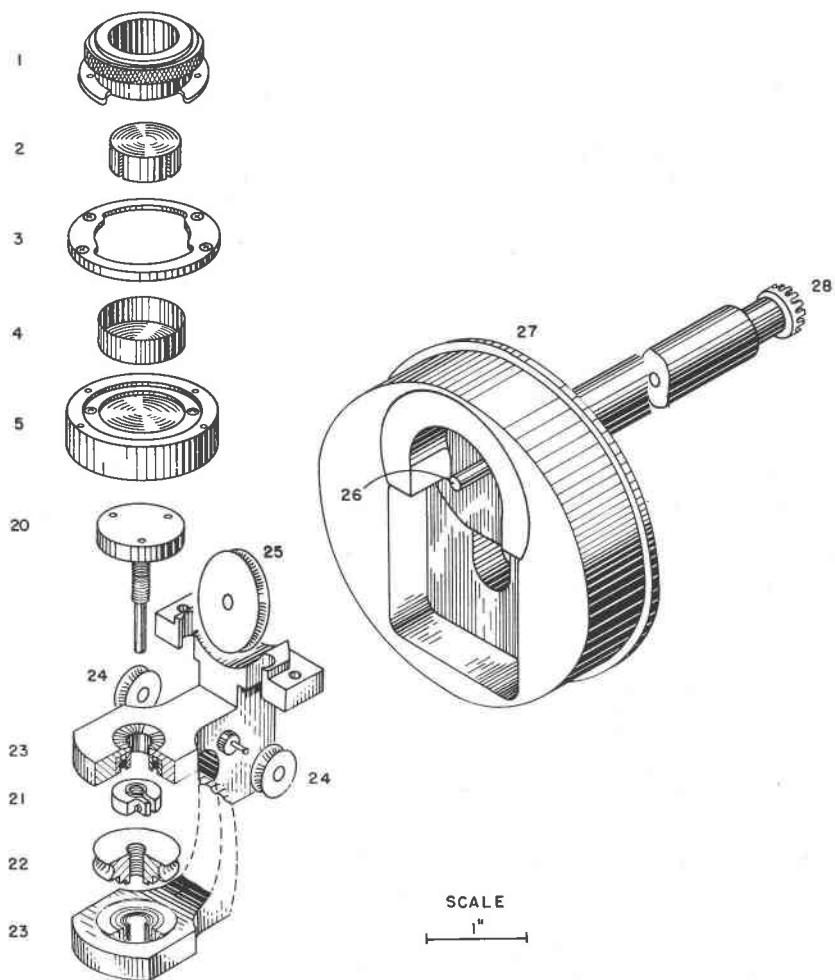


FIG. 2. Rotating sample holder for Norelco wide range goniometer.

the rotating mechanism or gear drive has been eliminated. The large diameter sample,  $\frac{7}{8}$ " , centered with respect to the goniometer may be used to give useful diffraction data to  $11^\circ$  ( $2\theta$ ).

The author wishes to acknowledge the assistance of William F. Mueller for mechanical design and construction of both models of this rotating sample holder, the permission of Everett D. Glover to include the description of the rotating sample holder as modified for the Norelco Wide Range goniometer, and the permission of the Socony Mobil Oil Company, Inc. to publish this paper.

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MINERALOGICAL EVIDENCE FOR THE PENECONTEMPORANEOUS  
LATERITIZATION OF THE BASALTS FROM NEW ENGLAND, N.S.W.

P. BAYLISS AND F. C. LOUGHNAN, *Department of Applied Geology,  
University of New South Wales, Australia.*

During a recent visit to a number of disused shafts and open-cuts in the New England district of New South Wales, it was noticed that well-developed bauxite profiles are rare, whereas most of the exposures appear to be a succession of profiles commonly superimposed on one another. Moreover, contrasting thin bands of light-colored and often highly-siliceous sediments are commonly interstratified with the weathered horizons. These observations suggest that the individual basalt flows were lateritized soon after emplacement and that successive outpourings covered previously weathered flows.

In a description of these bauxite deposits, Hanlon (1945) and Owen (1954) have implied that the lavas were lateritized during a period subsequent to emplacement.

Since the field observations appear incompatible with the implications of the above mentioned workers, it was decided to investigate the problem by specific mineralogical examinations of a few representative sections through the New England bauxites.

Three bauxite-sequences, exposed in a shaft near Emmaville, N.S.W., an open-cut west of Inverell, N.S.W., and a shaft north of Tingha, N.S.W., were sampled and subjected to detailed mineralogical analyses by the techniques of Loughnan and Bayliss (1961). The mineralogical results are quoted in Table 1. In addition, thin section studies were made to locate generic features such as relict textures and micro-structures.

Figures 1 and 2 are graphical representations of the mineralogy from Table 1 for the Emmaville shaft and the Inverell open-cut respectively.