TECHNICAL NOTE

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A Method for Recording X-Ray Diffraction Patterns for Trace Quantities of Crystalline Materials

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ABSTRACT: A procedure has been developed for recording diffraction patterns of small amounts of material and has been applied to the identification of silicon carbide in lubricating oil. To use this technique for the identification of material, the sample must be crystalline and the constituent of interest must be present in 1% or greater concentration.

KEY WORDS: criminalistics, radiography, crystalline material

X-ray diffraction procedures have been used quite sparingly in forensic science work. Usually the amount of sample available demands the use of the Debye-Scherrer photographic method because insufficient sample is available even for smear amounts for use in a recording diffractometer.

The usual technique for recording X-ray diffraction patterns requires at least 200 mg of material to obtain the required 2 mm optimum thickness in a standard Philips slide mount [*1*]. The photographic technique is in part inconvenient in the work involved in preparing and mounting the sample spindle and developing the film; it is even more inconvenient because of the time involved in obtaining the interplanar *d* spacings for the crystalline sample from the photographic film and because of the difficulty in estimating the line intensities.

A method previously described for recording the X-ray diffraction patterns of particulates on air filters [*2*] has been extended to the identification of a sample of forensic science interest where only 9 mg of sample was available. The sample was recovered from the crankcase oil in a diesel engine in a case where industrial sabotage was suspected. Only 0.9 L (1 qt) of oil was available for examination, and from this 9 mg of suspended solid was recovered.

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FIG. 1—*Assembled microfiltration apparatus.*

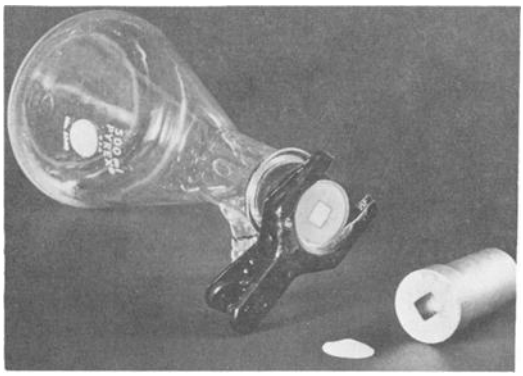

FIG. 2—*Exploded view of microfiltration device.*

TABLE 1—*Instrument conditions.*

Condition	Suspect Sample	Valve Grinding Compound
Voltage, kV	32	35
Amperage, mA	15	15
Range, count/s full scale	100	500
Time constant, s	5	2
Baseline, V (integral mode)	0.9	0.9
Scan rate, deg 2θ/min[a]	1	1
Chart rate, mm/h (in./h)	762 (30)	762 (30)

[a]Where 2θ is a standard notation for the angular relationship between the source beam and the sample.

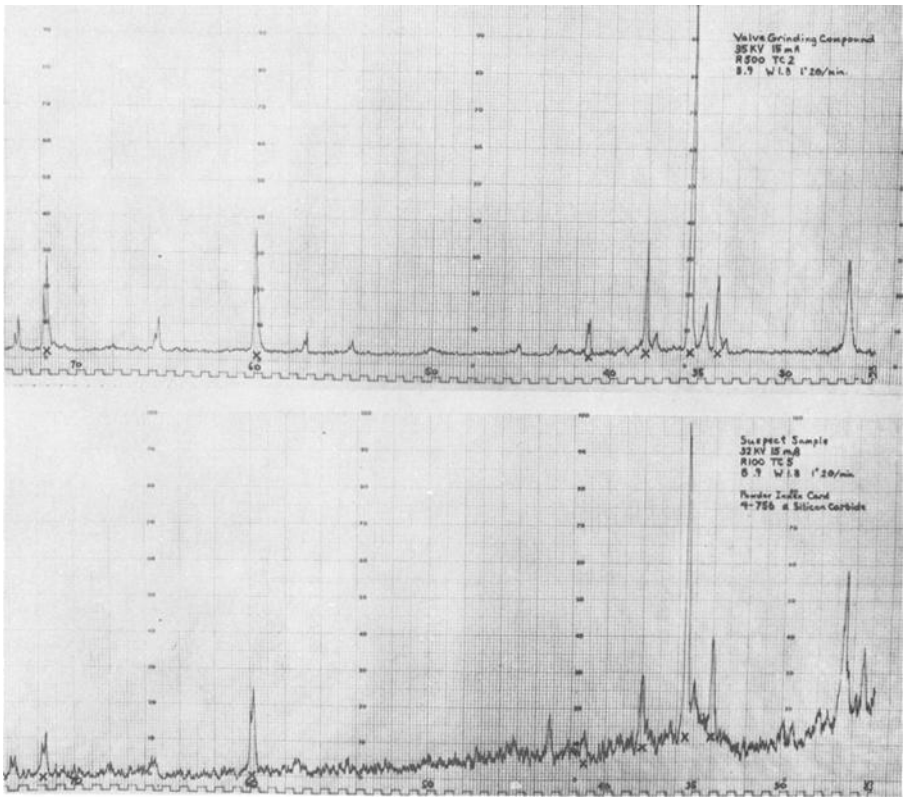

FIG. 3—*Patterns from commercial valve grinding compound containing α-silicon carbide* (top) *and suspect sample* (bottom).

Experimental Procedure

The engine oil was diluted with petroleum ether and passed through filter paper with a Buchner funnel. Approximately 9 mg of particles was collected. Microscopic examination showed sand-like particles exhibiting colors of clear yellow and green intermixed with black, lustrous particles exhibiting conchoidal edge chipping.

The sample was ground in an agate mortar to a fine dust that was then washed with a 5% Duco cement in acetone solution into the microfiltration device (Figs. 1 and 2). The prepared filter disk was mounted over a standard Philips Electronic Instruments small sample X-ray mount and placed in the goniometer. The instrument conditions are reported in Table 1. The remarkably clear pattern shown in Fig. 3 was recorded, suggesting that a single crystalline compound was present as the major constituent.

Reference to the *Inorganic Index* [*3*] indicated a match with α-silicon carbide indexed in the *Powder Diffraction File* [*4*] as Card 4-756. Comparison with the card confirmed the match. To further substantiate the match a sample of commercial silicon carbide valve grinding compound was prepared in the same manner as the sample; the matching pattern is also shown in Fig. 3.

References

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