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Isochron/West, Bulletin of Isotopic Geochronology, v. 49, pp. 4-7

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ISOCHRON/WEST
A Bulletin of Isotopic Geochronology

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K-AR AND Rb-Sr AGE DETERMINATIONS FROM CLAY MINERALS AND RELATED MINERALS FROM THE WIPP (WASTE ISOLATION PILOT PLANT), SOUTHEASTERN NEW MEXICO

DOUGLAS G. BROOKINS
STEVEN J. LAMBERT

Department of Geology, University of New Mexico, Albuquerque, NM 87110
Earth Sciences Division, Sandia National Laboratories, Albuquerque, NM 87185

Samples of evaporites, most with clay minerals and/or other secondary or non-evaporite minerals, were obtained from the underground WIPP (Waste Isolation Pilot Plant) facilities in southeastern New Mexico. The major purpose of the present investigation was to compile X-ray, K-Ar, and Rb-Sr data to assess the nature of the clay minerals and other insoluble minerals from the WIPP site area. These samples are briefly described in table 1. The approximate location of all samples is 32°22'10"N and 103°47'30"W. Both FH series and WP series of samples are from the underground workings of the WIPP site, developed in the lower Salado Formation (Ochoan, Permian). The series of samples designated FH were collected during excavation by IT Corporation while the samples designated WP were collected on site by us.

The samples were, whenever possible, split into two aliquots. One was used for dissolution, and the insoluble residue and the reprecipitated evaporite soluble fraction were analyzed separately. In some samples the clay minerals and other minerals could be hand separated from the

evaporite minerals. The second aliquot was analyzed directly as a whole rock. Separate sub-aliquots of the above materials were selected for X-ray study and for K-Ar and Rb-Sr age determination study.

X-RAY STUDY

Samples from both the FH and WP series were analyzed by X-ray diffraction to determine the mineralogy, with emphasis on the insoluble residues and/or clay mineral-rich separates from the whole rocks. The data are presented in table 1. The FH series samples, which were analyzed first, were not as well documented as the WP series, as the FH samples are fragments recovered during excavation of advancing drifts, and are therefore only approximately located. Hence, for the FH series in table 1, no attempt is made to estimate the relative proportions of minerals identified, whereas for the WP series such an attempt is made in order to evaluate their suitability for future geochronological determinations.

Smectites, including some possible corrensite and mixed layer smectite-illite and smectite-chlorite, are the dominant

TABLE 1

| X-RAY DATA—INSOLUBLE RESIDUES AND CLAYS—FH SERIES | | | | | | | | |
|---|----|----|----|----|----|----|----|---|
| Sample | HA | PH | GY | AN | SM | IL | CH | Q |
| FH 4-I | — | — | X | X | — | — | — | X |
| FH 6A-I | — | X | X | X | — | — | — | — |
| FH 6B-C | — | — | — | — | X | X | X | — |
| FH 27-I | — | — | X | X | — | — | — | — |
| FH 27-C | — | — | — | — | — | X | X | — |
| FH 32-C | — | — | — | — | — | X | X | X |
| FH 40-I | X | — | X | X | — | — | — | — |
| FH 42-I | X | — | X | X | — | — | — | — |
| FH 42-C | — | — | — | — | X | X | X | X |
| FH 45-C | — | — | — | — | X | X | X | X |

HA = halite, PH = polyhalite, GY = gypsum, AN = anhydrite, SM = smectite, IL = illite, CH = chlorite, Q = quartz.
X = present in readily identifiable amounts.

| X-RAY DATA—WP SERIES | | | | | | | | | | |
|--|----|----|----|----|-----|----|----|----|---|----|
| Sample | HA | PH | GY | AN | SM* | IL | CH | MG | Q | KA |
| i. Clay Separates and Insoluble Residues | | | | | | | | | | |
| WP-1 | — | — | — | — | M | X | t | t | M | — |
| WP-2 | — | — | — | — | M | X | — | X | M | — |
| WP-3 | — | — | — | — | M | M | — | t | X | — |
| WP-4 | — | — | — | — | M | X | t | t | M | — |
| WP-7 | — | — | — | — | M | X | t | t | M | — |
| WP-8 | — | — | — | — | M | M | — | t | X | — |
| WP-9 | — | — | — | — | M | X | t | t | M | — |
| WP-10 | — | — | — | — | M | X | — | t | M | — |
| ii. Salts | | | | | | | | | | |
| WP-5 | X | M | t | — | ? | — | — | — | — | — |
| WP-6 | X | M | M | X | — | — | — | — | — | — |
| WP-11 | M | M | M | M | — | — | — | — | — | ? |

HA = halite, PH = polyhalite, GY = gypsum, AN = anhydrite, Sm* = smectite, with possibly some corrensite, IL = illite (may include mixed layer smectite-illite in part), CH = chlorite, Mg = magnesite, Q = quartz, KA = kainite.

— = not detected, M = major, X = easily identifiable, t = trace, ? = uncertain.

clay minerals identified, with illite and chlorite subordinate. Magnesite is common in the WP series and probably also present in the FH series. In general, the clay minerals identified appear to be the usual suite from marine evaporites. Other minerals present after separation include quartz, anhydrite, gypsum, \pm halite, and \pm polyhalite.

K-Ar DATES

K-Ar dates were obtained through Geochron Laboratories, Cambridge, Massachusetts. Table 2 contains information on age (Ma) and $^{40}\text{Ar}^*/^{40}\text{Ar}$ (to indicate magnitude of air correction). The ages as reported by Geochron have been corrected to the new decay constants for ^{40}K by the authors. Background information on the K-Ar dating of WIPP and related evaporites and associated materials may be found in Brookins and others (1980), Register and Brookins (1980), Brookins (1981), and Brookins (1982).

Some earlier work indicated that polyhalite retained its $^{40}\text{Ar}^*$ sufficiently well to give reliable dates, many of these dates clustering in the age range 180–220 Ma. Brookins (1981) showed that an empirical correction factor could be applied to polyhalites for admixed halide minerals (i.e. halide minerals lose $^{40}\text{Ar}^*$ easily) with resultant ages corrected to in excess of 200 Ma. Register (1981; also see Register and Brookins, 1980) examined the Rb-Sr systematics of clay minerals from the WIPP area evaporites, with a resultant isochron date of 390 ± 77 Ma. This information was interpreted by Brookins and others (1980) and Brookins (1981) as evidence that these clay minerals are largely of detrital origin, and had not been so severely altered by their entrapment in the evaporating brines as to reset their internal Rb-Sr systematics to age of evaporites (e.g. 220–230 Ma).

In the present study, the focus was on clay minerals from the WIPP site underground workings. Two types of

samples were used for study; those from which enough clay (and oxyhydroxide) minerals were present for mechanical separation, and those in which an attempt was made to separate clays by dissolution of the salt samples. In addition, one sample (WP-5) contained enough polyhalite for hand separation. The attempt at concentrating sufficient insoluble, uncontaminated material by dissolution did not work well. The X-ray data (table 1) show that the samples so treated contain moderate amounts of gypsum, halite, anhydrite; their chemistry indicates the presence of significant amounts of K (table 2), presumably derived from the evaporite salts. This excess K is probably the reason why the K-Ar dates shown in table 2 for the insoluble residues range from 55 to 114 Ma. These dates, based on potassium derived from a mixture of phases, are interpreted as having no significance at all.

The K-Ar dates for the clay mineral (\pm oxyhydroxide minerals) separated by hand, however, yield dates from 365–390 Ma, in agreement with the Rb-Sr work of Register and Brookins (1980). These dates are offered as evidence that the K-Ar systematics of these clay minerals were not reset to age of evaporite mineral formation when entrapped in the evaporitic brine environment. The significance of this fact will be discussed below.

One hand picked polyhalite sample (FH6A) yields a date of 187 ± 7 Ma. Data are not available for Na content of the sample, hence a correction factor (e.g. Brookins 1981) cannot be applied. We infer that this polyhalite would yield a sodium corrected date of 210–230 Ma like the other samples from the area.

One sample of clay bearing halite was analyzed by the K-Ar method (FH27). Somewhat surprisingly, it yielded a K-Ar date of 324 ± 20 Ma. This age must reflect the presence of the > 365 Ma clay in the sample (FH27-C), diluted to a small degree with polyhalite and halite.

TABLE 2. Potassium-Argon Age Determinations.

| Sample | $^{40}\text{Ar}^*$ (ppm) | K (%) | $^{40}\text{Ar}^*/\Sigma^{40}\text{Ar}$ | t (Ma) |
|----------------------|--------------------------|--------|---|----------------|
| FH-6B-C ¹ | 0.02195 | 0.0694 | 0.387 | 378 ± 15 |
| | 0.02007 | 0.741 | 0.526 | — |
| FH-45-C,O | 0.02755 | 0.904 | 0.753 | 390 ± 15 |
| | 0.02583 | 0.860 | 0.657 | |
| FH-42-I | 0.005177 | 0.147 | 0.053 | 88.7 ± 5.3 |
| | 0.001335 | 0.197 | 0.087 | |
| | | 0.273 | | |
| FH-32-C | 0.02272 | 0.769 | 0.744 | 387 ± 15 |
| | 0.02305 | 0.757 | 0.555 | |
| FH-27-C | 0.02315 | 0.821 | 0.659 | 365 ± 14 |
| | 0.02259 | 0.806 | 0.546 | |
| FH-27-I(P) | 0.001913 | 0.491 | 0.125 | 55.5 ± 3 |
| | 0.001873 | 0.475 | 0.138 | |
| FH-27-WR | 0.000898 | 0.045 | 0.164 | 324 ± 20 |
| | 0.000555 | 0.050 | 0.193 | |
| | 0.002236 | 0.057 | 0.415 | |
| FH-26-C | 0.001311 | | 0.334 | |
| | 0.02783 | 0.931 | 0.548 | 382 ± 15 |
| | 0.02516 | 0.860 | 0.492 | |
| FH-6A-I | 0.09240 | 6.687 | 0.894 | 817 ± 7 |
| | 0.1056 | 7.245 | 0.795 | |
| | 0.08967 | 6.987 | 0.790 | |
| FH-4-I | 0.005578 | 0.634 | 0.243 | 114 ± 5 |
| | 0.004565 | 0.684 | 0.173 | |
| | 0.006156 | | 0.195 | |

¹C = clay mineral separate, O = oxide minerals, I = insoluble residue, P = polyhalite, WR = whole rock.

Rb-Sr DATES

Rb-Sr dates on the FH series of samples were determined in the UNM Geology Department Geochronology Laboratory. The data are given in table 3, and figure 1. The methods used for the study included standard dissolution

methods for silicate-oxide assemblages, involving dissolution in HF: deionized water, cooling, filtering, and separation of Rb and Sr by ion exchange chromatography using 2.2 N HCl as eluant. Samples were spiked with ^{84}Sr -enriched and ^{87}Rb -enriched tracers prior to dissolution.

TABLE 3. WIPP Site Rb-Sr Data Summary

| Sample | Rb(ppm) | Sr(ppm) | $^{87}\text{Sr}/^{86}\text{Sr}$ | $^{87}\text{Rb}/^{86}\text{Sr}$ |
|---------|---------|-----------|---------------------------------|---------------------------------|
| FH4ins | 10.96 | 9041.0(1) | 0.7072(8) | 0.00351 |
| FH6ins | 0.54 | 3481.7(1) | 0.7076(3) | 0.00045 |
| FH6Bins | 42.18 | 58.3(6) | 0.7199(9) | 2.0937 |
| FH26wr | 0.15 | 1.2(2) | 0.7083(0) | 0.35576 |
| FH27ins | 2.55 | 7449.1(4) | 0.7080(5) | 0.00099 |
| FH32wr | 0.45 | 1.7(6) | 0.7130(0) | 0.74016 |
| FH40ins | 11.41 | 4129.5(3) | 0.7073(5) | 0.00799 |
| FH42ins | 40.57 | 83.7(3) | 0.7159(9) | 1.40306 |
| FH45ins | 48.17 | 42.0(2) | 0.7271(0) | 3.32312 |
| WP-1ins | 46.31 | 25.1(4) | 0.7405(7) | 5.34694 |
| WP-2ins | 36.77 | 33.0(0) | 0.7259(2) | 3.22964 |

ins = insoluble, wr = whole rock

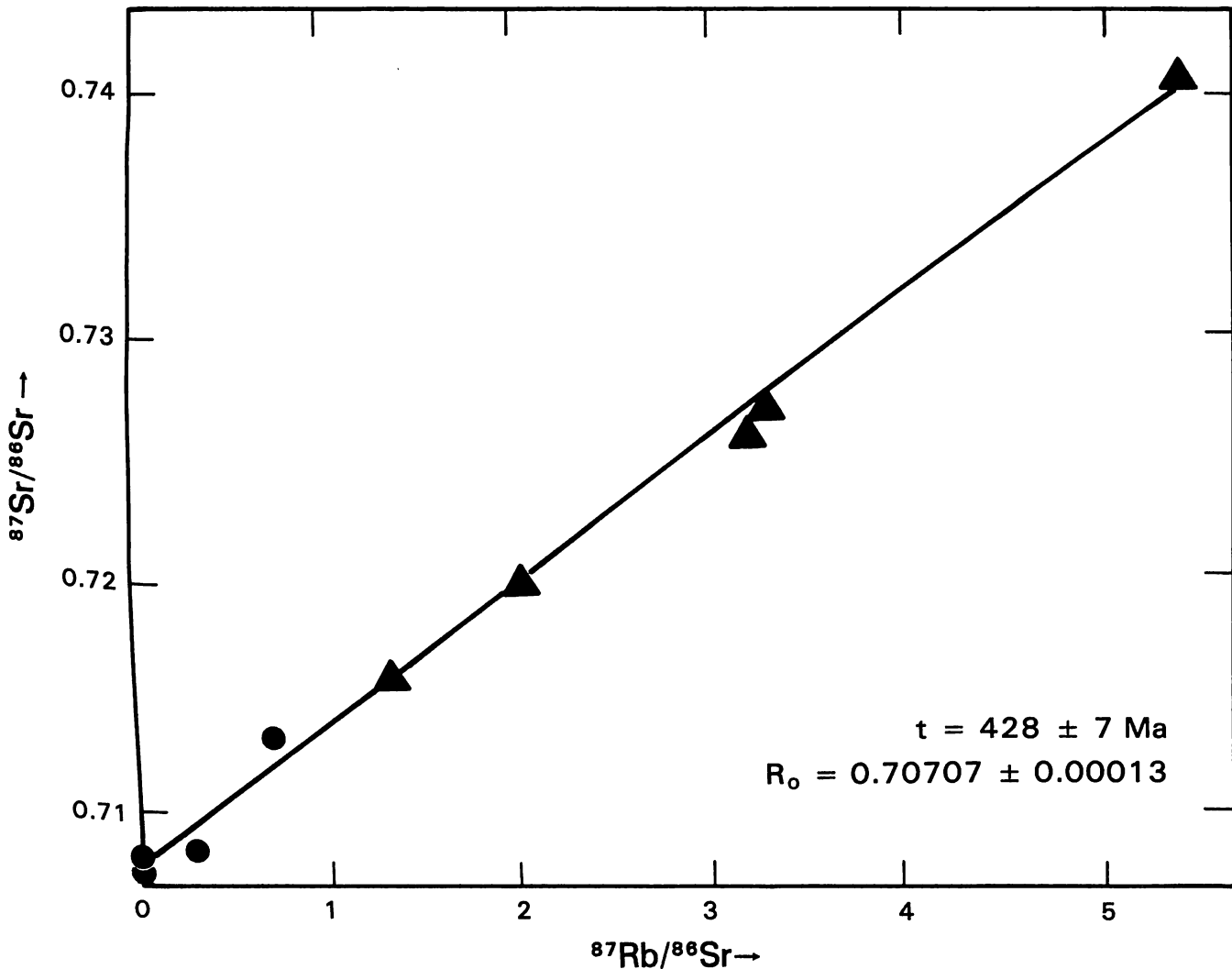


FIGURE 1. Rb-Sr isochron for clay minerals (▲) and oxy-hydroxide minerals (●) (see text for discussion).

After the ion exchange procedure, samples were fused in quartz beakers, cooled and stored. Samples were loaded onto rhenium filaments and isotopic data obtained using a Nier-design 1290 thermal ionization mass spectrometer. All $^{87}\text{Sr}/^{86}\text{Sr}$ values were normalized to $^{86}\text{Sr}/^{88}\text{Sr} = 0.1194$. Six runs of Eimer and Amund Standard SrCO_3 during the course of this study yielded $^{87}\text{Sr}/^{86}\text{Sr} = 0.70803 \pm 0.00005$ (one sigma).

DISCUSSION

Previous work shows a clustering of dates of clay minerals, both by Rb-Sr and K-Ar, between 330 and 430 Ma. Surprisingly, the insoluble residues from the samples of this study define a fairly good isochron with a 428 Ma date. This date is certainly within the limits of uncertainty of previously obtained K-Ar and Rb-Sr dates of clay minerals from the Salado Formation.

The original interpretation of these pre-Permian dates, by Brookins and others (1980) and Brookins (1982), was for an eolian origin for the clay minerals, and lack of re-setting of their K-Ar and Rb-Sr isotopic systematics due to incorporation into, and reaction with, the evaporitic brines. The 390 ± 77 Ma Rb-Sr isochron for clay minerals from several locations (Register, 1981) was badly scattered, and no quantitative comments concerning provenance, and thus extent of diagenetic reaction, could be made. The isochron of figure 1, however, is much more linear (i.e. the error on the age is only 7 Ma and the error on the initial intercept only ± 0.00013 using York's [1969] calculations). Since the samples used for this study are all from the WIPP mine, then problems of scattered sample provenance may have been avoided. For example, if samples with different prehistories, including different initial ratios, are homogenized at some time, t , then several parallel isochrons may result, all of the same age. This cannot be ruled out for the geographically scattered samples reported by Register (1981). The data of this study also suggest, but do not unequivocally prove, that there may have been a 400–450 Ma source area for the clay minerals which dominated the input. Since, in theory, source areas could

have included everything from earlier in the Permian to the Precambrian, this is surprising, and should be pursued for the purposes of paleogeography.

For the present study, however, the importance of the 428 Ma isochron of figure 1 is that, again, the samples were largely unaffected by their diagenetic residence time in the evaporitic brines.

ACKNOWLEDGEMENTS

Partial financial support was provided by Sandia National Laboratories Contract No. 52-9984. David Ward, Mark Murphy, and Ronald Matheney, UNM, carried out the Rb-Sr analyses.

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