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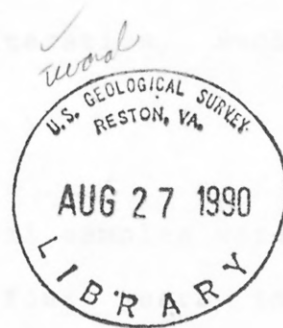
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Rehydration of Desiccated Argonne Premium Coal Samples

by

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Rehydration of Desiccated Argonne Premium Coal Samples

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The Argonne National Laboratory has taken measures to ensure that their eight premium coal samples retain their original properties during storage by sealing them in glass ampoules filled with an inert gas (argon) (Vorres, 1989). However, once the ampoules are opened in a laboratory, sample alteration, such as dehydration and oxidation, can occur.

We experienced a situation in which Argonne coal samples were stored improperly in our laboratory for two to four years in polyethylene bottles which allowed the samples to dehydrate (see columns 1 and 2 of Table 1). A substantial amount of moisture had been lost from each sample during storage.

This note is intended to illustrate the degree of dehydration and to discuss the possibility of rehydrating the samples.

Table 1. Moisture¹ of Argonne Premium Coal Samples Under Different Conditions. Values are in weight percent.

<u>Sample</u>	<u>Rank</u> ²	<u>"Fresh"</u> <u>Samples</u> ³	<u>"Aged"</u> <u>Samples</u>	<u>After</u> <u>Resaturation</u>
UF PC-1	Bit.	1.13	0.17 (15) ⁴	0.16 (14) ⁴
WY PC-2	Sub.	28.09	8.79 (31)	9.76 (35)
IL PC-3	Bit.	7.97	1.41 (18)	6.77 (85)
PITT PC-4	Bit.	1.65	0.97 (59)	1.57 (95)
POC PC-5	Bit.	0.65	0.10 (15)	0.23 (35)
UT PC-6	Bit.	4.63	0.21 (05)	3.29 (71)
WV PC-7	Bit.	2.42	0.85 (35)	1.29 (53)
ND PC-8	Lig.	32.24	1.94 (06)	11.90 (37)

1. All moisture values were determined using the ASTM recommended procedure (ASTM, 1988).
2. Bit. = bituminous, Sub. = subbituminous, Lig. = lignite
3. Samples in ampoules, data from Vorres, 1989.
4. Percent of original moisture content.

From 41 to 95 percent of the original moisture was lost during the unregulated storage of the "aged" samples. Sample WY PC-2 lost almost 20 weight percent moisture and ND PC-8 lost more than 30 percent. Sample POC PC-5 lost the least amount of moisture, just over 0.5 weight percent.

To resaturate the samples, weighed splits of -100 mesh coal were placed in uncovered petri dishes in a vacuum desiccator containing a barometer and a thermometer. Approximately 800 milliliters of deionized water was added at the base of the desiccator. The samples were kept under atmospheric pressure in the desiccator at 90 percent relative humidity and 72 F for 24 hours after which they were removed from the desiccator and weighed. Several samples had increases far in excess of the original moisture loss. For example, sample UF PC-1, which had lost about one weight percent moisture, increased over 5 weight

percent. This "excess" moisture may be due to condensation on the coal particles. We therefore allowed the resaturated samples to equilibrate with the ambient atmosphere for 24 hours (72-74 F, 60-64 percent relative humidity) prior to determining the moisture (referred to as resaturated moisture) by the ASTM (1988) procedure (Table 1, column 3).

After the 24 hour residency in the desiccator at 90 percent relative humidity and subsequent equilibration, there were substantial increases in moisture in most samples. Sample IL PC-3 regained 67 percent of the original moisture (Table 1, column 1), and UT PC-6 regained 66 percent. Sample IL PC-3 regained 5.36 weight percent moisture and ND PC-8 gained almost 10 weight percent. Only sample UF PC-1 showed no gain in moisture. Despite the substantial increases in moisture for most samples, none of the samples, with the possible exception of PITT PC-4, recovered all the moisture lost on drying.

Experiments on water desorption and adsorption have been conducted by varying vapor pressure and holding the temperature constant, usually less than 40 C (Gauger, 1945; Allardice and Evans, 1978). Results of these experiments indicate that, once dry, a coal sample will not adsorb enough water to regain its original moisture content. There is no generally accepted mechanism to explain this phenomenon (Allardice and Evans, 1978), however, several theories have been offered. These include: 1) the shrinking of coal upon drying which causes a collapse of some capillaries, so that the dried material can no longer hold or

take up as much water as it held originally; 2) the replacement of moisture on the walls of some capillaries by adsorbed gases making it difficult to re-wet the capillaries (Gauger, 1945).

Vorres and others (1988) and Vorres and Kolman (1988) conducted drying and rehydration studies of Argonne Premium Coal Samples. They concluded that coal rank, particle size, and degree of oxidation affected moisture removal and replacement.

The Argonne Premium Coal Samples behave in a typical fashion with respect to rehydration. Improper storage can lead to substantial moisture loss. Resaturation generally will restore some of the lost moisture. These observations on moisture loss are important if measurements of physical properties are to be made on samples that have been stored under non-controlled conditions for any length of time. These observations are also important for calculations in chemical analysis of the raw coal (for example, instrumental neutron activation analysis or x-ray fluorescence analysis). Assuming that the coal samples have retained their original moisture contents can lead to errors of as much as 30 percent for the low-rank coals (lignites and subbituminous; ND PC-8 and WY PC-2, respectively). Even for higher-rank coal (e. g. IL PC-3) the errors can be as high as 6.5 percent.

We recommend the following procedures to minimize errors caused by desiccation (especially for low-rank coal):

- 1) After opening the ampoules, samples should be stored in such a way as to minimize dehydration.
- 2) Moisture content of the coal samples should be determined just prior to chemical analysis of whole coal.
- 3) If there is insufficient sample for moisture determination, rehydrate the sample to minimize the error. The ASTM method for determining equilibrium moisture (ASTM, 1988, D 1412) could be used to rehydrate the sample, but the method requires at least a 20 gram sample, more time and equipment than the procedure described in this note.

An alternative method would be to analyze a moisture-free sample by drying it (105 C for one hour) prior to analysis. Two assumptions are necessary, 1) that the published moisture value is applicable so that the analysis can be recalculated to an as-received basis, 2) that the sample does not pick up moisture between drying and analysis.

References

- Allardice, D. J. and Evans, D. G., 1978, Moisture in coal. In Analytical Methods for Coal and Coal Products, Vol. 1. C. C. Karr, Jr., ed. Academic Press, New York. Ch. 7, p. 247-262.
- ASTM (American Society for Testing and Materials), 1988, Annual Book of ASTM Standards, Vol. 05.05. ASTM, Philadelphia, PA. Standards D 3173, p. 295-296; D 1412, p. 208-209.
- Gauger, A. W., 1945, Condition of water in coals. In Chemistry of Coal Utilization, Vol. 1. H. H. Lowry, ed. J. Wiley & Sons, New York. p. 600-626.
- Vorres, K. S., 1989, Users Handbook for the Argonne Premium Coal Sample Program. Argonne National Laboratory. 54p.

Vorres, K. S., Kolman, R., and Griswold, T., 1988, Kinetics of vacuum drying and rehydration of Illinois #6 coal samples. Implications for pore structure. Preprints, Fuel Chemistry Division, American Chemical Society, Vol. 33, no. 2, p. 333-342.

Vorres, K. S. and Kolman, R., 1988, Kinetics of the vacuum drying and rehydration in nitrogen of coals from the Argonne Premium Coal Sample Program. Preprints, Fuel Chemistry Division, American Chemical Society, Vol. 33, no. 3, 7-16.

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