

over, it is hoped that pitchblende freer from impurities, a sample of which has recently been received from Spence, will yield results subject to a smaller correction for "common" lead. In the meantime the above tentative age offers a basis for geologic correlation.

Summary

1. A sample of pitchblende from LaBine Point, Echo Bay, Great Bear Lake, N. W. T., Canada, yields a "corrected" lead-ratio of 0.193, corresponding to an age of 1323 million years.

2. As the atomic weight and isotopic composition of the lead, and the Pa/UI ratio of this same sample have been determined, ages, etc., calculated by different methods should agree. This agreement has not been obtained in all instances, and possible reasons have been outlined.

3. The age found indicates that a possible Keewatin granite may be the source of the pitchblende.

37 EAST BRADLEY LANE
CHEVY CHASE, MARYLAND

RECEIVED DECEMBER 24, 1935

[CONTRIBUTION FROM THE LABORATORY OF THE DODGE & OLCOTT COMPANY]

Sodium Benzylsulfonate: A Case of Dimorphism

BY FRANCIS D. DODGE

Some years ago the writer had occasion to prepare sodium benzylsulfonate by the usual reaction of benzyl chloride with sodium sulfite in alkaline solution. At the completion of the reaction, the mixture was filtered hot, and on slow cooling deposited the salt in transparent rectangular plates, which invited examination under the microscope. In parallel polarized light, the crystals showed parallel extinction; in convergent light, the symmetrical interference figure of a positive orthorhombic crystal.

For purification, the crystals were redissolved in sufficient warm water and again allowed to crystallize slowly. On examination, a week or so later, a change in the appearance of the crystals was noted. The thin plates were longer, and showed a tendency to radiate from a center. The terminations were less distinctly rectangular, with occasional faces inclined to the elongation. Under the microscope the crystals presented a picture different from the first crop. Very thin plates showed an extinction angle of about 28° , and, in convergent light, the emergence of one axis near the center of the field. Thicker plates showed unusual optical properties, as will be described below.

It was evident that the two crops of crystals were not identical, and the first obvious hypothesis was that two different hydrates existed.

Analysis of the recrystallized product gave figures agreeing with those of Fromm.¹

(1) Fromm, *Ber.*, **39**, 3308 (1906).

Anal. Calcd. for $C_7H_7SO_3Na \cdot H_2O$: loss at 110° , 8.49; Na_2SO_4 , 36.6 (on dry salt). Found: loss 8.77; Na_2SO_4 , 36.08.

The preparation of pure orthorhombic crystals for analysis presented at first some difficulty. Without going into details of numerous experiments, it was found that the orthorhombic form was comparatively stable in the original mother liquor, which contained sodium chloride, sulfite, sulfate and hydroxide, but in a pure solution the crystals were rapidly converted into the asymmetric form.

For example, if an orthorhombic plate, about 6 by 13 mm., is placed upright in a small tube, and covered by a solution of the pure salt, saturated at the room temperature, and the tube kept at this temperature, observation with a lens will show that the plate is gradually converted into a mass of small prisms.

This transformation occurs in the recrystallization of the crude orthorhombic salt. From strong solutions, orthorhombic crystals are deposited on cooling, which on standing pass into the stable form. From weak solutions, by slow evaporation, the stable form may be obtained directly. Conversely, the stable form may be changed into the orthorhombic form by recrystallization from the original mother liquor, or from water containing 5 to 10% of sodium chloride, and about 0.5% hydroxide.

For some reason the original mother liquor is especially favorable to the stability of the ortho-

rhombic crystals. They may remain unaltered in this medium for three months or more, while in pure solution the change may be complete in a few days.

Specimens of orthorhombic plates, mounted in balsam, showed partial alteration after nine months, and after several years were transformed into a mass of fine crystals showing no optical orientation. Mounts of the asymmetric crystals showed no change.

For analysis, orthorhombic crystals were prepared by redissolving, removing sulfate and sulfite ions by the addition of sufficient barium chloride, and adding sufficient salt to make the composition of the solution; benzylosulfonate, about 25%, sodium chloride, about 5%, sodium hydroxide, about 0.5%. On cooling, well-shaped plates were obtained, which were washed with a little ice water, pressed and dried at a low temperature.

Anal. Loss at 110°: 8.74%. Calcd. for $1\text{H}_2\text{O}$, 8.49%.

It is evident that the two forms are dimorphous, as was already indicated by the fact that no difference in solubility was noted, and also by the frequent occurrence of both types of crystals in the same solution. Both forms effloresce on exposure at room temperature.

These results recall the analogous case of the calcium chromates, described by Mylius and Wrochem.² The α -dihydrate, $\text{CaCrO}_4 \cdot 2\text{H}_2\text{O}$, crystallizes in monoclinic forms, which on standing in the solution pass into the stable orthorhombic dihydrate.

Optically, the orthorhombic benzylosulfonate presented nothing unusual, but the asymmetric form showed some interesting properties. As already noted, very thin plates show one axis, and inclined extinction. Thicker plates show no definite extinction, but in convergent light, two axes appear in a line perpendicular to the elongation. This is, obviously, no ordinary biaxial figure. The two axial figures are hardly ever alike. One may show many rings, indicating

(2) Mylius and Wrochem, *Ber.*, **33**, 3686 (1900).

high retardation; the other may be only the hyperbolic brushes.

The phenomena seem to be explainable by a twinning of the crystal, the twinning plane being parallel to the elongation and perpendicular to the principal face. In fact, the axial figure may be reproduced by breaking a thin homogeneous crystal along a line perpendicular to the elongation, and rotating one half around this line, superposing it on the other half. The combination gives the double figure in proper orientation. The operation is not exactly easy, as the crystals have a ready cleavage parallel to the elongation.

Beside the double axial figure just described, other combinations occur. Sometimes one axial figure appears enormously enlarged with brilliant colors; or two such are present, resulting in complicated spirals. Also, crystals having a very flat wedge shape are often found, radiating from a center. The thin end will be a single crystal with definite extinction. If, now, keeping the crystal in the extinction position, we slide it under the objective, moving toward the thick end, we come to a color band. At this point, in convergent light, the beginning of the second axial figure is seen with minimum retardation. Continuing along the crystal, the bands change in color, and the retardation in the axial figure progressively increases. Meanwhile the retardation of the first axial figure may increase, remain constant, or decrease: the crystal being apparently a curious combination of two very flat wedges, of varying angles.

The easy preparation and mounting of the crystals, and the variety of optical effects to be noted make this salt interesting material for study.

Summary

Sodium benzylosulfonate crystallizes in two distinct forms: an unstable orthorhombic, and a stable mono-(or tri-)clinic modification. The conditions of formation, and the unusual optical properties of the latter are described.

BAYONNE, N. J.

RECEIVED JANUARY 7, 1936