NOTES

The Preparation of 1,5-Dimethylnaphthalene

By Eleanore W. J. Butz

1,5-Dimethylnaphthalene has been prepared by several procedures.[1] A convenient synthesis, by well-known reactions, which can yield only the 1,5-isomer is presented here. No separation of isomeric products is involved at any stage. 5-Methyl-1-tetralone[2] was prepared by the following steps: *o*-bromotoluene → *β*-*o*-tolylethyl alcohol → *β*-*o*-tolylethyl bromide → diethyl *β*-*o*-tolylethylmalonate → *β*-*o*-tolylethylmalonic acid → *γ*-*o*-tolylbutyric acid → 5-methyl-1-tetralone.

Reaction of this ketone with methylmagnesium iodide gave a carbinol which was not purified, but was dehydrated directly by heating in a Claisen flask with a crystal of iodine in a slow stream of carbon dioxide at 200° for one hour. The mixture was then distilled at 28 mm. The main product, presumably 3,4-dihydro-1,5-dimethylnaphthalene (b. p. 130–3° at 25 mm.), was accompanied by 10% of 1,5-dimethylnaphthalene (m. p. 76°). Five grams of the liquid distillate was heated with palladium–charcoal at 250° for two hours. Extraction of this mixture with ether yielded 4 g. of 1,5-dimethylnaphthalene (m. p. 76–7°). After recrystallization from ether and methanol, the product was dried at 35° and 80 mm. for one hour; m. p. 80°; picrate, m. p. 137°.

Anal.[3] Calcd. for $C_{12}H_{12}$: C, 92.31; H, 7.69. Found: C, 92.19; H, 7.73.

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(1) Vesely and Stursa, *Coll. Czechoslov. Chem. Commun.*, **3**, 430 (1931); Anderson and Short, *J. Chem. Soc.*, 485 (1933); Manske and Ledingham, *Can. J. Research*, **17B**, 14 (1939).

(2) Harvey, Heilbron and Wilkinson, *J. Chem. Soc.*, 423 (1930).

(3) Microanalysis by Arlington Laboratories.

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The Heat of Solution of Gadolinium Sulfate Octahydrate and the Entropy of Gadolinium Ion

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As an aid to a better understanding of the process of hydration of ions from the thermodynamic viewpoint, additional ionic entropy values for monatomic tripositive ions are of considerable value. As a step in this direction an entropy value for Gd^{+++} has been calculated in the usual manner from entropy, heat of solution and estimated free energy of solution data.

The heat capacity of $Gd_2(SO_4)_3{\cdot}8H_2O$ has been measured by Giauque and Clark[1] and Ahlberg and Clark.[2] On the basis of these measurements Kelley[3] has obtained 155.7 cal./deg. for the entropy which includes the magnetic entropy $R \ln 8$ for each gram ion of Gd^{+++}.

The heat of solution of $Gd_2(SO_4)_3{\cdot}8H_2O$ was measured in a calorimeter already described in This Journal.[4] The sample used in these measurements was prepared from some gadolinium oxalate of about 95% purity which was donated by Dr. Herbert McCoy. The impurity is probably neighboring rare earths which would lead to no appreciable error in the measurements. The oxalate was ignited to the oxide, dissolved in sulfuric acid and recrystallized. After attaining constant water content at room temperature, the hydrated sulfate was, of necessity, powdered to assure a rapid rate of solution. An analysis for volatile constituents of the salt by ignition to the oxide indicated a deficiency of 0.3% as compared with the calculated value. A portion of this may be attributed to the impurities in the sample used. The results of the experiments are tabulated in Table I. The final volume in each case was 1050 cc. As no heat of dilution data are available for this salt, the measured value will be used uncorrected in the following calculations. The error involved from this procedure will be taken into account in the estimated error quoted for the ionic entropy.

Table I

Heat of Solution of $Gd_2(SO_4)_3{\cdot}8H_2O$ at 25°

Sample, wt. g.	Mole	Observed heat absorbed, cal.	Molal heat absorbed, cal.
13.3563	0.01788	−118.66	−6640
13.0707	.01750	−118.56	−6775
			−6710 ± 100

Interpolation of the "I. C. T."[5] values for the solubility of $Gd_2(SO_4)_3{\cdot}8H_2O$ gives 0.041 M for the saturated solution at 25°. Since no activity coefficient data are available for $Gd_2(SO_4)_3$, the activity coefficient was estimated from data for $La_2(SO_4)_3$ and $In_2(SO_4)_3$. For 0.01 M $La_2(SO_4)_3$,

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(1) Giauque and Clark, This Journal, **54**, 3135 (1932).

(2) Ahlberg and Clark, *ibid.*, **57**, 437 (1935).

(3) Kelley, "Bureau Mines Bulletin 394."

(4) Pitzer, This Journal, **59**, 2365 (1937).

(5) "International Critical Tables," Vol. IV, p. 227.