

cool slowly and crystals formed after ten minutes standing at room temperature. After one hour the crystals were separated by filtration, dried, and washed with petroleum ether. A 90% yield of hexagonal yellow crystals was obtained. The material melted with decomposition at 143–144° and was found to be soluble (> 1 mg./ml.) in carbon tetrachloride, acetone and benzene, and insoluble in petroleum ether, hexane and isoöctane.

The analysis of this compound was carried out with a Sargent microcombustion apparatus. The water was absorbed by a tube filled with anhydron and the carbon dioxide by ascarite. Final temperatures in each ignition were 850° and all were carried out in an atmosphere of commercial grade oxygen. The cerium was determined gravimetrically as the dioxide and the nitrogen by a modified Kjeldahl procedure.

*Anal.* Calcd. for  $Ce(C_8H_7O_2)_3 \cdot 2NH_4C_8H_7O_2$ : Ce, 20.9; C, 44.7; H, 6.46; N, 4.17. Found: Ce, 21.1; C, 44.8; H, 6.29; N, 4.28; molar ratios, C/Ce, 24.8; N/Ce, 2.03.

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RECEIVED FEBRUARY 25, 1952

#### Preparation of Praseodymium Ammonium Acetylacetonate

To 6 ml. (0.058 mole) of refluxing acetylacetonate was added 6 ml. (0.091 mole) of concentrated ammonium hydroxide and the mixture refluxed for ten minutes. A solu-

tion of approximately 500 mg. of praseodymium nitrate (from the oxide in concentrated nitric acid evaporated to dryness) in 3 ml. of water was then introduced dropwise and the resulting solution refluxed for five minutes. The solution was allowed to cool slowly at room temperature and crystals formed within a few minutes. These crystals were a mixture of praseodymium acetylacetonate and the double salt with ammonia, and after being allowed to stand in the solution for one day were completely converted to the double salt. The solid was then separated by filtration, dried, and washed with petroleum ether. A 90% yield of hexagonal green crystals was obtained. The material melted at 145° and was found to be soluble (> 1 mg./ml.) in carbon tetrachloride and acetone, and insoluble in petroleum ether, hexane and isoöctane.

The analysis of this compound was carried out with Sargent microcombustion apparatus. The water was absorbed by a tube filled with anhydron and the carbon dioxide by ascarite. Final temperatures in each ignition were 850° and all were carried out in an atmosphere of commercial grade oxygen. The praseodymium was determined gravimetrically as  $Pr_2O_3$  and the nitrogen by a modified Kjeldahl procedure.

*Anal.* Calcd. for  $Pr(C_8H_7O_2)_3 \cdot 2NH_4C_8H_7O_2$ : Pr, 20.9; C, 44.6; H, 6.45; N, 4.17. Found: Pr, 20.7; C, 44.8; H, 6.33; N, 4.03; molar ratios, C/Pr, 25.4; N/Pr, 1.96.

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RECEIVED FEBRUARY 25, 1952

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## COMMUNICATIONS TO THE EDITOR

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#### CHROMATOGRAPHY OF STEREOISOMERS WITH "TAILOR MADE" COMPOUNDS

Sir:

Two years ago F. H. Dickey<sup>1</sup> published the results of his experiments on specific adsorption of silica gels prepared according to Pauling's suggestions<sup>2</sup> in the presence of the molecules for which specificity is desired. Since then no other news has appeared on this subject, in spite of the great interest in this new field. In previous work<sup>3</sup> we have established the conditions for the preparation of highly specific silica gels, which we used as chromatographic adsorbents. The influence of the acid used for the preparation of silica gel has been studied (we obtained good results with acetic acid and with cation exchange resins); it has also been pointed out that the most alkaline sodium silicate is the best starting material. Temperature has to be kept constant and at the same value at which the chromatographic experiments will be carried out.

Optical antipodes of camphorsulfonic and mandelic acids have been separated<sup>4</sup> using specific silica gels. An enrichment of 30% *l*-camphorsulfonic acid was obtained in the percolate by only one passage through a column containing 5 g. of silica gel specific to *d*-camphorsulfonic acid; 200 ml. of 0.01 *m* *dl*-camphorsulfonic acid was used in this

experiment. This gel had been prepared by adjusting the pH of 30 ml. of sodium silicate ( $d = 1.4$ ;  $Na_2O:SiO_2 = 3.34$ ), in which 2 g. of *d*-camphorsulfonic acid had been dissolved, to 4 and diluting with 250 ml. of water. The acid was extracted with methanol at room temperature and, since no optical activity was observed, we proceeded with the chromatography of *dl*-camphorsulfonic acid. Twenty-ml. fractions of the percolates were submitted to polarimetric measurements. A levo activity was observed to such an extent to leave no possible doubt of the effectiveness of the method. This activity increases in the second and third fraction of eluate; then it decreases, reaching the value of zero after 200 ml. has been chromatographed. The maximum of activity observed is of the order of a half degree, which corresponds to a 30% enrichment in *l*-camphorsulfonic acid. The apparent incongruence of an activity that increases passing from the first to the second and third fraction of percolate may be explained by the fact that a gel is not completely unadsorbent for the antipode opposite to the one for which the gel is specific.

Another series of experiments with mandelic acid confirmed the general applicability of this method, though the efficiency of the adsorbent was not as good as before, reaching now the value of 10% in the best fraction of percolate.

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RECEIVED MAY 29, 1952

(1) F. H. Dickey, *Proc. Nat. Acad. Sci.*, **35**, 229 (1949).

(2) *Chem. Eng. News*, **37**, 913 (1949).

(3) R. Curti, U. Colombo and F. Clerici, in press.

(4) R. Curti and U. Colombo, Communication at the Meeting of the Italian Chem. Soc. (Basilian Section) Dec., 1949.