

*Anal.* Calcd. for  $C_{15}H_{10}O_7$ : C, 59.6; H, 3.3. Found: after drying at  $170^\circ$  *in vacuo*, C, 59.2; H, 3.8.

The pentaacetate was prepared and melted at  $200^\circ$  (cor.). The mixed melting point with a known sample of the pentaacetate of quercetin was  $200^\circ$  (cor.). Sando gives  $194$  to  $196^\circ$  for the melting point of the pentaacetate of quercetin.<sup>1</sup> The absorption curves of the two samples of the pentaacetate in 95% ethanol were practically identical. The absorption maxima were at 2530 and 2990 Å. with extinction coefficients (*E*, g. per liter, 1 cm.) of 40.0 and 34.5, respectively. Quercetin was regenerated from the pentaacetate preparations. The absorption curves of the regenerated quercetin preparations in 95% ethanol were practically identical. The maxima were at 2570 and 3750 Å. with extinction coefficients of 67.0 and 75.5, respectively, calculated on the dry basis. Grinbaumówna and Marchlewski give 2555 and 3755 Å. for the absorption maxima of quercetin.<sup>3</sup>

The micro-analyses were made by G. Warren Buckaloo and Lawrence E. Brown.

(1) C. E. Sando, *J. Biol. Chem.*, **117**, 45 (1937).

(2) R. Grinbaumówna and L. Marchlewski, *Biochem. Z.*, **290**, 261 (1937).

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## Observations on the Rare Earths. LII. The Preparation of Rare Earth Bromates from the Perchlorates

By HOWARD E. KREMERS<sup>1</sup> AND THERALD MOELLER

In the preparation of rare earth bromates by metathetical reaction between rare earth sulfates and barium bromate,<sup>2</sup> significant quantities of rare earth materials are occluded by the precipitated barium sulfate. Furthermore, the method is complicated by the limited solubility of barium bromate.<sup>2</sup> Reactions between rare earth perchlorates and the more soluble potassium bromate overcome these objections, the precipitated potassium perchlorate showing less tendency to occlude rare earth salts than barium sulfate because of its somewhat greater solubility and consequent slower rate of precipitation. Bromates are also more readily prepared in this fashion than by treatment of rare earth oxides or hydroxides with bromic acid and are suited to fractional crystallization.

### Experimental

Nearly neutral rare earth perchlorate solutions, prepared from yttrium group oxides by action with perchloric acid and containing the equivalent of 15 to 20% rare earth oxide, were treated with powdered potassium bromate and the resulting mixtures boiled for one hour. After being cooled to  $15^\circ$ , the suspensions were filtered and the residues washed with saturated potassium perchlorate solution until the washings were rare earth-free. These precipitates generally contained about 0.1% rare earth calculated as oxide, and never more than 0.5%.

Each filtrate was systematically fractionally crystallized to six fractions after fifteen crystallizations. Analyses of

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(2) James, *THIS JOURNAL*, **30**, 182 (1908).

these fractions by standard methods showed the most insoluble fractions to consist of potassium bromate with traces of potassium perchlorate, the middle fractions to consist of rare earth bromates with traces of potassium bromate, and the most soluble fractions to consist of rare earth bromates with traces of rare earth perchlorates.

During the course of the fractionation, small amounts of potassium perchlorate and basic rare earth bromates precipitated. The latter never amounted to more than 1% of the total fraction, and such precipitations did not prove objectionable.

The preparation of rare earth bromates from perchlorates is more convenient and rapid than the preparation involving barium bromate, but the removal of by-products is not as complete. Since potassium bromate and perchlorate rapidly concentrate in the most insoluble fractions while remaining traces of perchlorate are carried through to the most soluble fractions, fractional crystallization is not impaired. Avoidance of loss of rare earth material in the initial precipitation constitutes the chief recommendation for the method.

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## Sterols from Peruvian Guano

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The sterol present in comparatively large amounts in Peruvian guano and provisionally called "guanosterol" or "guanosterine"<sup>1</sup> is really cholesterol as shown by the m. p. and m. m. p. of both the sterol and its acetate. Marker<sup>2</sup> has shown that the sterol present in largest amount in chicken feces is sitosterol. The cholesterol present in guano reflects the diet of the marine birds which produce the deposits.

**Procedure.**—Three pounds of Peruvian guano was stirred with 4 liters of ethanol at  $40$ – $50^\circ$  for several hours, and the mixture then allowed to stand overnight. The residue was filtered and the filtrate was evaporated. The residue from the evaporation was refluxed with excess alcoholic sodium hydroxide, diluted with water, then extracted into ether. The pale tan sterol obtained by evaporation of the washed ether solution was recrystallized from 30 cc. of ethanol to yield 4.0 g. of cholesterol of m. p.  $142^\circ$  which showed no depression in m. m. p. when mixed with cholesterol. The acetate, prepared in the usual way, melted at  $113^\circ$  and showed no depression in m. m. p. when mixed with cholesteryl acetate.

(1) del Aguila, *Bol. soc. Quim. Peru*, **4**, 199–200 (1938) (*C. A.*, **33**, 2270 (1939)).

(2) Marker and Shabica, *THIS JOURNAL*, **62**, 2523 (1940).

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## Electrophoresis of Rat Sera<sup>1</sup>

By CHOH HAO LI

In the last few years, many investigators<sup>2</sup> have studied the electrophoresis of animal and human sera, but apparently no studies with rat sera have

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(2) Referred to, for example, by H. Svenson, *J. Biol. Chem.*, **139**, 805 (1941).