

FIG. 2.—Optische Absorption der Blanklösungen.

Gemessen wurde in Lange-Kolorimeter mit Blaufilter sofort nach Zusatz der Reagenzien. Für die Uranbestimmung bei diesen Phosphorsäurekonzentrationen, muß die Phosphorsäure vor der kolorimetrischen Bestimmung abgetrennt werden.

#### LITERATUR

- <sup>1</sup> J. E. Currah und F. E. Beamish, *Analyt. Chem.*, 1947, **19**, 609.
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- <sup>3</sup> C. J. Nelson und D. N. Hume in C. J. Rodden: *Analytical Chemistry of the Manhattan Project*. McGraw-Hill, New York 1950, S. 104.

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### The improved preparation of sulphatoceric acid for preparation of standard titrimetric solutions

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PROBABLY the most frequent difficulty met with in popularizing the use of sulphatoceric acid as a standard oxidant involves the preparation of the solution of  $Ce^{IV}$  in sulphuric acid without the troublesome appearance upon standing of an insoluble residual cerium compound.

Ceric hydroxide,  $Ce(OH)_4$ , when properly prepared eliminated this difficulty entirely.

#### PREPARATION OF CERIC HYDROXIDE

Add a saturated solution of ammonium nitratocerate,  $(NH_4)_2Ce(NO_3)_6$ , in water to an excess of reagent ammonia, sp.gr. 0.90, diluted with an equal volume of water, and stir vigorously. Ceric hydroxide,  $Ce(OH)_4$ , is quantitatively precipitated in readily filterable form. Filter precipitate, and wash it free of ammonia and ammonium nitrate. Air dry this precipitate, requiring two to three weeks. Do not carry the dehydration lower than that which corresponds to the formula  $Ce(OH)_4$ , preferably leaving 2 to 3% unremoved moisture. Grind the horny product thus obtained to pass a 100-mesh sieve.

#### PREPARATION OF SULPHATOCERIC ACID IN SULPHURIC ACID

Place 10.4 g of  $Ce(OH)_4$  in a dry 800-ml beaker. Add 50 ml of reagent sulphuric acid. Stir well, best with a magnetic stirring rotor. Now add slowly 150 ml of water. In 30 to 60 seconds the solution will be clear and reddish-orange in colour. If a minute insoluble portion remains, added heat with stirring is required. Dilute to 650–750 ml with water, transfer to a 1000-ml graduated flask and dilute to the mark. An approximately 0.05 normal solution of  $Ce^{IV}$  which is 0.75 formal in sulphuric

acid is obtained (actual standard factor 0.0485 normal). The solutions thus obtained do not form any insoluble product upon long continued storage and their standard factor does not change.

This quality of  $\text{Ce}(\text{OH})_4$  is available commercially and one pound will prepare more than 40 litres of approximately 0.05 normal solution at a cost of less than 20 cents (1s 6d) per litre.

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### Metal complexes of $\text{N}:\text{N}:\text{N}':\text{N}'$ tetrakis-(2-hydroxypropyl)-ethylenediamine

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THE structural similarity of  $\text{N}:\text{N}:\text{N}':\text{N}'$ -tetrakis-(2-hydroxypropyl)-ethylenediamine, \* THPED (Figure 1), to ethylenediaminetetraacetic acid, EDTA, suggests that THPED may form complexes of analytical interest similar to those formed by EDTA.

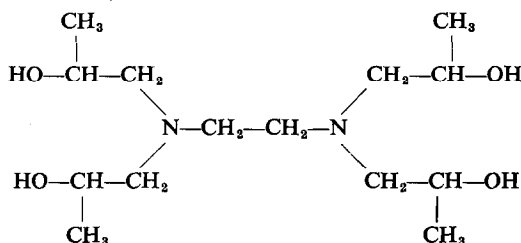


Fig. 1.  $\text{N}:\text{N}:\text{N}':\text{N}'$ -Tetrakis-(2-hydroxypropyl)-ethylenediamine

TABLE I.— $\text{N}:\text{N}:\text{N}':\text{N}'$ -TETRAKIS-(2-HYDROXYPROPYL)-ETHYLENEDIAMINE  
METAL ION COMPLEXES

Cation	Complex	$K_f$	$D_1$	$D_2$	Colour
$\text{Cu}^{++}$	$\text{Cu}(\text{THPED})^{++}$	$2.6 \times 10^9$	$8.8 \times 10^{-7}$	$6.3 \times 10^{-10}$	blue
$\text{Ni}^{++}$	$\text{Ni}(\text{THPED})^{++}$	$7.1 \times 10^8$	$3.2 \times 10^{-10}$	—	blue
$\text{Zn}^{++}$	$\text{Zn}(\text{THPED})^{++}$	$2.2 \times 10^5$	$5.0 \times 10^{-8}$	$1.0 \times 10^{-9}$	colourless
$\text{Cd}^{++}$	$\text{Cd}(\text{THPED})^{++}$	$4.2 \times 10^7$	$3.2 \times 10^{-11}$	—	colourless
$\text{Pb}^{++}$	$\text{Pb}(\text{THPED})^{++}$	$3.1 \times 10^7$	$5.0 \times 10^{-7}$	$8.0 \times 10^{-11}$	colourless
$\text{Co}^{++}$	$\text{Co}(\text{THPED})^{++}$	$5.0 \times 10^5$	$4.5 \times 10^{-8}$	$1.3 \times 10^{-10}$	purple
$\text{Hg}^{++}$	$\text{Hg}(\text{THPED})^{++}$	$1.2 \times 10^8$	$1.0 \times 10^{-7}$	$4.0 \times 10^{-11}$	colourless
$\text{Ag}^+$	$\text{Ag}(\text{THPED})^+$	$2.4 \times 10^4$	—	—	colourless

Constants were determined at  $27^\circ$ , ionic strength  $\mu = 0.05$ . THPED complexes of  $\text{Ni}^{+2}$  and  $\text{Cu}^{+2}$  have been previously reported by Hall *et al.*<sup>1</sup>

A survey of common metal cations ( $\text{Fe}^{+3}$ ,  $\text{Fe}^{+2}$ ,  $\text{Cr}^{+3}$ ,  $\text{Hg}^{+2}$ ,  $\text{Cd}^{+2}$ ,  $\text{Al}^{+3}$ ,  $\text{Bi}^{+3}$ ,  $\text{Pb}^{+2}$ ,  $\text{Zn}^{+2}$ ,  $\text{UO}_2^{+2}$ ,  $\text{Sn}^{+2}$ ,  $\text{Mg}^{+2}$ ,  $\text{ZrO}^{+2}$ ,  $\text{Ba}^{+2}$ ,  $\text{Ca}^{+2}$ ,  $\text{Sr}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Ag}^+$ ,  $\text{Cu}^{+2}$ ,  $\text{Ni}^{+2}$ ,  $\text{Pt}^{+4}$ ,  $\text{Ce}^{+4}$ ,  $\text{Ce}^{+3}$  and  $\text{Th}^{+4}$ ) showed that  $\text{Ag}^+$ ,  $\text{Pb}^{+2}$ ,  $\text{Hg}^{+2}$ ,  $\text{Cu}^{+2}$ ,  $\text{Cd}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Ni}^{+2}$  and  $\text{Zn}^{+2}$  were sequestered by THPED. The formation constants,  $K_f$ , were estimated by potentiometric titrations. The complexes showed acidic properties; and where the acidity was titratable in aqueous solutions with 0.1N NaOH potentiometrically, first ( $D_1$ ) and second ( $D_2$ ) acid dissociation constants were estimated (Table I).

\*  $\text{N}:\text{N}:\text{N}':\text{N}'$ -tetrakis-(2-hydroxypropyl)-ethylenediamine is commercially available from Wyandotte Chemicals Corporation, Wyandotte, Michigan, under the registered trademark "QUADROL."