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PHYSICAL AND CHEMICAL PROPERTIES OF TBP,

by

Mary E. Brennan

March 9, 1951

KNOLLS ATOMIC POWER LABORATORY*
General Electric Company
Schenectady, New York

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The following information has been compiled from sources in the open literature as well as from project reports. It is believed to be complete through

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CHEMICAL PROPERTIES

I. Preparations

- a. $n\text{-BuOH} + \text{POCl}_3$ most common (20), (1), (49)
- b. $3n\text{-BuOH} + \text{POCl}_3 + 3\text{C}_5\text{H}_5\text{N} \longrightarrow \text{PO}(\text{OBu})_3 + 3\text{C}_5\text{H}_5\text{NHCl}$ (19)
- c. $3 \text{ BuI} + \text{Ag}_3\text{PO}_4 \longrightarrow \text{Bu}_3\text{PO}_4 + 3 \text{ AgI}$ (1)
- d. Alkyl phosphites + $\text{SO}_3 \longrightarrow$ Alkyl phosphates (2)
- e. Alkyl phosphites + Ag_2O_5 or H_2SO_4 or V_2O_5 , or
 H_2O_2 or BaO_2 or N oxides or persulfuric acid
 $\longrightarrow$ phosphates (50)

II. Purification

- a. From acid phosphates: wash with 0.1N NaOH followed
by H_2O washes. (94)
- b. From asphaltic impurities: adsorption followed by (53),
solvent extraction (55), (57), (58).
or
distillation in presence of xs alkali to prevent
decomposition of TBP. (63)
- c. From oxidizing impurities: contacting with $(\text{NH}_4)_2 \cdot$
 $\text{Ce}(\text{NO}_3)_6$ in acid solution followed by removal of
Ce with H_2O_2 and H_2O washes. (106)

III. Stability

- a. Stable toward high radiation (ten times that expected
in Column A), on basis of U, Pu, and β extraction
distribution co-efficients. (96)

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- b. No appreciable hydrolysis evident from Pu and β distribution data over a two-week period of storage in stainless steel containers or during six days contact with active IAF. (94)
- c. No apparent hydrolysis (on basis of $E_{a,k}$ of UNH values) to acid butyl phosphates after 20 hours contacting of 15% by volume TBP in Deo-base with 4 M HNO_3 . (71)
- d. Some hydrolysis of TBP does occur on contacting with acid, however, since the amount of Zr extracted increases with the length of time the solvent is pretreated with acid. (101)
- e. Reasonably stable toward concentrated HNO_3 as evidenced by U extraction coefficient after 5 hours refluxing; the increased Pu and β extraction, however, indicated some BuOH formed. (99)
- f. Slight decomposition of 15% TBP in Deo-base after 72 hours contacting with concentrated HNO_3 as evidenced by n_D measurements; but stable toward 1 M HNO_3 , 1 M KOH and distilled H_2O over 2-hour period according to n_D values. (68)
- g. No hydrolysis evident after shaking TBP with two volumes of various organic acids for 1 min. (46a)
- h. Reasonably stable under strong oxidizing conditions as shown by the slow rate of reduction of Ce^{IV} by TBP in 1 M HNO_3 . (106)
- i. Stable toward 0.2 M dichromate - 1 M HNO_3 as shown by n_D measurements after a two-hour contacting of 15% TBP in Varsol with said aqueous phase. The TBP extracts the $\text{Cr}_2\text{O}_7^{=}$. (68)

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- j. Alkyl phosphates in general are more stable at high temperatures (200°C) than alkyl sulfates. (46b)
- k. TBP is decomposed by Na_2O_2 . (38)
- l. Probably stable toward hydrazine and/or hydroxylamine since tri-ethyl phosphate gives no reaction on refluxing with these reagents. (48)

IV Reactions

- a. In general not very reactive. (44)
- b. With strong base $\longrightarrow$ salts of primary and secondary phosphates. (44)
- c. With dry $\text{HCl} \longrightarrow \text{BuCl}$. (40)
- d. With P_2O_5 in varying amounts $\longrightarrow$ pyro, tripoly, tetrapoly or meta phosphates, which in turn readily condense with H_2O , alcohol, amines, etc. (44)
- e. With $\text{POCl}_3 \longrightarrow (\text{BuO})\text{POCl}_2$ or $(\text{BuO})_2\text{POCl}$. (40)
- f. With SO_2 and $\text{Cl} \longrightarrow$ Bu phosphate monosulfonyl chloride. (59)
- g. With benzene + $\text{AlCl}_3 \longrightarrow$ sec - BuPh. (39)
- h. With phenol $\longrightarrow$ Ph OBu + acid phosphates. (37)
- i. With Na acetate $\longrightarrow$ n - butyl acetate. (46b)
- j. Acts as plasticizer for cellulose acetate. (51)

V. Isotopic Exchange

Tracer studies indicate that there is no isotopic exchange of P between the phosphate ion of Na_2HPO_4 and TBP on 20 - 30 hours heating at $100 - 300^{\circ}$ in dioxane solutions in sealed tubes. (64)

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VI. Extraction Properties

- a. Complexes UO_2^{++} (2 moles TBP + 2 moles HNO_3 /mole U) (84)
- b. Extracts short chain aliphatic organic acids from aqueous solutions, the data indicating that TBP is not an associating agent for them. (46a)
- c. Extracts Ce^{IV} , UO_2^{++} , Th, La, Zr, Pr, Br_2 , (Fe). (106)
- d. Extracts $\text{Cr}_2\text{O}_7^{=}$ from 0.2M $\text{Cr}_2\text{O}_7^{=}$ - 1 M HNO_3 into 15% TBP in Varsol. (68)
- e. Extracts H_2SO_4 to the extent of 1% over a concentration range of from 0.1 to 2.0 N acid. (46a)
- f. Extracts HCl to an extent of from 0.8 - 4.2% over a concentration range of from 0.1 to 2.0 N acid. (46a)
- g. Extracts HNO_3 to an extent of from 14.4% to 31.6% over a concentration range of from 0.10 - 2 N acid. (46a)

VII. Methods for the Determination of TBP in Hydrocarbon Solvents

- a. Dielectric constant measurements. (92)
- b. Infra-red method: good to $\pm 0.35\%$; H_2O and UNH don't interfere; HNO_3 corrodes crystal and ther core must be removed. (73)
- c. Colorimetric method. (92)
- d. Specific gravity measurements. (24)
- e. Acid digestion method. (27)
- f. Bomb method. (27)

VIII. Determination of P in TBP

- a. Acid digestion method. (62)

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PHYSICAL PROPERTIES

I. Physical Constants

- a. Melting point: $< -80^{\circ}\text{C}$ (20), (22), (26)
- b. Flash point: 146°C (20), (22)
- c. Molecular heat of vaporization: 14,680 Cals. (35)
- d. Parachor: 629.2 (54)
- e. Dipole moment: 3.05 as compared to 1.92 for butylphosphite. (65)
- f. Surface tension: at 16.6°C = 28.15 (54)
at 20°C = 27.79 dyne/cm (61)
- g. Boiling point: at 760 mm = 264°C (44)
289 $^{\circ}\text{C}$ with decomposition (20), (26)
at 27 mm = 177 - 178°C (20), (22)
at 15 mm = 160 - 162°C (15)
at 6 mm = 138.5°C (54)
- Boiling point equation and values for 10, 50, 100,
150 mm given in reference: (35)
- h. Refractive index: $n_D^{25} = 1.4215$ (20)
= 1.4224 (35)
 $n_D^{20} = 1.4226$ (22)
= 1.42496 (54)
 $n_F^{25} = 1.4274$ (35)
 $n_F^{20} = 1.42988$ (54)
 $n_C^{25} = 1.4203$ (35)
 $n_C^{20} = 1.42295$ (54)
 $n_G^{20} = 1.43343$ (54)

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i. Molecular refraction coefficient. $M_{\frac{n_2}{n_1}}^{20} = 379.50$ (54)

Molecular and group refractivities given in reference: (35)

j. Viscosity: at $20^\circ = 3.885$ centipoises)
 at $25^\circ = 3.422$ ")
 at $30^\circ = 3.021$ ") (36)
 at $35^\circ = 2.695$ ")
 at $40^\circ = 2.437$ ")

k. Specific gravity: at $20^\circ\text{C} = 0.973 - .983$ (22)

 at $d_4^{20} = 0.9766$ (54)

 = 0.979 (15)

 at $d_4^{25} = 0.9727$ (35)

 at $25^\circ/25^\circ = 0.976$ (20), (26)

 at $d_4^{40} = 0.9596$ (35)

l. Solubility: in H_2O : 0.6% (46a), (15), (26)

 1 cc in 165 cc H_2O (20)

H_2O in TBP: $\sim 7.5\%$ by volume at 25° (15), (46a)

 in alcohol, ether, etc.: ∞ (26)

 in various aqueous solutions: (71)

Aqueous Phase	g/l TBP in aqueous phase from pure TBP at 20°C .	g/l TBP in aqueous phase from 15% by volume TBP + Deo-Base:	
		at 20°C	at 50°C
H_2O	0.14	(0.14)	0.18
3 M HNO_3	0.35	0.22	0.30
6 M HNO_3	0.28	0.13	0.22
1 M UNH	0.04	0.04	0.18
RAW	0.10	0.12	0.20

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