benzene but no isopropylbenzene and (2) the dinitro derivative melted 16–17° higher than the literature value.

That the compound was really 1,3-di-*t*-butylbenzene is suggested by the formation of considerable 1,3-dialkylbenzene in other alkylations.[3] This compound, which was first described only recently, has been obtained by aluminum chloride rearrangement of 1,4-di-*t*-butylbenzene,[4] under conditions similar to those producing the so-called 1-isopropyl-3-*t*-butylbenzene,[2] and by synthesis from 2,4-di-*t*-butylphenol.[5] The physical properties of 1,3-di-*t*-butylbenzene correlate well with those of the so-called 1-isopropyl-*t*-butylbenzene, as shown in the following compilation:

	d^t_4	n^t_D	B.p., °C., at *P*, mm.	
1,3-Di-*t*-butylbenzene[4]	.....	1.4870^{20}	106.5	18.2
	.....	......	78.5	4.4
1,3-Di-*t*-butylbenzene[5]	0.8589^{20}	1.4879^{20a}	101	11.2
	$.8547^{25}$	1.4874^{25a}	89.5	6.6
	.....	......	73	2.5
"1-Isopropyl-3-*t*-butylbenzene"[1]	0.8512^{30}	1.4832^{30}	221–222	760

[a] One of these appears in error, perhaps typographically.

The synthesis of 1,3-di-*t*-butylbenzene from 2,4-di-*t*-butylphenol has been repeated. Acetylation of the 1,3-di-*t*-butylbenzene yielded a ketone that gave a 2,4-dinitrophenylhydrazone melting at 211–212° (cor.), in comparison with 208–210° reported.[5] Nitration[6] of the 1,3-di-*t*-butylbenzene gave a dinitro derivative that, twice recrystallized from ether, had a melting point of 164.5–166.0° (cor.), which is practically identical with that of 165.4–166.3° (cor.) reported for the dinitro derivative of the so-called 1-isopropyl-3-*t*-butylbenzene.[1]

Anal.[7] Calcd. for $C_{14}H_{20}N_2O_4$: C, 59.98; H, 7.19. Found: C, 59.93, 60.01; H, 7.39, 7.27.

Reduction of the dinitro derivative with tin and hydrochloric acid[6] gave a diamino derivative that, once recrystallized from isoöctane, had a melting point of 153–154° (cor.), in comparison with 159.1–160.6° (cor.) for the diamino derivative of the so-called 1-isopropyl-3-*t*-butylbenzene.[1]

Anal.[7] Calcd. for $C_{14}H_{24}N_2$: C, 76.31; H, 10.98. Found: C, 75.09, 74.53, 74.14; H, 10.98, 10.90, 10.49. (There was insufficient sample for further purification and analysis.)

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(3) Berry and Reid, THIS JOURNAL, **49**, 3142 (1927); Newton, *ibid.*, **65**, 320 (1943); Simons and Hart, *ibid.*, **69**, 979 (1947).

(4) Johnson and Adams, U. S. Patent 2,429,691 (1947).

(5) Pines, Czajkowski and Ipatieff, THIS JOURNAL, **71**, 3798 (1949).

(6) Ipatieff and Schmerling, *ibid.*, **59**, 1056 (1937).

(7) By Mr. J. E. Puckett.

RESEARCH AND DEVELOPMENT DEPARTMENT
PHILLIPS PETROLEUM CO.
BARTLESVILLE, OKLA.

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The Aqueous Solubility of Hafnium Oxide by the Radioactive Isotope Technique

BY R. A. COOLEY AND H. O. BANKS

Except for thorium oxide whose solubility in water is given[1] as less than 2 × 10^{-5} g. per liter at 25°, little information is available on the solubility of the oxides of group IVa of the periodic table. Since a knowledge of the solubility of the group IVa oxides may be of some value in geochemical, metallurgical and other considerations, we investigated briefly the solubility of hafnium oxide in water by utilizing hafnium 181, a beta and gamma emitter with a half-life of 46 days. Fortunately 99.99% of hafnium 181 decays to stable tantalum 181 so that complications due to radioactive daughter isotopes are not troublesome.

Experimental

The radioactive hafnium oxide was purchased from the Atomic Energy Commission and had been prepared by neutron irradiation of hafnium oxide in a pile. According to the supplier's spectrographic analysis of the sample it contained not more than 0.75% ZrO_2.

The hafnium oxide was placed in contact with distilled water agitated and thermostated to ±0.3° at various temperatures. After 24 to 72 hours it was assumed that equilibrium had been reached and a 200-, 300- or 500-ml. amount of the liquid phase was removed by a previously warmed pipet equipped with a cotton filter. This solution was then usually evaporated in a beaker for radioactivity measurement of the remaining HfO_2. However, another method which may be novel was examined briefly and might prove particularly valuable in cases where a large amount of solution must be evaporated without loss of salt, however small, by deposition on the walls of the container. In this method the container is lined with a thin sheet of polythene before being filled. The solution is then evaporated to dryness under reduced pressure and at a temperature of less than 90°. The polythene sheet is then folded to occupy a minimum of surface on a counting planchet. Toluene, which dissolves polythene, is then added and the toluene–polythene mixture is stirred and finally ignited to leave a uniform layer of the active oxide on the planchet without any layer of unnecessary absorbing material. The substitution of cellophane and acetone for polythene and toluene in the above method did not prove satisfactory since the cellophane was not sufficiently water-proof.

Results

Table I shows the average solubility indicated by duplicate runs at different temperatures. The duplicates agreed within a range of 1.5 to 10%.

Equilibrium was not approached from both sides of the temperatures chosen.

TABLE I

SOLUBILITY OF HfO_2 IN H_2O AT VARIOUS TEMPERATURES

Temp., °C.	Micrograms HfO_2 / 100 g. H_2O
34.6 ± 0.3	2.3
49.7 ± .3	2.8
60.0 ± .3	2.9
70.3 ± .3	3.2
89.7 ± .3	4.7

The experimental points do not obey the van't Hoff equation well, but by the method of least squares the heat of solution was estimated as about 3 kcal./mole.

A few determinations were made in which sodium sulfate or ammonium nitrate were added to the water–HfO_2 mixture and it was found that the solubility of HfO_2 increased as would be predicted by the Debye–Hückel theory.

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(1) A. Seidell, "Solubilities of Inorganic and Metal Organic Compounds," Vol. I, 3rd ed., D. Van Nostrand Co., New York, N. Y., 1940, p. 1533.

UNIVERSITY OF MISSOURI
SCHOOL OF MINES AND METALLURGY
ROLLA, MISSOURI

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