sities of finely powdered substances can be determined with an accuracy which in general agrees with the precision of measurement and is of the order of ±0.1% in cases of favorable ratios of bulk to true density (about 0.5).

The elimination of the air adsorption error by the use of helium is well illustrated by the case of zinc oxide. As is seen in the table, when compression was carried out in helium, the measured density differed from the literature value by −0.29%, but when the determination was carried out in air, the divergence was found to be +15%, even when the sample had been first evacuated and then dry air admitted.

Summary

1. A modification of the Karns densitometer for solid materials has been described, which is applicable to fine powders which do not appreciably adsorb helium and do not possess appreciable vapor pressures at room temperature.

2. The method is accurate to ±0.1% using samples of about 0.5-cc. volume and a ratio of bulk to true density of about 0.5.

CAMBRIDGE, MASSACHUSETTS RECEIVED APRIL 20, 1943

[CONTRIBUTION FROM THE CHEMISTRY LABORATORY OF THE UNIVERSITY OF CALIFORNIA AT LOS ANGELES]

The Solubility of Potassium Iodide in Potassium Hydroxide Solutions at 20°

BY H. DARWIN KIRSCHMAN AND RICHARD POMEROY

During the course of work at this Laboratory it became desirable to determine the solubility of potassium iodide in solutions of potassium hydroxide from 0 to 14.35 *N*. To facilitate the use of these data, density determinations were later made at a number of concentrations.

Brönsted[1] gives the results of seven determinations at concentrations of potassium hydroxide from 9.41 to 15.02 *N*. His results differ from ours by 10–20%. The complete absence of experimental procedures in his report makes it difficult to suggest the cause of this variation.

The results of our measurements are presented in Table I and Fig. 1.

TABLE I

Solubility Data at 20°		Density Data at 20°	
KOH[a] moles/liter	KI moles/liter	KOH[a] moles/liter	Density g./ml.
14.35	0.556	14.35	1.593
13.28	.651	13.28	1.576
12.51	.759	12.04	1.555
11.05	.981	11.11	1.539
10.53	1.107	10.01	1.529
8.814	1.541	9.010	1.524
8.161	1.733		
7.050	2.171	7.767	1.525
6.624	2.292	6.810	1.531
6.453	2.369[b]	6.524	1.533
5.853	2.634		
5.816	2.650	5.002	1.553
4.753	3.145		
4.111	3.485		
4.000	3.525	3.642	1.582
2.405	4.454	2.480	1.613
1.860	4.818		
1.358	5.132	1.386	1.653
0.783	5.539	0.1235	1.703
.000	6.089		
.000	6.109[c]	0.000	1.716[c]

[a] Including about 1% of potassium carbonate as equivalent potassium hydroxide. [b] 0.40 *N* in added carbonates. [c] From Seidell, "Solubilities of Inorganic Compounds," Third edition, Vol. I, p. 812.

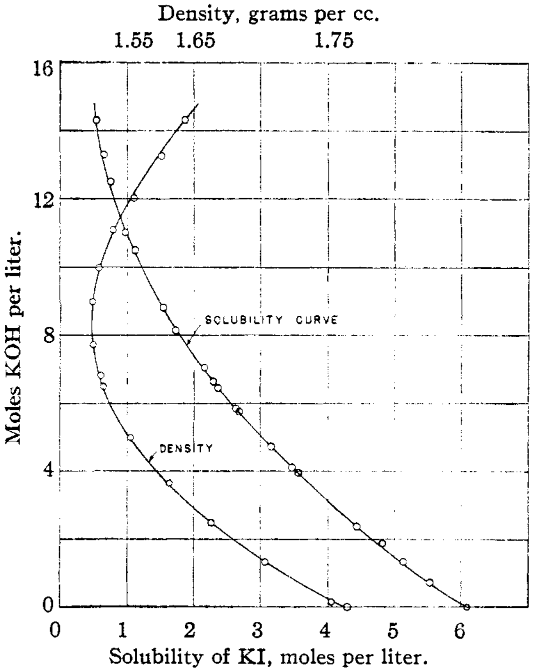

Fig. 1.

Experimental

In general, a saturated solution of potassium iodide in distilled water was added to a warm solu-

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(1) Brönsted, THIS JOURNAL, 42, 1448 (1920).

tion of potassium hydroxide. The sample bottle was immersed in a water-bath from two to seven days and samples removed as convenient for analysis. The alkalinity was determined by titration with sulfuric acid solution using methyl orange indicator. The iodide content was determined gravimetrically as silver iodide. The density was determined by weighing a 25-ml. sample of the saturated solution. Weights were corrected for the buoyant effect of air. All chemicals used were of Reagent grade.

Equilibrium seemed to be quickly attained from the supersaturated condition. In certain experiments, equilibrium was approached from the undersaturated side and no significant variation in solubility was found.

From a number of titrations for alkalinity in the equilibrium mixtures, it was determined that about 1% of the total alkalinity was in the form of carbonate. To estimate the magnitude of the effect of this impurity on the solubility determinations, one solution was prepared with added carbonate to give a carbonate alkalinity of 0.40 *N* in a total alkalinity of 6.453 *N*., *i.e.*, 6.2%. The solubility point determined for this solution lies on the smooth curve drawn through the other experimental points, indicating that small proportions of carbonate are without appreciable effect on the solubility determinations.

Summary

The solubility of potassium iodide at 20° was determined in potassium hydroxide solutions from 0 to 14.35 *N* in alkali. The density and solubility curves show no discontinuities. The accuracy of the results is estimated to be 0.5%.

Pasadena, California Received February 23, 1943

[Contribution from the Baker Laboratory of Chemistry at Cornell University]

Chlorine Azide, ClN_3. I[1]

By W. Joe Frierson, J. Kronrad and A. W. Browne

Although chlorine azide was recognized as a chemical individual so long ago as 1908,[2] only the most meager information concerning its properties and reactions is available at the present time. This is no doubt attributable to the extremely explosive character of the compound, which in undiluted condition usually detonates violently whatever the temperature, without apparent provocation.

A preliminary study of the behavior of chlorine azide toward numerous inorganic and organic compounds has led to the conclusion that this "halogen-halogenoid" compound[3] is chemically very reactive and that further investigation of its properties and reactions is abundantly justified, in spite of the somewhat hazardous nature of the work. Protection against personal injury has been afforded by masks and breast-plates of sheet iron worn by observers during times of danger. Each mask is provided with a rectangular pane (7 × 3 inches) of shatter-proof glass. Although scores of violent detonations have occurred, with resultant demolition of much apparatus, no personal injury has been suffered.

Preparation.—Chlorine azide has been obtained in this Laboratory either (1) by passing chlorine gas into an ethereal suspension of silver azide at ordinary temperature

$$AgN_3 + Cl_2 \longrightarrow AgCl + ClN_3$$

or (2) by the method of Raschig,[2] which involves the gradual addition of acetic acid to equimolecular amounts of sodium azide and sodium hypochlorite in aqueous solution. This reaction may be expressed by the equilibrium

$$HN_3 + HOCl \rightleftarrows ClN_3 + H_2O$$

For the purposes of the present investigation the second method was found to be preferable. In the earlier experiments, in which the chlorine azide, undiluted with air or nitrogen, was distilled from the generator under reduced pressure as recommended by Raschig, frequent explosions occurred unless the operation was conducted very slowly. Later it was found possible greatly to expedite distillation and to minimize danger of explosion by using a current of air or nitrogen at atmospheric pressure. The mixture of gases was dried thoroughly with phosphorus pentoxide, and was then passed either into suitable non-aqueous solvents or into the apparatus in which its properties or reactions were to be investigated.

Analysis.—Since a direct weighing of samples of the pure product seemed out of the question, the analytical

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(1) This article is based upon parts of the theses presented to the Faculty of the Graduate School of Cornell University by W. Joe Frierson in partial fulfillment of the requirements for the degree of Doctor of Philosophy, and J. Kronrad for the degree of Master of Science.

(2) Raschig, *Ber.*, **41**, 4194 (1908).

(3) Gardner and Browne, This Journal, **49**, 2759 (1927).