ciation to Messrs. Max Wolfe and Warren Olsen for assistance in some of the experiments cited here, to the Work Projects Administration for financial support in carrying out this research and the Brittingham Research Foundation for use of their spectrophotometer.

Summary

1. A colorimetric procedure for the determination of semi-micro amounts of molybdenum is given.

2. The effects of various electrolytes on this procedure are listed.

3. To explain the color phenomena observed a four color component mechanism is offered.

4. Spectrophotometric studies of this system are included.

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[CONTRIBUTION FROM THE T. JEFFERSON COOLIDGE, JR., MEMORIAL LABORATORY, HARVARD UNIVERSITY]

The Specific Gravity of Iodine Pentoxide and the Atomic Weight of Iodine

BY G. P. BAXTER AND W. M. KELLEY

On the basis of a new determination of the specific gravity of iodine pentoxide by Moles and Villan,[1] Moles[2] has recalculated the atomic weight of iodine from the ratio $I_2:I_2O_5$ determined by Baxter and Butler[3] and from the ratio $I_2O_5:Na_2CO_3$ determined by Baxter and Hale.[4] The specific gravity found by Moles and Villan, 5.28, differs from that previously found by Baxter and Tilley,[5] 4.80, by nearly 0.5 unit and leads to a vacuum correction somewhat more than 0.002% smaller than that used by Baxter and collaborators. The vacuum corrections corresponding to the two densities are incorrectly calculated by Moles, but the *difference*, which apparently was applied as a correction, is correct. In Moles' paper many of the ratios as well as the atomic weights of iodine are incorrectly calculated. Furthermore Moles arbitrarily rejects the highest result in one series while retaining the lowest which differs nearly as much from the mean.

Apart from the question of the density of iodine pentoxide Moles' treatment of data is incorrect. The weights of iodine pentoxide given in the papers by Baxter, *et al.*, have been subjected to two corrections, one of +0.00011 g./g. for buoyancy of air and one of −0.00001 g./g. for air adsorbed on the powder. The latter correction was found in this Laboratory by weighing iodine pentoxide first in vacuum, then in air of known density. To compute the air displaced by the iodine pentoxide the density 4.80 was employed. If the density 5.00 is used for iodine pentoxide the apparent air adsorption disappears in these experiments, while with the density 5.28 the apparent adsorption is *negative* in sign to the extent of more than 0.001%.

Obviously if the higher density is used in computing the vacuum correction, the correction for air adsorption should be omitted. The correction applied by Moles to the weights of iodine pentoxide of −0.002% should therefore be reduced by the amount of the correction already applied for air adsorption, −0.001%, and therefore is twice too large.

The large difference between the specific gravities of iodine pentoxide found by Baxter and Tilley and by Moles and Villan, nearly 0.5 unit, is hard to understand even when the difficulty of filling with liquid the interstices of the very porous iodine pentoxide, formed by a process of double efflorescence, is taken into consideration. We have therefore made new determinations of the specific gravity of iodine pentoxide by displacement of various liquids. While these new experiments have given results higher than that found by Baxter and Tilley, they are still far lower than that found by Moles and Villan.

Recrystallized iodic acid was carefully dehydrated in two steps in a current of dry air, finally for two hours at 240°. The product was only very slightly discolored, an evidence of considerable purity. It was then rapidly transferred to the pycnometer and weighed. After being covered with the liquid it was placed under a bell jar, which was exhausted, and the air in the powder was dislodged by jarring. This process always required some time. The pycnometer was then set in the usual way and weighed. With xylene the setting remained constant over twelve hours, although there was evidence of slight attack of the xylene by the iodine pentoxide. With mesitylene there

(1) Moles and Villan, *Anales soc. espan. fis. quim.*, **34**, 787 (1936).
(2) Moles, *ibid.*, **34**, 859 (1936).
(3) Baxter and Butler, THIS JOURNAL, **53**, 968 (1931).
(4) Baxter and Hale, *ibid.*, **56**, 615 (1934).
(5) Baxter and Tilley, *ibid.*, **31**, 213 (1909).

was a noticeable though unimportant shrinkage with time. This may have been due to slow penetration of the liquid into interstices of the solid. But in addition there was even more evidence of attack than with xylene. With kerosene there was less evidence of attack but the liquid in the pycnometer *expanded* slightly but steadily with time. With chlorobenzene the original setting was permanent and there was little if any evidence of attack. On the whole the experiment with chlorobenzene was the most satisfactory.

The xylene, mesitylene and chlorobenzene were particularly pure specimens. The first two were dried over sodium, the third over calcium chloride, and all were distilled shortly before use. The kerosene was a cut between 180 and 250°. It was shaken many times with concentrated sulfuric acid, washed with water, dried over sodium and redistilled, again with a cut between the same temperatures. Since the liquids were subjected to low pressure during the filling of the pycnometer the densities of the liquids were found both as prepared and after exposure to low pressure as in the experiments with iodine pentoxide. The latter produced no effect with xylene and mesitylene but raised the density of the kerosene and chlorobenzene slightly. The values obtained after the vacuum treatment were used in computing the specific gravity of iodine pentoxide although at most the difference in the specific gravities of iodine pentoxide computed with the two densities was not over 0.002.

TABLE I

THE DENSITY OF IODINE PENTOXIDE

Liquid	Specific gravity of liquid	I_2O_5 in vacuum, g.	Liquid displaced in vacuum, g.	Specific gravity of I_2O_5
Xylene	0.85502	36.5544	6.3694	4.907
Mesitylene	.85925	37.5522	6.5785	4.905
Kerosene	.76940	32.8083	5.0978	4.952
Chlorobenzene	1.1008	29.9697	6.6242	4.980

Since the most serious difficulty in finding the specific gravity of iodine pentoxide lies in securing penetration of all the interstices of the solid with liquid, the determination of density by displacement of a gas (air)[6] seems worthy of attention. With a different object this has been done first by Baxter and Tilley[7] and later in this Laboratory by Dr. A. C. Titus (unpublished). Baxter and Tilley found that 25.84 g. of iodine pentoxide displaced 0.00633 g. of air at 19° and 758 mm. and 0.00647 g. of air at 20° and 768 mm. These figures lead to the densities 4.90 and 4.84. Humidity was not noted in these experiments. An assumption of 60% relative humidity has been made in the calculations.

In similar experiments by Dr. Titus the air admitted to the tubes had been dried. In ten experiments with 24.161 g. of iodine pentoxide ten values for the density between 5.021 and 4.921 with an average of 4.983 were obtained. In view of the considerably lesser accuracy of this method the agreement of the values obtained by displacement of air with those found by displacement of a liquid is as close as could be expected.

Using as the density of iodine pentoxide 4.98 the vacuum correction for iodine pentoxide at Cambridge, Massachusetts (average density of air, 1.200; weights, 8.3) is +0.000096. In all but one of the papers by Baxter and others the vacuum correction, based on the old, evidently erroneous, specific gravity of iodine pentoxide 4.799, was taken as +0.000106, but in addition a correction of −0.00001 was applied for adsorbed air. The algebraic sum of these corrections is +0.000096, which is identical with the vacuum correction calculated from the new specific gravity, so that the weights of iodine pentoxide as given in the papers require no appreciable correction. In the work of Baxter and Tilley[5] the weights of iodine pentoxide should be decreased by 0.000010 g. per gram since no correction was applied for adsorbed air.

(6) Baxter and Hilton, THIS JOURNAL, **45**, 700 (1923).

(7) Baxter and Tilley, *ibid.*, **31**, 215 (1909).

Summary

1. It is pointed out that Moles' correction of the weights of iodine pentoxide found by Baxter and others, on the basis of a new determination of the specific gravity of iodine pentoxide, is unjustified.

2. The specific gravity of iodine pentoxide has been redetermined by displacement of various liquids, with results falling between 4.905 and 4.980. The latter value on the whole seems most reliable.

3. The specific gravity of iodine pentoxide found by displacement of air is 4.98.

4. With the new specific gravity of iodine pentoxide 4.98 no correction is necessary for the weights of iodine pentoxide given by Baxter and others, except in the work of Baxter and Tilley.

5. The specific gravity of iodine pentoxide found by Moles and Villan appears to be nearly 0.3 unit too high, while that found by Baxter and Tilley is 0.2 unit too low.

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