

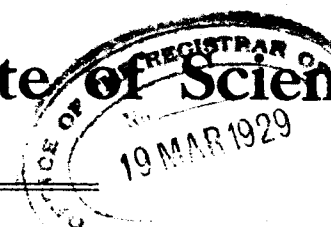
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[Vol. 11A, Part XIII, pp. 161 to 172.]

JOURNAL

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K. R. Krishnaswami.

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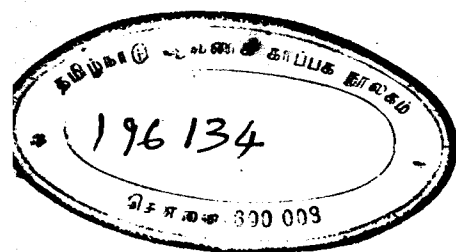
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THE ATOMIC WEIGHT OF ANTIMONY FROM DIFFERENT SOURCES.

By K. R. Krishnaswami.*

The atomic weight of antimony has been determined by numerous observers, a brief outline of the methods employed and the results obtained being as follows.

Berzelius (*Pogg. Ann.*, 1826, 8, 1) treated metallic antimony with nitric acid, ignited the residue and weighed as the oxide Sb_2O_3 . He gives no details except that 124.8 parts of oxide were obtained from 100 parts of the metal, and he calculates the atomic weight to be 129. Kessler (*Ibid.*, 1855, 95, 204) determined volumetrically the amount of oxygen necessary for the conversion of trivalent compounds of antimony to the pentavalent state by employing potassium chlorate and potassium permanganate as oxidising agents. He also sought to determine the ratio $\text{SbCl}_3 : 3\text{AgCl}$. The several determinations comprising over thirty experiments gave values ranging from 122.1 to 122.3 for the atomic weight of antimony. Schneider (*Ibid.*, 1856, 97, 483; 1856, 98, 293) determined the percentage of sulphur in the trisulphide by reducing a specimen of fairly pure natural stibnite in a current of hydrogen. He conducted eight experiments using in each case three to ten grams of the material, and applying corrections for errors due to impurities in the materials as well as to imperfections in the experiments he obtained the mean value 122.55. Rose (*Ibid.*, 1856, 98, 455) published the result of a single analysis of antimony trichloride made under his supervision by Weber, from which he calculated the value 120.7. Dexter (*Ibid.*, 1857, 100, 563) improved the method of Berzelius and using materials purified with great care he carried out thirteen experiments which yielded very concordant results with a mean of 79.283 as the percentage of metal in the tetroxide. From this the atomic weight was calculated to be 122.46.

Dumas (*Ann. Chim. Phys.*, 1859, 55, 175) determined the ratio $\text{SbCl}_3 : 3\text{AgCl}$ using antimony trichloride produced by the action of dry chlorine on metallic antimony and distilled several times over antimony powder. Seven determinations gave him the mean value 121.8. Unger (*Arch. Pharm.*, 1871, 197, 194) analysed Schlippe's salt ($\text{Na}_2\text{SbS}_4 \cdot 9\text{H}_2\text{O}$) and obtained the value 119.8. Cooke (*Proc. Amer. Acad. Arts Sci.*, 1877, 13, 1; 1880, 15, 251; 1881, 17, 1) conducted several series of experiments along different lines. In all he made

* Reprinted with additions from the *Journal of the Chemical Society*; 1927, 2534.

thirteen determinations of the percentage of sulphur in the trisulphide, seventeen of the ratio $\text{SbCl}_3 : 3 \text{AgCl}$, fifteen of $\text{SbBr}_3 : 3 \text{Ag} : 3 \text{AgBr}$, and seven of $\text{SbI}_3 : 3 \text{AgI}$. In the first series the red sulphide pointed to the value 119.994 but when converted to the purer black variety the value obtained for the atomic weight was 120.295, supporting Schneider's value (120.55). The analysis of the chloride, on the other hand, gave 121.89 in close agreement with Dumas (121.8). The analysis of the bromide and iodide, however, yielded very concordant results establishing the value 120.00. Rejecting the values obtained with the chloride as being vitiated by the constant impurity of the oxychloride as well as by the solubility of silver chloride in the wash-water, and combining the other results, he obtained the mean value 120.00.

F. W. Clarke (*Smithsonian Mis. Coll., The Constants of Nature*, 1882, 5), considered the results of Cooke and of Schneider to be very reliable and rejected altogether the data of Dexter, Dumas, and Kessler. He favoured 120.2, the mean of the Cooke and Schneider values adopted in 1903 by the International Committee on Atomic Weights. Brauner (*Abegg Handbuch*, 1907, 5, 556) bestows great praise on the work of Cooke and credits it with having opened a new era in atomic weight inquiry, but Willard and MacAlpine (*J. Amer. Chem. Soc.*, 1921, 43, 797) and Hönigschmid (*Z. anorg. Chem.*, 1924, 136, 257) have pointed out that the results of Cooke are not trustworthy; because it is certain that, owing to insufficient precaution, his halides always contained some oxyhalides which would yield low values for the atomic weight.

Pfeiffer (*Annalen*, 1881, 209, 173) and Popper (*Ibid.*, 1886, 233, 153) determined the electrochemical equivalent of antimony by comparing the amounts of antimony and silver thrown down by the same current in the same time. The values obtained ranged from 121.15 to 122.30. Bongartz (*Ber.*, 1883, 1942) converted metallic antimony into the sulphide and estimated sulphur in the product by oxidation with hydrogen peroxide and weighing as barium sulphate. His estimate of the atomic weight was 120.63. Friend and Smith (*J. Amer. Chem. Soc.*, 1901, 23, 502) converted tartar emetic into potassium chloride by heating in a current of hydrogen chloride. Employing about two grams of the salt for the conversion they made eight determinations of the ratio of potassium antimonyl tartrate to potassium chloride: the values ranged from 120.311 to 120.385 which on recalculation gives the mean value 120.43. Winkler (*Diss. Techn. Hochschule, München*, 1917) determined the weight of the oxide Sb_2O_3 formed from a known weight of the metal. He undertook several preliminary studies on the purification of antimony and the temperature to which the oxide Sb_2O_3 had to be heated in order to obtain the tetroxide Sb_2O_4 in a condition fit for

weighing. The values obtained from four determinations, in each case made with about five grams of the metal, were 121.460, 121.430, 121.444, and 121.448, giving the mean value 121.44. Willard and MacAlpine (*loc. cit.*) determined the value of the ratios $\text{SbBr}_3 : 3 \text{Ag} : 3 \text{AgBr}$. Employing all modern refinements for the preparation of materials in a state of high purity and conducting their analysis by methods of great precision, they arrived at 121.773 as the mean of nineteen determinations.

Muzaffar (*J. Amer. Chem. Soc.*, 1923, 45, 2009) carried out determinations of the ratio $3 \text{Sb} : \text{KBrO}_3$ with specimens of antimony prepared according to the method of Willard and MacAlpine from stibnites from Hungary, Borneo, Peru and Bolivia. He found an atomic weight varying from 121.144 to 122.374 according to the source of the metal. Schreiner (*Tids. for Kemi Bergvesen.*, 4 & 5, 1924) obtained values for the ratio $\text{SbBr}_3 : 3 \text{Ag} : 3 \text{AgBr}$ by following the method of Willard and MacAlpine. Eight determinations were made which gave the mean value 121.73. Weatherill (*J. Amer. Chem. Soc.*, 1923, 46, 2437) obtained values for the ratio $\text{SbCl}_3 : 3 \text{Ag}$ by the nephelometric method. The great merit of this work is that it involved no transference of material or filtration during analysis. Ten concordant results gave the mean value 121.748. Hönigschmid, Zintl, and Linhard (*loc. cit.*) carried out determinations of the ratios $\text{SbBr}_3 : 3 \text{Ag} : 3 \text{AgBr}$ and $\text{SbCl}_3 : 3 \text{Ag} : 3 \text{AgCl}$. The general method adopted was essentially that of Willard and MacAlpine but additional precautions were taken during the preparation of the halides. All the four ratios yielded an identical mean value of 121.76.

The results of recent workers are in excellent agreement, but in all cases the antimony used was obtained from Kahlbaum. Muzaffar's results, however, indicate that there may be distinct variations with the source of the materials. In order to see if similar variations were to be found with antimony from a different part of the world, determinations have been made with four ores of Indian and Burmese origin, whilst for comparison parallel determinations have been conducted with a sample of Kahlbaum's antimony. The results agree very closely with the more modern determinations, and the variation for different ores is within the limit of experimental error.

EXPERIMENTAL.

A few trials were made of Muzaffar's method, but in spite of its apparent simplicity certain disadvantages became evident in practice, and it was abandoned. These attempts will be referred to later. The method of Willard and MacAlpine was then adopted in its entirety and

found very satisfactory. Antimony tribromide is prepared in an atmosphere of nitrogen and distilled in a vacuum into a bulb which is sealed, weighed, and finally broken under a solution of tartaric acid; the quantity of bromine is then estimated by conversion into silver bromide, the amount of silver required being determined nephelometrically or weighed as bromide.

PREPARATION OF MATERIALS.

The various processes of purification were all conducted in a glass hood in a special laboratory free from dust and fumes. The vessels employed were either of silica, pyrex or the very resistant T-glass of Greiner and Friedrichs. The nitric acid, tartaric acid, and sodium and potassium cyanides offered no difficulties in purification. When examined with the nephelometer they were all found to be free from halogen.

Silver.—The silver was prepared according to the method recommended by Richards and Wells (*J. Amer. Chem. Soc.*, 1905, 27, 472), consisting of six recrystallisations of a high grade sample of silver nitrate from dilute nitric acid solution, and precipitation of the silver with pure ammonium formate, prepared from redistilled ammonia and formic acid; the metal was thoroughly washed, dried and fused into buttons in a boat of pure lime in the vacuum from a 'Hyvac' pump.

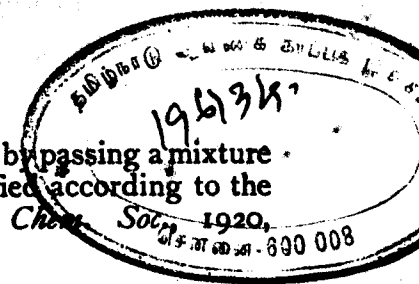
Bromine.—The starting material was Merck's pure bromine which contained traces of iodine; it was purified by the method of Baxter, Moore, and Boyleston (*J. Amer. Chem. Soc.*, 1912, 34, 259) as modified by Willard and MacAlpine.

Sodium Chloride.—Kahlbaum's pure product was twice reprecipitated from a saturated solution by a stream of pure hydrogen chloride. It was freed from mother-liquor by centrifugal drainage, fused in a platinum dish, powdered in an agate mortar and kept in a desiccator.

Water.—The distilled water of the laboratory was boiled for some time with alkali and potassium permanganate and then distilled with a quartz condenser. After discarding the first fraction it was collected and redistilled, being always prepared just before use and kept in vessels of Jena glass or T-glass.

Hydrogen was generated by electrolysis of a 15 per cent. solution of sulphuric acid between platinum electrodes, and purified by passage over heated platinised asbestos and copper gauze, bubbling through a 20 per cent. solution of caustic potash containing some dissolved lead oxide, and drying by passage over phosphorus pentoxide (see Noyes, *Bull. Bureau Standards*, IV, 179).

Nitrogen was prepared in an all-glass apparatus by passing a mixture of air and ammonia over heated copper, and purified according to the method of Richards and Krepalka (*J. Amer. Chem. Soc.*, 1920, 42, 2225).



BALANCE AND WEIGHING.

A long-beam Oertling balance was employed. Successive weighings of the same object, which were made by substitution, agreed to 0.02 mg. The weights were carefully calibrated and counterpoises were used whenever necessary. Calcium chloride was kept in the balance case and 50 per cent. humidity was assumed when calculating the vacuum correction.

PREPARATION OF ANTIMONY.

Five specimens of antimony were prepared from the following sources: (1) Kahlbaum's purest antimony trioxide, (2) stibnite from the Mysore State, (3) cervantite from the same area, (4) stibnite from the Amherst District in Burma, and (5) stibnite from the Southern Shan States.

The method of preparing the metal was the same in all cases. About 500 gms. of the finely powdered material were digested with hot concentrated hydrochloric acid in successive portions until extraction was practically complete. The excess of acid was distilled off and the antimony trichloride distilled 4 or 5 times. It boiled regularly at 210–211°/680 mm., and was always obtained as a clear, pale yellow liquid solidifying to an ivory-white solid. The further steps were according to the method devised by Groschuff (*Z. anorg. Chem.*, 1918, 103, 168). The trichloride is treated with chlorine and hydrogen chloride to give chloroantimonic acid, SbCl_3 , HCl , $\frac{1}{2} \text{H}_2\text{O}$; this is dissolved in a little water, and large dilution with water then precipitates antimonious acid, which is washed, evaporated to dryness with nitric acid to expel the last traces of chlorine, and ignited. The oxide thus obtained is reduced with sodium cyanide in a porcelain beaker in a muffle furnace at about 650°. The buttons of metal obtained are cleaned as recommended by Muzaffar, powdered in an agate mortar, dried in a current of hydrogen at 400°, and sealed in the containing tube.

PREPARATION OF ANTIMONY BROMIDE.

Antimony bromide was prepared in an apparatus very similar to that used by Willard and MacAlpine. The essential parts are shown in Fig. I. The parts C to L were thoroughly dried by heating and evacuation. Pure nitrogen was admitted through the stopcock M after passage through the purifying train, consisting of red-hot copper

gauze in the tube Q, soda-lime in the tube P, sulphuric acid which had been previously boiled with ammonium sulphate in O, and resublimed phosphorus pentoxide in N. The stopcock of the separating funnel was opened and the current of nitrogen passed through the apparatus for 3-4 hours, the various portions of the apparatus being kept hot all the time. Then the tip of the tube E was broken to admit 20-30 gms. of coarsely powdered antimony which had been previously dried in hydrogen. The tube was then sealed off and the apparatus evacuated and filled again with nitrogen, thus ensuring the total absence of air in the system. The bromine bulb C was then cooled to room-temperature and a quantity of bromine (10 to 12 c.c.) slightly less than that required to combine with all the antimony was run into it through the separating funnel in which some phosphorus pentoxide had just then been placed. Great care was taken to prevent entry of air into the bulb. The bromine bulb was then sealed at the constriction. A beaker of water at about 50° was kept under the bulb, when the bromine began to distil very slowly into the preparation chamber (kept at 120-130°) containing the antimony. The antimony bromide being in a molten state drained down to the lower part of the tube leaving fresh surfaces of the antimony exposed to the action of the bromine. When most of the bromine had distilled (usually in 2½ to 3 hours) the preparation tube was sealed at the constriction D. The temperature of the tube was then raised to 160-170° and the digestion of the materials allowed to proceed overnight for 14-16 hours when the antimony bromide appeared as a clear, pale-yellow liquid. The preparation tube was next cooled to about 140° and evacuated, the bromide slowly distilling into the flask H, which was kept at about 90°; after most of material had been distilled the flask was sealed at the constriction G. By further evacuation and gentle heating most of the bromide was distilled into the bulb J which was then sealed at I. The apparatus was once again evacuated and sealed at the constriction L when the portion I to L was left in a sealed state and could be easily handled. The instructions of Willard and MacAlpine were followed and adequate samples of the bromide collected in the sampling bulbs and sealed at the constrictions.

The antimony tribromide was always obtained as a clear pale yellow liquid which solidified to a practically snow-white solid.

PURITY OF THE SILVER.

As a check on the purity of the silver, the ratio $\text{AgCl}:\text{Ag}$ was determined by following exactly the procedure of Richards and Wells (*loc. cit.*). This work, and all subsequent operations in which silver salts were involved, were conducted in a special laboratory illuminated by ruby light. Table I shows the results of several determinations.

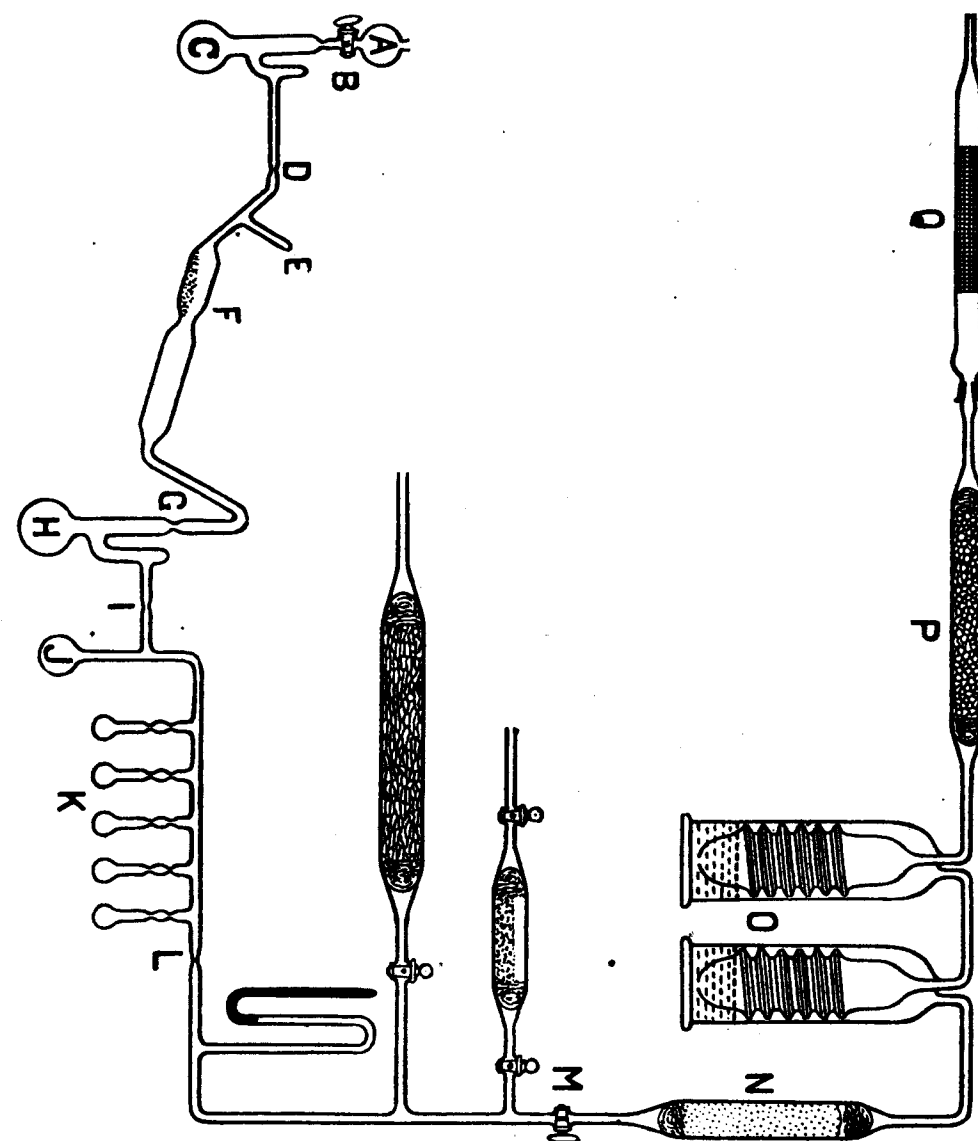


TABLE I.

Ratio AgCl: Ag.

Wt. of Ag, gms.	Wt. of AgCl, gms.	100 X AgCl/Ag	Wt. of Ag, gms.	Wt. of AgCl, gms.	100 X AgCl/Ag
7.4055	9.8547	132.866	7.5700	10.06474	132.850
7.41387	9.85066	132.867	7.74410	10.32871	132.862
7.38879	10.09663	132.870	7.48068	9.93930	132.866

The first two experiments were made with silver from silver nitrate, and the remainder with Johnson and Matthey's pure silver, which is evidently of a remarkable degree of purity for a commercial article. The mean value obtained by Richards and Wells was 132.864 and thus the agreement is as close as can be expected.

WEIGHING AND ANALYSIS OF ANTIMONY BROMIDE.

In this part of the work, also, the general method adopted was the one used both by Willard and MacAlpine and by Hönigschmid. After being cleaned and dried as recommended by Baxter (*Proc. Amer. Acad. Arts Sci.*, 1924-25, 60, 228), the bulb containing the antimony bromide was suspended from the balance by means of a platinum wire and weighed in air and under water. The platinum wire also was weighed in air and in water with the same portion of it immersed as when the bulb was weighed under water. The bulb was then broken with a Pyrex rod under a solution of tartaric acid (250 c.c. of 4-5 per cent. solution) in a thick-bottomed, tall beaker. Solution was complete in about 10 hours.

In Expts. Nos. 1-6, the solution was passed through a Gooch crucible to collect the glass. Subsequently, a silica crucible with a porous sintered bottom was used and proved much more satisfactory. In both cases, the glass was ignited to a dull red heat before weighing. Its density was determined and found to be 2.56 g. per c.c. (T-glass).

The analysis of the antimony bromide was effected by determination of the ratio $\text{SbBr}_3 : 3 \text{ AgBr}$, and in two cases of the ratio $\text{SbBr}_3 : 3 \text{ Ag}$ in addition. For the latter purpose a nephelometer of the Kober type with a Klett top-reader was used.

The gravimetric determination was at first (Expts. 1-6) carried out exactly in accordance with the description given by the authors already mentioned. A Gooch crucible was used, and the solution was refiltered through a small filter-paper to secure any particles of asbestos. As the volume of the solution was about 700 c.c. this was a tedious operation. A great improvement was effected by the use of a Jena-glass crucible with a sintered bottom. In these experiments, the silver

bromide was dried for 14–18 hours at 180°, and the moisture content determined by fusing the bulk of the precipitate in a porcelain crucible. Subsequently, the drying was conducted at 300° for 12–18 hours, as recommended by Hönigschmid, and the loss on fusion of the bromide was then found to be negligible, so that it was not determined in every case. The bromide was, however, always fused and its appearance after fusion noted; it was found that when dark spots or a discoloration appeared (due to traces of silver nitrate) a low value for the atomic weight was usually obtained. Such experiments are distinguished by an asterisk in Table II, and are not included in calculating the mean. In all other cases, the fused silver bromide was quite clear.

As an example of procedure in calculation the details of Expt. No. 7 with antimony from Kahlbaum's antimony trioxide are given below, all weighings being made in air and corrected for weight errors.

Weight of Antimony Tribromide.

	Gms.
Wire with bulb of SbBr ₃	6.14336
„ only	0.33412
Bulb of SbBr ₃ ...	5.80924
* Vacuum correction	0.00384
Bulb of SbBr ₃ (in vacuum)	5.81308
* Vacuum correction—	
Wire with bulb of SbBr ₃ in water	1.883
„ only in water ...	0.333
Bulb in water	1.550
Bulb in air (see above)	5.809
Loss of weight of bulb in water	4.259
Temp. 21.0°, press. 685 mm., d. of brass weights 8.3, vacuum correction, 3.84 mgms.	

	Gms.
Gooch and glass less counterpoise	2.53582
„ less counterpoise	0.17106
Glass	2.36476
Vacuum correction (temp. 20.6°, press. 685 mm., d. of glass 2.56)	0.00066

Glass (in vacuum)	2.36542
Bulb of SbBr ₃ (see above)	5.81308
Weight of antimony tribromide (in vacuum)	3.44766

Nephelometric Determination of the Ratio SbBr₃ : 3 Ag.

If Sb = 121.75, Ag = 107.880 and Br = 79.920, the above weight of antimony tribromide will correspond to 3.0865 gms. of silver. Accordingly, 3.08537 gms. of silver (being 3.08545 gms. diminished by the vacuum correction, 0.008 mgm.) was dissolved in nitric acid and added to the bromide solution. After a sufficient interval 10 c.c. of the clear solution was tested in the nephelometer and a deficiency of silver was observed. A standard solution of silver containing 0.10 mgm. per c.c. was prepared; 5 c.c. was added and still showed a deficiency, which became very slight with a further 5 c.c., equilibrium being reached on the final addition of 1 c.c. Thus 1.12 mgms. of silver (calculated on the original volume of liquid) having been added the weight of silver needed for equivalence was 3.08649 gms., whence the ratio SbBr₃ : 3 Ag = 3.44766 : 3.08649 = 1.11702, and the atomic weight of antimony is 121.751.

Gravimetric Determination of the Ratio SbBr₃ : 3 AgBr.

Weight of silver bromide—

	Gms.
Gooch and glass less counterpoise	5.54433
„ less counterpoise	0.17106
Silver bromide	5.37327
Loss on fusion	0.00056
Add correction for solubility	5.37271
* Add vacuum correction	0.00006
Weight of silver bromide (in vacuum)	5.37296

** Vacuum correction—*

Temp. 22.3°, press. 685 mm., d. of AgBr 6.85, vacuum correction 0.19 mgm.

Loss on fusion was calculated from the loss undergone by 4.772 gms., namely, 0.50 mgm.

Thus the ratio $\text{SbBr}_3 : 3 \text{AgBr} = 3.44766 : 5.37296 = 0.641669$, and the atomic weight of antimony is 121.756.

Table II gives the results of the determinations. For calculating the atomic weight, the values $\text{Ag} = 107.880$, $\text{Br} = 79.920$ have been adopted.

TABLE II.

Expt. No.	Wt. of SbBr_3 , gms.	Wt. of AgBr , gms.	$\text{SbBr}_3 : 3\text{AgBr}$	Atomic weight of Sb.
Kahlbaum's Antimony Trioxide.				
1	4.44079	6.92064	0.641664	121.753
2	3.15437	4.91639	0.641603	121.719
3	3.64246	5.67747	0.641566	121.701
4	3.97152	6.18954	0.641650	121.746
5	3.47255	5.41185	0.641654	121.750
6	3.80868	5.93549	0.641679	121.762
7	3.44766	5.37296	0.641669	121.756
8	4.12164	6.42335	0.641665	121.754
			Mean	121.754
Mysore Stibnite.				
9	4.29146	6.68901	0.641569	121.700
10	4.03746	6.29236	0.641645	121.743
11	3.85444	6.00720	0.641637	121.738
12	4.25750	6.63517	0.641656	121.749
13	4.16876	6.49684	0.641639	121.731
14	3.40170	5.30160	0.641636	121.738
			Mean	121.744
Mysore Cervantite.				
15	3.35625	5.23081	0.641631	121.735
16	3.98107	6.20437	0.641659	121.751
17	3.82381	5.95934	0.641650	121.746
			Mean	121.744
Amherst Stibnite.				
18	4.61160	7.18715	0.641657	121.750
19	4.61851	7.19785	0.641651	121.746
20	4.11282	6.40690	0.641635	121.737
21	3.50233	5.45818	0.641666	121.753
22	3.98801	6.21524	0.641650	121.745
			Mean	121.747
S. Shan States Stibnite.				
23	3.74847	5.84185	0.641658	121.750
24	4.94145	7.70114	0.641652	121.747
25	3.82442	5.96012	0.641668	121.756
			Mean	121.751

In Expts. 7 and 8, the ratio $\text{SbBr}_3 : 3\text{Ag}$ was determined by the nephelometer before filtering the silver bromide. The values 121.751 and 121.735 were found for the atomic weight, the agreement with the gravimetric value being fair. It was not, however, considered necessary to determine this ratio in every case, as it is sufficient for the present

purpose to compare the relative values for the different samples of antimony, the absolute value of the atomic weight being of secondary importance.

The mean results show an extreme variation of 0.010 in the atomic weights; this is considerably less than the variation among individual experiments and may reasonably be ascribed to experimental error. The difference between these results and those of Muzaffar is considerable, this author having found a variation of 0.93 unit in the atomic weight of different samples of antimony. His individual results are not very concordant, the ratio $3\text{Sb} : \text{KBrO}_3$, ranging from 2.1802 to 2.1875 in the case of one sample, corresponding with 0.41 unit in the atomic weight, whilst the preliminary determination of the ratio $3\text{As}_2\text{O}_3 : 4\text{KBrO}_3$, gave results fluctuating over a range of 0.4 per cent., showing that one of these materials was not very pure, or that the end-point of the reaction was not definite.

An attempt was made to investigate the bromate method as conducted by Muzaffar, but a difficulty was experienced in dissolving the pure antimony in concentrated sulphuric acid except by very prolonged boiling. On cooling, the mass became semi-solid, and although solution could be effected by the addition of a little water, too much water produced a precipitate and transference was troublesome. As Muzaffar gives no details of manipulation, it was considered preferable to make use of a method which had been fully described and experiments by the bromate method were not continued.

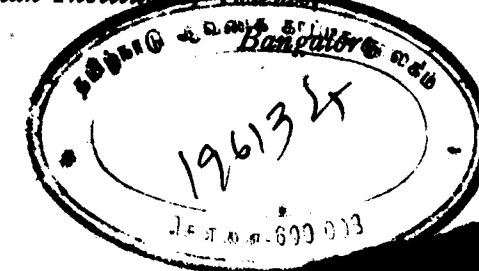
SUMMARY.

The atomic weight of antimony has been determined in five samples, one from Kahlbaum and the others from India and Burma. Willard and MacAlpine's method was used, and the results ranged from 121.744 to 121.754. These are in good agreement with the best previous determinations; there is no indication that the variation is due to a change in atomic weight, and in any case such variation does not exceed 0.01 unit for the samples examined.

In conclusion, I wish to express my gratitude to Professor H. E. Watson for his valuable suggestions during the course of this work, and to the Directors of Geology of the Governments of India and of Mysore for their kindness in supplying the necessary ores of antimony.

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