

even under the most favorable conditions. It is soluble in alcohol, acetone, chloroform, benzene, or toluene; slightly soluble in ether; essentially insoluble in water, or naphtha. The solution of the compound is always attended with decomposition shown by the appearance of a red substance and of a tar if the solution is allowed to stand for some time.

Similar black crystals were obtained, when alcoholic solutions of dimethylamine and tetrachloro-*o*-quinone were mixed after having been cooled with ice. An analysis after washing with water gave 39.50% Cl instead of the 42.27% Cl required, if one molecule of the quinone had combined with two of the amine. Owing to the instability of the substance, which prevented purification, a better result could not be expected. On standing dry or being dissolved with a view to purifying, it was converted into a tar, and it decomposed on heating. It seems, therefore, to be a little less stable even than the corresponding bromine compound, and is less soluble than this, since it is only slightly soluble in alcohol, or acetone, and nearly insoluble in chloroform, tetrachloride of carbon, disulfide of carbon, benzene, or toluene, but soluble in ether.

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[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF HARVARD UNIVERSITY.]

NOTE ON TETRABROMO-*o*-PHENYLENEDIACETAMID.

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Received January 6, 1916.

In some earlier work² in this laboratory an attempt was made to prepare tetrabromo-*o*-phenylenediamine by the action of bromine on the free base, but the only products were amorphous red substances, which were supposed to be formed by the action of bromine on the amino groups. Accordingly in taking up the work again we protected these groups by the introduction of acetyl, and had no difficulty in obtaining tetrabromo-*o*-phenylenediacetamid (not melting below 280°) by the direct action of bromine on dibromo-*o*-phenylenediacetamid. When we attempted to remove the acetyls from this substance with sulfuric acid, we found that a molecule of water was removed instead. This was not unexpected, but we had hoped that some other saponifying agent might be found, which would yield the free diamine. With this end in view we tried a solution of sodium hydroxide in an open vessel, and alcoholic ammonia in a sealed tube, but each gave the same result as the sulfuric acid, that is, a compound melting at 265–266° (uncorr.), which without doubt is the acet

¹ The work described in this paper formed part of a thesis presented to the Faculty of Arts and Sciences of Harvard University for the degree of Doctor of Philosophy by Sydney Adams Beggs.

² Jackson, Russe, *Am. Chem. J.*, 35, 148 (1906).

derivative of the 2-methyltetrabromobenzimidazol of Baczynski and Niementowski.¹

In preparing the dibromo-*o*-phenylenediacetamid on one occasion, when the action was carried on in the cold, we obtained instead the *o*-aminodibromophenylacetamid which melted at 189° (uncorr.).

Experimental.

4,6-Dibromo-2-nitroaniline prepared according to Russe and one of us² was warmed on the steam bath with an excess of tin and strong hydrochloric acid, until the yellow color had gone; and after it had cooled, and an excess of sodium hydroxide had been added, it was extracted with ether, which gave a yield of about 78% of the theoretical, whereas Russe by distillation with steam obtained only a little over 5%. This process, however, had the advantage of yielding a pure product, whereas ours was still impure.

2-Amino-4,6-dibromoacetanilid, $C_6H_2Br_2NH_2NHC_2H_3O$.—When the dibromo-*o*-phenylenediamine was mixed with a slight excess of acetic anhydride, a considerable amount of heat was given off, and the mixture solidified. After it had been allowed to stand for 10 minutes, the product was treated with water, and warmed, until the excess of acetic anhydride had been destroyed. The crude acetamid from 6.5 g. of the amine weighed 7 g. It was purified by frequent crystallization from dilute alcohol, until it showed the constant melting point 189° (uncorr.).

Subs., 0.1479. AgBr, 0.1797. Calc. for $C_6H_2Br_2NH_2NHC_2H_3O$: Br, 51.94. Found: Br, 51.71.

Properties.—White needles from dilute alcohol melting at 189° (uncorr.). It is soluble in alcohol, ether, chloroform, tetrachloride of carbon, disulfide of carbon, benzene, or toluene; essentially insoluble in naphtha.

Tetrabromo-*o*-phenylenediacetamid, $C_6Br_4(NHC_2H_3O)_2$.—The dibromodiacetamid used in this work was made by the method of Russe and one of us,³ warming the amine with acetic anhydride for 15 minutes. We found that it could be crystallized either from dilute alcohol, or dilute acetic acid. Ten grams of this substance were dissolved in glacial acetic acid, and boiled under a return condenser with 10 g. of bromine in the sunlight. During the first 8 hours of boiling there was no apparent action, but when, after standing overnight, the mixture was heated on the following day, hydrobromic acid was given off, showing that the reaction had started. As the boiling went on, more hydrobromic acid came off, and a white solid was deposited; and when after 11 hours no more gas appeared, the product was filtered out, and crystallized three times from dilute alcohol.

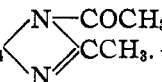
Subs., 0.1113. AgBr, 0.1655. Calc. for $C_6Br_4(NHC_2H_3O)_2$: Br, 62.98. Found: Br, 63.30.

¹ *Zentrbl.*, 1902, II, 940.

² *Am. Chem. J.*, **35**, 149 (1906).

³ *Ibid.*, **35**, 152 (1906).

Properties.—White prisms from dilute alcohol, which did not melt even at 280° . It is soluble in alcohol; slightly soluble in ether; essentially insoluble in water, chloroform, acetone, glacial acetic acid, naphtha, or benzene.

2-Methyltetrabromobenzacetimidazol, C_6Br_4 . — One gram

of tetrabromo-*o*-phenylenediacetamid was boiled under a return condenser with equal parts of sulfuric acid and water for 4 hours, when upon adding sodium hydroxide to the solution a precipitate was thrown down, which after 3 crystallizations from dilute alcohol melted constant at $265\text{--}266^{\circ}$ (uncorr.). The same product was obtained, when 0.5 g. of the diacetamid was heated for 4 hours in a sealed tube with alcoholic ammonia. After evaporating off the solvent, and recrystallization from dilute alcohol, the melting point was 265° (uncorr.). Upon boiling some of the diacetamid for 2 hours with a solution of sodium hydroxide, diluting with water, and extracting with ether, a product was obtained, whose melting point in the crude state showed it was identical with the other two.

Subs., 0.1077, 0.1207. AgBr, 0.1646, 0.1848. Calc. for $C_{10}H_6Br_4N_2O$: Br, 65.30. Found: Br, 65.03, 65.18.

Properties.—White crystals melting at $265\text{--}266^{\circ}$ (uncorr.). It is soluble in all the common organic solvents, but insoluble in water.

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[CONTRIBUTION FROM THE DEPARTMENT OF APPLIED CHEMISTRY, ENGINEERING COLLEGE, IMPERIAL UNIVERSITY OF JAPAN.]

THE ESSENTIAL OIL OF SUGI (*Cryptomeria japonica*) LEAVES.

By So UCHIDA.

Received December 8, 1915.

The Sugi (*Cryptomeria japonica*, Don.) is a coniferous tree, indigenous to Japan, and is extensively cultivated throughout the whole empire as a valuable timber tree. The wood is most widely used and prized as a timber wood and for general woodwork. The stems, leaves and roots contain essential oil. No study, as far as I know, has hitherto been made of the oil contained in the leaves.

The green leaves of Sugi, weighing 87.4 kg., on distillation with steam, yielded 612 g. of volatile oil, which is equivalent to 0.70%. The oil is mobile, brownish yellow in color and has a fresh aromatic odor. When the oil was cooled to -5° for an hour in a mixture of ice and salt, no solid substance separated. A small amount of the oil was distilled in a flask under atmospheric pressure; it began to boil at about 155° and continued to distil until the thermometer reached 350° , the following fractions being obtained: $155\text{--}190^{\circ}$ 33%, $190\text{--}230^{\circ}$ 4%, $230\text{--}270^{\circ}$ 4%, 270--