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# Amyl Nitrite

$C_5H_{11}NO_2$ 117.15

Mixture of nitrous acid, 2-methylbutyl ester, and nitrous acid, 3-methylbutyl ester CAS RN®: 8017-89-8; UNII: 22T8Z09XAK. CAS RN®: 110-46-3; UNII: 5N0U5TUC9Z.

» Amyl Nitrite is a mixture of the nitrite esters of 3-methyl-1-butanol and 2-methyl-1-butanol. It contains not less than 85.0 percent and not more than 103.0 percent of $C_5H_{11}NO_2$.

[**Caution—**Amyl Nitrite is very flammable. Do not use where it may be ignited.]

**Packaging and storage**—Preserve in tight containers, and store in a cool place, protected from light.

**USP Reference standards** ⟨11⟩—

USP Benzyl Benzoate RS

**Identification**—

**A:** The NMR spectrum recorded as directed in the *Assay* exhibits, among other peaks, a doublet with a band centered at about 1 ppm and a multiplet with a band centered at about 4.8 ppm representing methyl protons and methylene protons alpha to the nitrite group, respectively, both relative to the tetramethylsilane singlet at 0 ppm.

**B:** To a few drops of it add a mixture of 1 mL of ferrous sulfate TS and 5 mL of 3 N hydrochloric acid: a greenish brown color is produced.

**Specific gravity** ⟨841⟩**:** between 0.870 and 0.876.

**Acidity**—To 0.30 mL in a glass-stoppered cylinder add a mixture of 0.60 mL of 0.1 N sodium hydroxide, 10 mL of water, and 1 drop of phenolphthalein TS, and invert the cylinder three times: the red tint of the water layer is still perceptible.

**Limit of nonvolatile residue**—Allow 10 mL to evaporate at room temperature in a tared evaporating dish, *in a well-ventilated hood*, and dry the residue at 105° for 1 hour: the weight of the residue does not exceed 2 mg (0.02%).

**Content of total nitrites**—Inject a portion of Amyl Nitrite of suitable volume, but not more than 2 µL, into a suitable gas chromatograph (see *Chromatography* ⟨621⟩) equipped with a thermal conductivity detector. Under typical conditions, the instrument contains a 3-mm × 2-m column packed with a methyl polysiloxane oil, 25% by weight on suitable calcined diatomite, the column is maintained at about 80°, the injection port and detector block are maintained about 10° above the temperature of the column, and helium is used as a carrier gas at a flow rate of about 60 mL per minute. From the area under the curve, calculate the percentage (a/a) of total nitrites, represented by the area under the main peak of the chromatogram, in the Amyl Nitrite taken: not less than 97.0% is found.

**Assay**—

*Solvent:* carbon tetrachloride.

*Internal standard*—USP Benzyl Benzoate RS.

*Procedure*—Transfer 4 to 5 mEq of *Internal standard*, accurately weighed, to a semimicro sampling tube, add 2 to 3 mL of carbon tetrachloride, apply a sampling valve and septum,* thereby sealing the tube, and determine the weight of the sealed assembly. Open the valve, introduce about 500 µL of Amyl Nitrite with a syringe, close the valve, and determine the weight of the sealed assembly when it has attained constant weight. Shake the sampling tube and valve assembly, and transfer about 500 µL of the solution to a precision NMR tube as directed for *Absolute Method of Quantitation* under *Nuclear Magnetic Resonance* ⟨761⟩. With no spinning, or with the spinning adjusted so that the spinning side bands of neither the substance under assay nor the *Internal standard* interfere with the regions to be integrated, record as $A_S$ the average area of the *Internal standard* singlet appearing at about 5.3 ppm, representing the methylene protons of benzyl benzoate, and record as $A_U$ the average area of the multiplet with a band center at about 4.8 ppm, representing the alpha methylene protons of amyl nitrite, with reference to the tetramethylsilane singlet at 0 ppm. Calculate the quantity of $C_5H_{11}NO_2$ in the Amyl Nitrite taken, using 58.57 as the equivalent weight of amyl nitrite ($EW_U$) and 106.12 as that of benzyl benzoate ($EW_S$).

* Suitable sampling tubes, sampling valves, and septums are available, respectively, as catalog Nos. K-749000, K-749100, and K-749102 (50 septums) or K-749101 (100 septums), from Kontes Glass Company, Vineland, NJ 08360.

| Topic/Question | Contact | Expert Committee |
|---|---|---|
| AMYL NITRITE | Documentary Standards Support | SM22020 Small Molecules 2 |

**Chromatographic Database Information:** Chromatographic Database

**Most Recently Appeared In:**
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