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BIS-METHYLTHIOMETHANE, AN ODOROUS SUBSTANCE FROM WHITE

TRUFFLE, TUBER MAGNATUM PICO

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The white truffle Tuber Magnatum Pico is highly appreciated for its unique aroma.

This report deals with identification of bis-methylthiomethane as an odorous principle of this truffle.

One truffle (50 g) was thinly sliced and lyophilized at -10° and 10^{-3} torr., the volatile vapours, mainly water, being condensed at -78° . The first 10 ml of condensate were separated, saturated with sodium chloride, extracted with ethyl ether and the ethereal solution with characteristic smell of truffle was injected at 80° into a L.K.B. 9000 A gas-chromatograph mass-spectrometer (5% ethylene glycol succinate on 80-100 Chromosorb glass packed column).

In this way we could observe presence of a compound, showing in its mass spectrum three peaks, accompanied with isotope peaks at two mass units higher, thus proving the presence of sulphur atoms. Ion at m/e 61 (C_2H_5S) was the base peak, the ion at m/e 108 ($C_3H_8S_2$, molecular ion) was 55% of base peak and that at m/e 35 (H_3S) formed 25% of base peak. Their isotope abundance ratios were respectively 5.3%, 9.1%, 5.2% in good accord with calculated values of 4.4% for one sulphur atom and 8.8% for two sulphur atoms (1).

Another valuable information obtained from mass spectrum was the fragmentation pattern of the molecular ion at m/e 108 showing the elimination of CH_3S fragment from the parent molecule (ion at m/e 61 and triplet at m/e 45, CHS ; m/e 46, CH_2S ; m/e 47, CH_3S).

The absence of a peak at m/e 74 ($M-H_2S$) (2) and insolubility in aqueous alkali excluded that the compound could be a thiol.

Because the mass spectrum of ethylmethyldisulphide was different from that of our compound we conclude that it could be only bis-methylthiomethane.

The latter fact we proved by comparison of mass spectra of the substance from truffle and of an authentic sample of bis-methylthiomethane (3), that we found completely identical.

Owing to the volatility of bis-methylthiomethane and its very poor content in truffle, it could not be characterized directly.

Thus the condensate from 400 g of truffles was treated with dilute potassium permanganate and acetic acid and the solution after filtration was evaporated to a small volume and percolated through a Dowex 50 (H⁺ form) column. The eluted acidic solution was evaporated to dryness and the residue crystallized from water, yielding bis-methylsulphonylmethane, m.p. 142-43°, undepressed on admixture with an authentic specimen (3) (Anal. C 20.98, H 4.68; C₃H₈S₂O₄ requires C 20.92, H 4.68).

The I.R. spectrum of the former compound in nujol was wholly superimposable on that of a synthetic specimen.

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