Crystal structure of 3-(propan-2-yl)-1,3-oxazolidine-2-thione, C₆H₁₁NOS

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Abstract

C₆H₁₁NOS, orthorhombic, P₂₁₂₁₂₁ (no. 19), a = 6.0291(6) Å, b = 10.615(1) Å, c = 11.806(1) Å, V = 755.6 Å³, Z = 4, Rgt(F) = 0.0244, wRref(F²) = 0.0645, T = 100 K.

Source of material

Sodium N-isopropyl-N-hydroxyethyldithiocarbamate (1 g, 5 mmol) was stirred in ethanol (50 ml) and a 50% sodium hydroxide solution (1 ml) was added at which point the solution turned clear. The solution was refluxed at 353 K for 1 h. After cooling to room temperature, the solution was filtered and the same portion of chloroform added. The mixture was homogenised by shaking. The colourless crystals formed after the mother liquor had almost completely evaporated. M.p: 340 K.

Discussion

Functionalised dithiocarbamate ligands such as those containing hydroxyethyl groups, being capable of hydrogen bonding, offer opportunities in crystal engineering [1-4]. During the course of recent studies [4], it was noted that decomposition/cyclisation of S₂CN(iPr)CH₂CH₂OH occurred during the synthesis of metal complexes. Subsequently, it was possible to prove this reaction was base-mediated as outlined in "Source of Material". The oxazolidine ring is close to planar with the deviations from the least-squares plane being –0.0582(11), 0.0563(13), 0.0930(17) and –0.0923(16) Å for N1–C1–O1 to 128.70(11)° for N1–C1–S1, reflecting the influence of the larger thione-S (C1=S1 = 1.6680(15) Å) atom. The sum of the angles about the N1 atom is 359.8° confirming its sp² character. The molecular structure is stabilised by an intramolecular C4–H4/S1 interaction. There are no specific intermolecular interactions of note. The most closely related structure in the literature is that of 3-(3-chlorophenyl)-4,5-dimethyl-4-oxazoline-2-thione which features a formal C=O double bond within the ring [5].

Table 1. Data collection and handling.

Table 2. Atomic coordinates and displacement parameters (in Å²).

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References

1. Benson, R. E.; Ellis, C. A.; Lewis, C. E.; Tiekink, E. R. T.: 3D-, 2D- and 1D-supramolecular structures of \( \text{Zn}[\text{S}_2\text{CN(CH}_2\text{CH}_2\text{OH})\text{R}]_2^2 \) and their \( \{\text{Zn}[\text{S}_2\text{CN(CH}_2\text{CH}_2\text{OH})\text{R}]_2^2 \}(4,4'-\text{bipyridine}) \) adducts for \( \text{R} = \text{CH}_2\text{CH}_2\text{OH}, \text{Me or Et} \): polymorphism and pseudo-polymorphism. CrystEngComm. 9 (2007) 930-941.


