

Synthesis of potassium nitrilosulphonate

Summary of the analysed protocols

$\text{KNO}_2 + 4\text{KHSO}_3 \rightarrow \text{K}_3[\text{N}(\text{SO}_3)_3] + \text{K}_2\text{SO}_3 + 2\text{H}_2\text{O}$	(1)
$\text{KOH} + \text{SO}_2 \rightarrow \text{KHSO}_3$	(2a)
$\text{KNO}_2 + 4\text{KHSO}_3 \rightarrow \text{K}_3[\text{N}(\text{SO}_3)_3] + \text{K}_2\text{SO}_3 + 2\text{H}_2\text{O}$	(2b)
Protocol A¹	
Reaction (R₁): equation (1), 43% exc. potassium bisulphite, water, concentrated ammonia solution and potassium hydroxide (auxiliary substances), 0 °C < T < 100 °C	
Isolation (I₁): filtration (suction) → washing (cold dilute ammonia solution)	
Purification (Pu₁): re-suspension (cold dilute ammonia solution) → filtration (suction) → washing (ethanol → acetone) → drying (air); detection test (with potassium permanganate)	
Protocol B²	
Reaction (R₁): ≡ Pr A (scale enlarged to double)	
Isolation (I₁): ≡ Pr A	
Purification (Pu₁): ≡ Pr A (scale enlarged to double)	
Protocol C³	
Reaction (R₂): equation (1), 43% exc. potassium bisulphite, water and concentrated ammonia solution (auxiliary substances), 0 °C < T < 100 °C	
Isolation (I₁): ≡ Pr A	
Purification (Pu₁): ≡ Pr A	
Protocol D⁴	
Reaction (R₃): equations (2a) and (2b), 14% exc. potassium hydroxide, water (auxiliary substance), above ambient temperature and < 100 °C	
Isolation: not prescribed	
Purification (Pu₂): recrystallization – dissolution (hot water) → cooling → filtration (suction) → washing (cold water → ethanol → ethyl ether) → drying (desiccator); auxiliary substance: potassium hydroxide	
Protocol E⁵	
Reaction (R₃): ≡ Pr D	
Isolation: not prescribed	
Purification (Pu₃): recrystallization (hot potassium hydroxide 1M solution)	

^α → – Sequential

References

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- (3) Adams, D.M.; Raynor, J.B. *Advanced Practical Inorganic Chemistry*. John Wiley & Sons, Ltd: London, 1965, pp. 40.
- (4) Sisler, H.; Audrieth, L.F. Potassium Nitrilosulfonate. *J. Am. Chem. Soc.*, **1938**, *60*, 1947-1948.
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