

# THE DETERMINATION OF INORGANIC SULPHATE IN SODIUM LAURYL SULPHATE

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The official method for the determination of inorganic sulphate in sodium lauryl sulphate is time-consuming. Separation of organic and inorganic sulphate via the lead salts followed by complexometry leads to a rapid and convenient method with readily reproducible results.

SODIUM lauryl sulphate first became the subject of an official monograph in the British Pharmacopoeia, 1948. The specified method<sup>1</sup> for determination of inorganic sulphate requires its separation by dissolving organic sulphate by ethanol followed by a classical gravimetric determination as barium sulphate. A determination commenced on one day cannot be completed before noon of the following day.

An alternative method<sup>2</sup> converts both forms of sulphate to the corresponding benzidine salts. These are then separated by means of their differing solubilities in ethanol, and titrated with alkali. Fischer and Chen<sup>3</sup> have, however, pointed out certain inherent errors in this approach to the determination of (inorganic) sulphate.

It has previously been shown that barium lauryl sulphate is readily soluble in hot water and it was suggested that it might be possible to separate inorganic and certain organic sulphates by this means<sup>4</sup>.

## EXPERIMENTAL

Barium chloride was added to boiling aqueous solutions of synthetic mixtures of sodium sulphate and sodium lauryl sulphate under various conditions of pH, and the resultant precipitates collected and thoroughly washed with hot water. They were then dissolved in excess of ammoniacal EDTA<sup>5</sup> and the excess of the latter titrated with magnesium chloride to Eriochrome Black T.<sup>6</sup> Poor separations and variable recoveries were obtained, possibly due, in part at least, to insufficient ageing of the precipitates.

Barium lauryl sulphate is also soluble in certain organic solvents, for example, formamide and dimethyl formamide. But, attempts to separate it quantitatively from barium sulphate by this means were also unsuccessful. Other cations forming insoluble or sparingly soluble sulphates were then investigated as precipitants and lead (-ic) proved to be suitable. Chloride is usually present in significant quantities in sodium lauryl sulphate, but both lead chloride and lead lauryl sulphate are freely soluble in hot 50 per cent ethanol, whereas the solubility of lead sulphate is greatly reduced under these conditions<sup>7,8</sup>. Separations were thus readily effected.

Lead sulphate is conveniently determined by complexometry<sup>9</sup> and Sporek<sup>8</sup> has recently described the determination of sulphate in ores by

## ASSAY OF INORGANIC SULPHATE IN Na LAURYL SULPHATE

this route. He prefers to depress the solubility of lead sulphate by addition of *isopropanol* and dissolves the precipitate in EDTA by the addition of ammonia; excess EDTA is then titrated with zinc chloride. In a re-appraisal of this approach, we have found that use of an ammonia: ammonium chloride buffer with the EDTA solution is less likely to lead to frothing during the final titration. Furthermore, zinc sulphate has proved to be of greater convenience as the final titrant.

### METHOD

#### *Reagents*

*Lead nitrate solution* 0.1 M. Dissolve 33.1 g. of lead nitrate in water and dilute to 1 litre.

*EDTA solution* 0.1 M. Dissolve 37.225 g. of the purified<sup>10</sup> disodium salt of ethylenediaminetetra-acetic acid in water and dilute to 1 litre. While this is regarded as a primary standard<sup>10</sup>, it may if necessary be checked against dried A.R. sodium sulphate by the proposed method.

*Ammoniacal buffer solution.* Dissolve 8.25 g. of ammonium chloride in water, add 113 ml. of 0.88 ammonia solution and dilute to 1 litre with water.

*0.05 M zinc sulphate solution.* Dissolve 14.4 g. of A.R. zinc sulphate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) in water, dilute to 1 litre and standardise against the above 0.1 M EDTA solution.

#### *Procedure*

Weigh accurately about 1 g. of sodium lauryl sulphate into a 250-ml. beaker. Add 35 ml. of distilled water and warm to dissolve. Add  $2.0 \pm 0.2$  ml. of 5 per cent nitric acid, mix, and then add 50 ml. of ethanol. Heat to boiling on a hot plate and slowly add 10 ml. of 0.1 M lead nitrate solution, with stirring. Cover with a clock glass and simmer for 5 minutes. Allow to settle. If the supernatant liquid is hazy, stand for 10 minutes, re-heat to boiling, and again allow to settle.

While the solution is still almost boiling, decant as much liquid as possible through a 9 cm. no. 41 Whatman paper. Wash four times by decantation, each time using 50 ml. of 50 per cent ethanol and bringing the mixture to the boil. Finally transfer the filter paper to the original beaker, and immediately add 40 ml. of water, 10.0 ml. of 0.1 M EDTA and 5 ml. of ammoniacal buffer solution. Warm to dissolve the precipitate and titrate with 0.05 M zinc sulphate solution to Eriochrome Black T.

Then  $\text{Na}_2\text{SO}_4 = (10f - tb) \times \frac{14.2}{w}$  per cent

where  $f$  = molarity of EDTA solution

$b$  = molarity of zinc sulphate solution

$t$  = ml. zinc sulphate solution required

and  $w$  = g. of sample taken.

### RESULTS

The recovery of lead sulphate was first checked in the absence of organic sulphate. Results were very satisfactory and typical figures are given in Table I.

TABLE I  
RECOVERY OF SODIUM SULPHATE BY PRECIPITATION  
AS LEAD SULPHATE AND DETERMINATION BY EDTA  
AND ZINC SULPHATE

mg. Na <sub>2</sub> SO <sub>4</sub> taken .. ..	25.3	50.6	63.2	75.9
mg. Na <sub>2</sub> SO <sub>4</sub> found .. ..	25.7	50.7	63.0	76.0

For a more realistic check, a sample of sodium lauryl sulphate known to be already of very low inorganic sulphate content<sup>4</sup> was repeatedly extracted into absolute ethanol to free it from inorganic salts<sup>1</sup>. It was estimated finally to contain 0.00012 per cent sodium sulphate. From this highly purified material, synthetic mixtures with A.R. sodium sulphate and of known composition were prepared. The proposed method was then applied to these and typical results are shown in Table II; results by the official method<sup>1</sup> are included for comparison.

## DISCUSSION

Using the proposed method, the (inorganic) sodium sulphate content of sodium lauryl sulphate B.P. can be determined conveniently in 30–40 minutes. This compares favourably with 18–24 hours actual elapsed time by the official method<sup>1</sup>, of which about 1.5 hours represent actual manipulative time. Additionally, in our laboratories the new method has yielded more readily reproducible and more precise results (cf. Table II).

TABLE II

RECOVERY OF SODIUM SULPHATE FROM SYNTHETIC MIXTURES CONTAINING PURIFIED  
SODIUM LAURYL SULPHATE

(Total mixture per determination: 0.95–1.05 g., weighed accurately)

Na <sub>2</sub> SO <sub>4</sub> per cent in mixture	mg. Na <sub>2</sub> SO <sub>4</sub> taken	Proposed method		Official method	
		mg. Na <sub>2</sub> SO <sub>4</sub> found	error per cent	mg. Na <sub>2</sub> SO <sub>4</sub> found	error per cent
Nil	Nil	Nil	—	Trace	—
1.63	15.7	14.9	–5.1	14.6	–7.0
3.17	31.5	32.0	+1.6	33.2	+5.4
4.68	47.2	47.6	+0.8	45.6	–3.4
6.15	63.0	64.0	+1.6	65.9	+4.6
7.52	78.8	80.0	+1.5	79.8	+1.3
9.04	94.4	94.0	–0.4	97.8	+3.6

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