



TECHNICAL GUIDELINES

STABILISATION OF SULFATE-BEARING SOILS

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The British In-situ Concrete Paving Association

Summary

The potential for sulfate-induced expansion in stabilised soils is well documented and understood. To avoid problems, adequate site investigation is needed prior to stabilisation and, in particular, it is essential to test for sulfides as well as sulfates.

These guidelines for the stabilisation of sulphate bearing clays aim to:

- Explain the mechanisms that cause sulphate heave;
- Recommend methods for sampling and testing for sulfates and sulfides;
- Describe measures to minimise the risk of sulfate-related disruption.

This guidance is based on current knowledge and experience.

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The Association has a broad corporate membership base that includes contractors, consulting engineers and designers, suppliers of plant, equipment, and materials, academics and clients, both in the UK and internationally.

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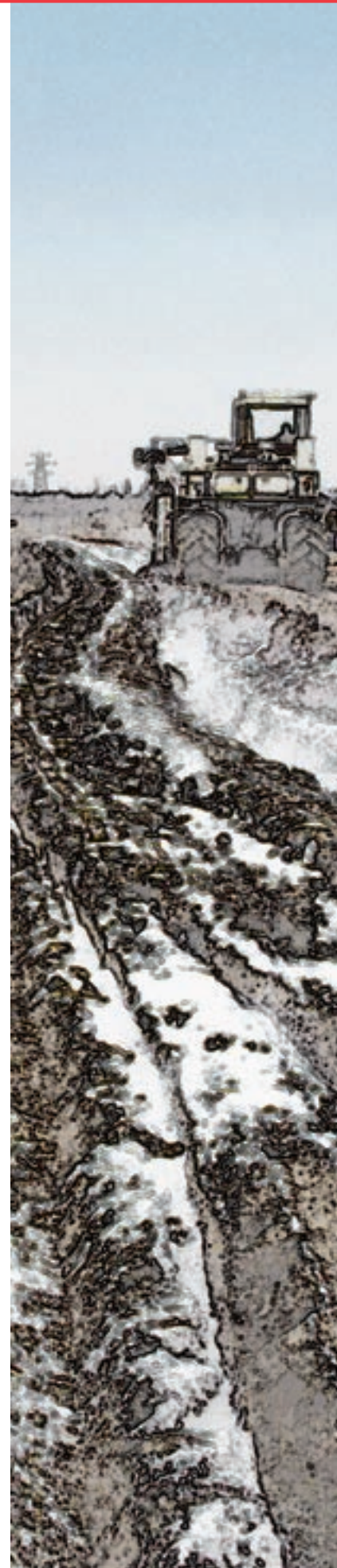
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Guidelines for the stabilisation of sulfate-bearing soil

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1 The sulfate heave mechanism

Since the late 1950s, it has been well known [1] that sulfates can cause expansion problems in soils stabilised with calcium-based stabilisers (e.g. lime and Portland cement). In the presence of water (particularly excess water), the reaction of calcium (from lime or cement), alumina (a primary constituent of clay) and sulfate produces calcium-aluminate-sulfate-hydrate minerals. These have very large expansion potential, in some cases as high as 250%. One of these minerals is ettringite, which holds very large quantities of water within its structure. During its formation, very high swell pressures can develop with disruptive increases in volume.

Sulfides, especially pyrites, also constitute a risk factor for soil stabilisation. The report [2] from the Thaumassite Expert Group, set up to investigate sulfate-related deterioration in concrete, highlighted the extent to which disturbance of a soil can induce pyrites to oxidise and significantly increase the sulfate level. This oxidation is accelerated during soil stabilisation operations by both the pulverisation process and the use of lime and/or cement, which increases the pH level and thereby decreases the chemical stability of the pyrites [3].

2 Sulfates and sulfides in clay

Sulfates and/or sulfides are found in many UK sedimentary strata, including ancient marine deposits such as carboniferous shales, clays and glacial tills, as well as recent estuarine silts and clays. In most affected strata, the top metre or so of undisturbed ground is normally low in sulfate, due to leaching by rainfall. High levels of sulfate are often found around tree roots and at the base of the weathered zone. Their location is dependent on the weathering history and the groundwater flow.

3 Site assessment

The importance of reviewing all readily available information prior to commencement of the relatively more costly intrusive sampling and testing, cannot be overemphasised. A phased investigation will usually be the most efficient approach. The level of detail and time spent on each stage can be varied depending on the scale and nature of the project.

The sampling and testing strategy should be based on a number of factors, including:

- Information already available.
- Any risks identified from the immediately available information.
- The consequences of inadequate performance (different criteria may be appropriate according to the application).

It is often appropriate to target specific areas for sampling and testing, for example:

- Geological strata with potentially high sulfate and/or sulfide contents.
- Locations of high sulfate concentrations (hotspots) resulting from natural weathering of the soil profile [3]. Particular care should be taken to visually identify any weathering zones within soil layers.

In the case of soils to be stabilised in situ, it is important that tests are conducted on samples taken from levels that equate accurately to the material likely to be stabilised. For cuttings, samples should not only be from a location within the stratum equivalent to the proposed stabilised layer but also to at least 500 mm below the base of this layer. Material to be moved before stabilisation may present less risk because mixing of material during excavation and placement is likely to dilute sulfate hotspots.

Digging trial pits often provides a cost effective means to both visually assess the ground conditions and provide good quality samples for subsequent testing. When sampling from deeper levels within the soil (for example from proposed cuttings) or where site access is limited, window sampling and/or boreholes can be used. It may be prudent to take more samples than are initially required, to allow for subsequent targeted testing.

Consideration should also be given to the potential for groundwater to bring in sulfates from outside of the stabilised ground. Testing the groundwater (if present) is useful as an additional pointer to the overall conditions, but should not be used in isolation. An SO_4 concentration in the groundwater in excess of 0.4g/l, the upper limit of 'Design Sulfate Class 1' for groundwater in BRE Special Digest 1 [5], indicates the presence of significant levels of sulfate, but a lower concentration would not necessarily guarantee the absence of sufficient sulfate to cause a problem.

4 Testing for sulfate and sulfide in soils

There are a large number of test methods for the determination of sulfate/sulfide in soils (see Appendix). TRL Report 447 *Sulfate specification for structural backfills* [6] reviewed these and recommended test methods. These take advantage of advances in instrumentation and are quicker and less expensive than the historical 'wet-chemistry' methods. The TRL447 test methods were introduced into Highway Agency Specifications in November 2003 and Britpave generally recommends them as the preferred methods.

To assess a soil for its potential for sulfate-induced expansion, it is helpful to know the following:

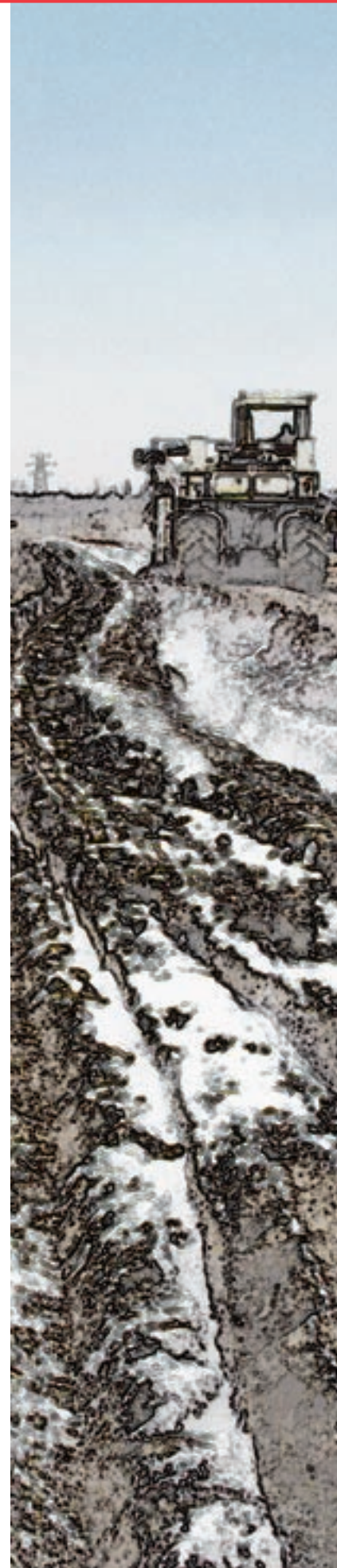
- The acid-soluble sulfate (AS) content, which is a measure of the immediately-available sulfate (use TRL447 Test No. 2).
- The total potential sulfate (TPS) content, which is the total sulfate that would become available if all the sulfide converted to sulfate (use TRL Test No. 4). TPS (expressed as % SO_4) is calculated by multiplying the total sulfur (TS) value (%S) by 3.

The value of a soil's total potential sulfate should always be greater than that for the acid-soluble sulfate (if not, question the testing!). The difference provides a measure of the presence of materials that contain sulfur, but in a form other than sulfate. If the sulfur is present as sulfide (e.g. pyrite) then it can readily convert to sulfate and contribute to heave. However, sulfur in organic or other matter that is unlikely to convert naturally to sulfate is not considered a risk factor with respect to heave. Whilst it is possible to analyse further to distinguish between these forms of sulfur, the necessary tests are complex and expensive. Measuring TPS is therefore a conservative approach, which may significantly overestimate the potential for sulfate heave. Where there is a large difference between TPS and AS, it is usually beneficial to understand the reason. This may require geological expertise or further testing.

5 Trigger levels for sulfate/sulfide

Sulfates and sulfides in the soil occur in many different chemical forms, and there is no simple relationship between sulfate/sulfide content and expansion. This is reflected in Highways Agency Advice Note HA 74/00 *Treatment of fill and capping materials using either lime or cement or both* [7], which suggests that the limits for acceptability of sulfide and sulfate content should be determined on a site-by-site basis, by testing appropriate samples and relating the swelling measured on soaked CBR tests to the sulfate and sulfide content. HA 74/00 recommends that the upper limiting value of total sulfate content should not exceed 1.0% and warns that there is evidence that, for some materials, values as low as 0.25% may cause swelling.

In the United States, the National Lime Association has published a Technical Memorandum [8], setting out a Protocol for lime stabilisation of clay soils containing soluble sulfates. This suggests that where the total level of soluble sulfates is below 0.3% SO_4 the potential for a harmful reaction is low and should not be of significant concern. Above this level the potential for some localised distress due to seams of higher sulfate concentration not detected in testing is considered a 'fact of life'. The National Lime Association Protocol does not cover the possibility of sulfides being present and oxidising to sulfates.



6 Choice of binder to minimise the risk of sulfate/sulfide disruption

Where it has been established that the TPS content in a soil is below 0.25% SO₄, there is minimal risk of sulfate-induced disruption and lime and/or cement can safely be used. However, site investigations may miss isolated hotspots and before totally discounting the risk, consideration should be given to whether the site is in a part of the country likely to contain sulfate/sulfide and to whether the geology has a potential for high sulfate/sulfide concentrations. Figure 1 is reproduced from BRE SD1 [5] and shows the sulfate-bearing strata of greatest significance in the UK.

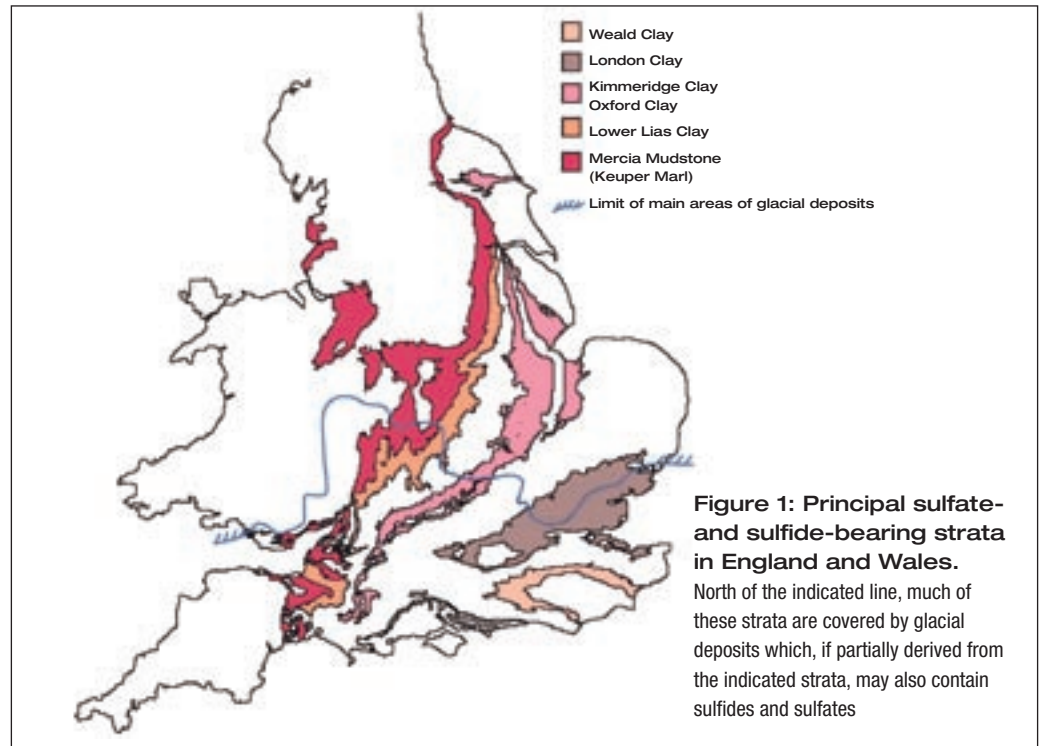


Table 1 summarises information provided in Appendix B of HA 74/00 [7].

Table 1: Geological strata with a potential for high sulfate/sulfide

<ul style="list-style-type: none"> Alluvium Bembridge beds Sandgate beds Woolwich and Reading Beds Gault Clay London Clay Kimmeridge Clay Oxford Clay Weald clay Blue Lias Middle & Lower Lias 	<ul style="list-style-type: none"> Upper Fuller's Earth Mercia Mudstone Edlington Formation Portsmouth Sand Sherwood Sandstone Nursling Sand Whitecliffe sand Carboniferous shales Stonesfield slate Peat Quaternary deposits such as glacial till*
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* Quaternary deposits are unlikely to contain high sulfate/sulfide levels because conditions during deposition were not anaerobic; however some deposition of sulfates may have occurred during formation.

Where the TPS is greater than 0.25% SO_4 , stabilisation with lime and/or Portland cement may involve a risk of sulfate expansion and it would be prudent to consider the use of other binders in combination with lime.

The use of sulphate-resisting Portland cement does not reduce sulfate-swelling in stabilised clays [1]. However, significantly enhanced resistance can be achieved by using ground granulated blastfurnace slag (ggbs) in combination with lime. In laboratory tests on clays containing up to 3% SO_4 [9], combinations of ggbs and lime reduced expansion to within the HA 74/00 allowable limits for capping; even at higher levels of SO_4 , the expansions only just exceeded allowable limits. In a site trial with clay containing around 2% TPS, no disruptive expansion was observed on the ggbs/lime sections but an equivalent section stabilised with lime/PC did heave [10]. Combinations of lime and ggbs have been successfully used on UK sites with clays containing up to 1% AS and up to 2% TPS. The proportion of ggbs should be at least equal to that of the lime (by weight, and assuming use of quicklime, CaO). Increasing this proportion improves the resistance to expansion, and typically for high resistance, a ratio of 3:1 ggbs to lime might be chosen.

It should be noted that there may be other materials or methods besides the use of ggbs that are potentially beneficial options, for example:

- Pulverized fuel ash (pfa).
- Increased mellowing periods.
- A second stage lime addition following an extended mellowing period.

Extensive laboratory testing should be carried out to verify the suitability of these options.

7 Laboratory testing of stabilised mixtures

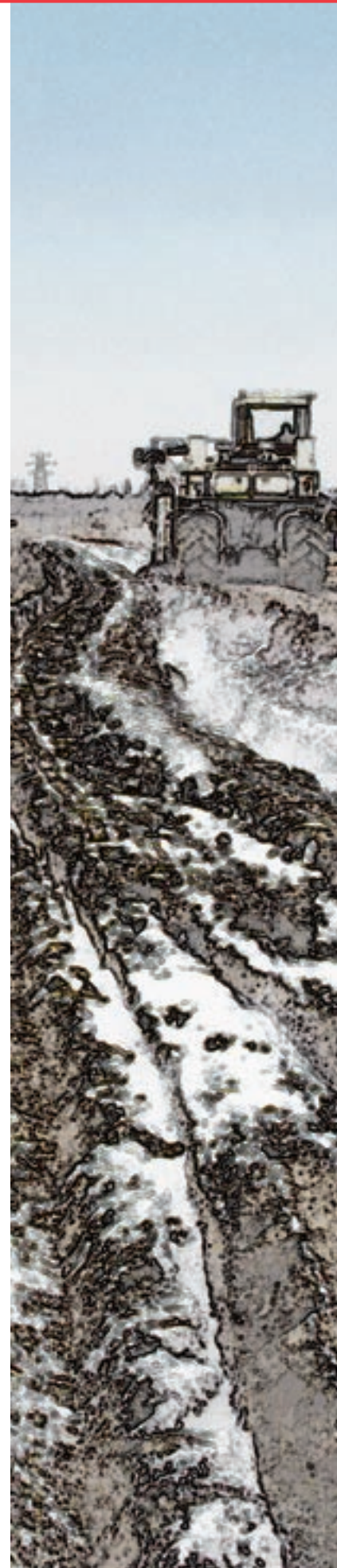
Detailed guidance on laboratory testing can be found in HA 74/00 [7]. Where the TPS is less than 0.25% SO_4 , the choice of the binder(s) will be determined by factors other than sulfate/sulfide content of the soil, and swell testing may not be considered essential.

Where the TPS is above 0.25% SO_4 , the risk of sulfide/sulfate swelling must be a factor in determining the binder(s) selected for test. Swell tests are highly advisable, particularly when the AS is also above 0.25% SO_4 .

HA 74/00 suggests that swell testing should be carried out on soaked specimens prepared and tested in a CBR mould. The recommended limits are that the average degree of swelling should be less than 5 mm (4%), with no individual test specimen swelling more than 10 mm (8%). HA 74/00 emphasises the importance of continuing measurements for at least 28 days to ensure that all swelling has ceased (which normally occurs within 14 days). It states that if swelling is still occurring after 28 days but is still below this limit, some subjective assessment of changes in the rate of swell may be necessary: alternatively the swelling test period could be extended to 56 days provided the rate of swell was declining.

However for swell tests to be fully effective, water and air need to have full access to the specimens. Firstly, ready access of water is needed for the sulfates to cause expansion. Secondly, any sulfides present will not contribute to expansion until air has diffused into the specimens and oxidised them to sulfate. (Expansion that continues past 14 days is indicative of the presence of sulfides). It has been suggested that water and air may not have ready access in the HA 74/00 method because of the protection offered by the CBR mould and the method of soaking employed. The use of unconfined specimens, manufactured in Proctor or MCV moulds, fully immersed in water, perhaps aerated, is considered a more accurate and robust method. A variation of such a test now exists in European Standard BS EN 13286: Part 49.

Where the TPS exceeds 1.0% SO_4 , stabilisation should be carried out only with extreme caution and the risk of swelling must be a major factor in the choice of binder. Extensive laboratory testing, including swell tests, must be employed. Extending the immersion testing to 56 days may be appropriate in situations where significant sulfide is present, because the time required for sulfide to oxidise may delay expansion to the extent that it is not complete within 28 days.



8 Good construction practice for the stabilisation of clays

Irrespective of whether sulfates are present or not, sufficient water must be available to fully hydrate the quicklime as any that remains unhydrated in a well compacted layer is a potential source of future expansion when it hydrates to $\text{Ca}(\text{OH})_2$. To ensure full hydration, the target MCV during mellowing should be 10, but not exceeding 12. At final compaction, the MCV should also not exceed 12.

Where sulfate/sulfide is considered a risk, it is advisable to:

- Avoid the use of soil from areas known to have high sulfate contents.
- Mix soils to dilute any high concentrations of sulfate, e.g. over-dig the formation at the bottom of a cut and replace with other soil, or at the very least with the same soil that has been excavated, maybe stockpiled and then re-laid.
- Ensure good compaction by using the MCVs recommended above. This will achieve a dense layer with minimal air voids thereby reducing the potential for water ingress.
- Employ drainage systems that minimise the potential for subsequent water entry into the layer from either the side, below or above.
- Apply an effective seal coat following final compaction. This will provide some barrier to water, but more importantly will reduce the risk of drying-shrinkage cracks, which would allow easy ingress of water.

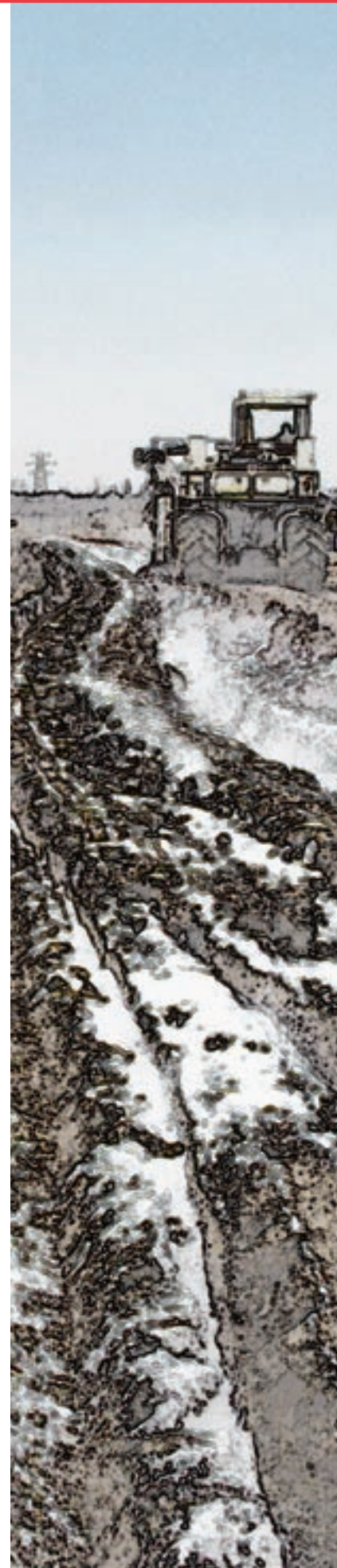
Where a soil is known, or suspected, to contain minor quantities of sulfate, but the level is considered low enough to be acceptable for lime-only stabilisation, it is beneficial to encourage the formation of any expansive sulfate compounds prior to compaction, as any expansive minerals already formed before placement and compaction will not cause disruption. This can be done by delaying the final compaction and providing adequate water during mellowing - the longer the mellowing period and the higher the ambient temperature, the better. Less than 48 hours would probably be ineffective for this purpose. An adequate amount of water is typically 3 to 5 percentage points above the optimum, say an MCV of 10. As usual, second stage mixing and compaction without further stabiliser addition should follow mellowing. If an extended mellowing period is envisaged, this should be reflected in the procedures used for the laboratory testing.

Where lime with ggbs is used, the ggbs is normally mixed in separately from the lime at the second stage. With clay soils, the time between the two mixings need only be sufficient for the lime to modify and mellow the clay such that it loses its cohesivity and can be mixed intimately with the ggbs



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10 Appendix

Testing of soils for sulphates and sulfides

This note is intended for those not familiar with the detail of the chemical testing of soils. It provides background information on the test methods (including those in TRL Report 447) that are commonly used for measuring sulfate and sulfide.

Sulfur may be present in soils as sulfate (SO_4) e.g. gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) or as sulfide (S), e.g. pyrite (FeS_2). It is well established that the presence of excessive gypsum or other sulfates can cause stabilised soils to expand due to ettringite formation. However, sulfides such as pyrite can also be disruptive because they have the potential to oxidise to sulfates and subsequently cause expansion. Consequently when testing soils for soil stabilisation, it is important to establish the content of both sulfate and sulfide.

Historically, testing of soils for sulfate has been carried out in accordance with BS 1377, Part 3: 1990, *Soils for civil engineering purposes: Chemical and electro-chemical tests*. BS 1377 does not contain a test for sulfide, and this has generally been carried out using the test method included in BS 1047 1983, *Specification for air-cooled blast furnace slag aggregate for use in construction*. BS 1047 was withdrawn in June 2004, but an essentially identical test method for determination of total sulfur content appears in BS EN 1744-1: 1998, *Tests for chemical properties of aggregates, Part 1: Chemical analysis*.

TRL Report TRL 447 *Sulfate specification for structural backfills* reviewed the methods for analysing soils for sulfate and sulfide and proposed new test methods, which take advantage of advances in instrumentation and are quicker and less-expensive than the historical 'wet-chemistry' methods. The TRL 447 test methods are now being called up in Highway Agency Specifications and their use is recommended in preference to the older methods. However results obtained with the BS 1377 and BS 1047 methods can still be used. Sulfate/sulfide distributions within a site tend to be variable and any difference between a TRL 447 test result and that obtained using the historical equivalent is unlikely to be of critical importance.

TRL 447 introduces several new acronyms/abbreviations, which may take some time to get used to. These include:

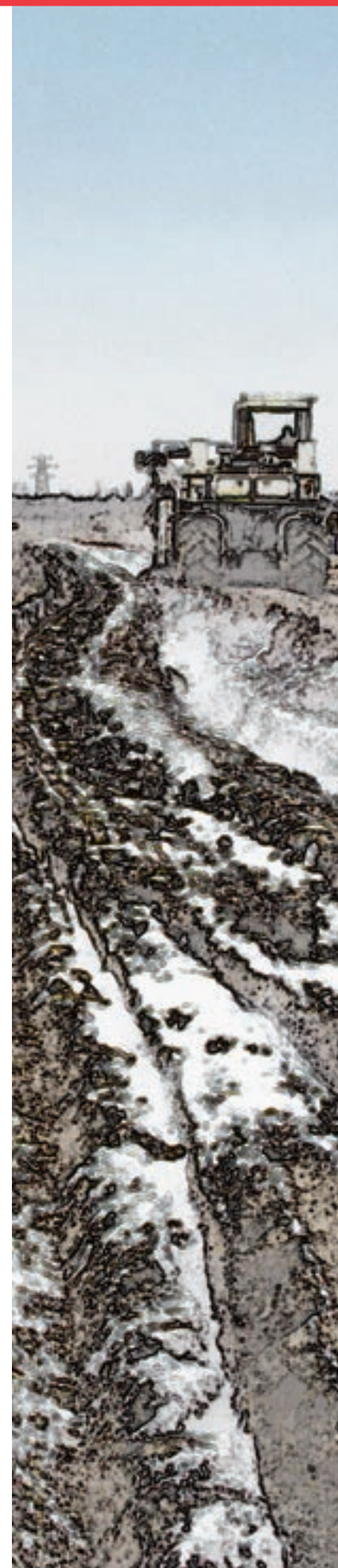
WSS	water-soluble sulfur	determined in 2:1 water extract and expressed as % S in the sample
WS	water-soluble sulfate	expressed as g/l SO_4 in the 2:1 water extract
ASS	acid-soluble sulfur	expressed as % S in the sample
AS	acid-soluble sulfate	expressed as % SO_4 in the sample
TS	total sulfur	expressed as % S in the sample
TPS	total potential sulfate	'TPS' is the TS content, converted to % SO_4
OS	oxidisable sulfide	'OS' is the TS content less any sulfur present as sulfate



The following table outlines the basis of the more commonly used tests and indicates their usefulness and limitations in regard to assessing the potential for sulfate disruption of stabilised soil.

Tests for sulfates and sulfides

Test	Water soluble sulfates 2:1 water:soil extract	Water soluble sulfates 10:1 water:soil extract	Acid soluble sulfates or 'total sulfate'	Total sulfur
Examples of test methods	BS 1377 Part 3 or TRL 447 Test No. 1: Water soluble sulfur (WSS)	Texas DoIT method: Tex-620-J	BS 1377 Part 3 or TRL 447 Test No. 2: Acid-soluble sulfur (ASS)	BS 1047 or TRL Test No. 4: Total sulfur (TS)
Method of extraction	The water soluble sulfates (WS) are extracted by shaking distilled water with the soil in the ratio 2:1 water:soil parts by weight for 16 hours.	The water soluble sulfates are extracted by shaking hot ('near boiling') distilled water with the soil in the ratio 10:1 parts by weight for 24 hours.	The sulfate is extracted using hydrochloric acid.	Various methods of extraction are employed but the aim of all methods is to extract and measure all the sulfur, irrespective of whether it is present as sulfate or sulfide.
How the results are expressed	BS 1377 results are expressed as g SO ₃ /l * in the water extract TRL 447 results are expressed as % S * in the soil sample (WSS) and also as g SO ₄ /l (WS) in the water extract .	The result is expressed as % SO ₄ in the soil sample.	BS 1377 results are expressed as % SO ₃ * in the soil. TRL 447 results are expressed as % S * in the soil sample (ASS) and also as % SO ₄ (AS) in the soil sample.	BS 1047 results are expressed as % S in the soil sample. TRL 447 results are expressed as %S (TS), as % SO ₄ (TPS) and also as % SO ₄ (OS), in the soil sample.
Comments	This test is of limited use for assessing the gypsum content of soils, because calcium sulfate is only sparingly soluble (1.5 g SO ₄ /l water). If the gypsum content of the soil is greater than 0.3% SO ₄ , any excess will not be extracted or measured.	By using a larger water:soil ratio it is possible to extract more gypsum. A ratio of 10:1 will be effective in extracting gypsum up to a content in the soil of 1.5% SO ₄ .	The acid extracts all the sulfate present in the soil, possibly including some that is not soluble in water. However, any slight 'overestimate' is unlikely to be significant. This method will not detect sulfide present in the soil. Sulfides have the potential to convert to sulfates and cause sulfate expansion.	This method measures all the sulfides/sulfates. Conversion of total sulfur to sulfate calculates the worst-case potential for sulfate but may somewhat overestimate the risk because chemical species such as organic sulfides, which are unlikely to contribute to sulfate expansion, are detected.
*To convert SO ₃ to SO ₄ , multiply by 1.2; to convert S to SO ₄ , multiply by 3.				





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