酸-硫酸-硝酸-酸混酸工控平台

Fast and reliable detection of phosphoric, sulfuric, nitric and hydrofluoric acids

Determination of the acid concentration in mixed acid solutions is a critical quality control step for successful etching processes. While primary analytical methods such as thermometric titration are well known, difficulties arise when mixtures of three or more acids need to be analyzed or if the time to result is a critical aspect. This application note discusses an alternative near-infrared (NIR) spectroscopy method that can reliably determine all parameters within a minute.

Ω Metrohm

EXPERIMENTAL EQUIPMENT

Mixed acid solutions based on four different acids (H_3PO_4, H_2SO_4, HNO_3, and HF) were measured in transmission mode with a DS2500 Liquid Analyzer over the full wavelength range (400–2500 nm). Disposable vials with a pathlength of 2 mm were used for convenient and fast measurement. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

Figure 1. DS2500 Liquid Analyzer and a sample filled in a disposable vial.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
DS2500 Liquid Analyzer	2.929.0010
DS2500 Holder 2 mm vials	6.7492.000
Disposable vials, 2 mm diameter, transmission	6.7402.070
Vision Air 2.0 Complete	6.6072.208

RESULTS

27 measured Vis-NIR spectra (**Figure 2**) were used to create a prediction model for quantification of the different acid concentrations (H_3PO_4, H_2SO_4, HNO_3, and HF). The quality of the prediction models was evaluated using correlation diagrams, which show a very high correlation between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.
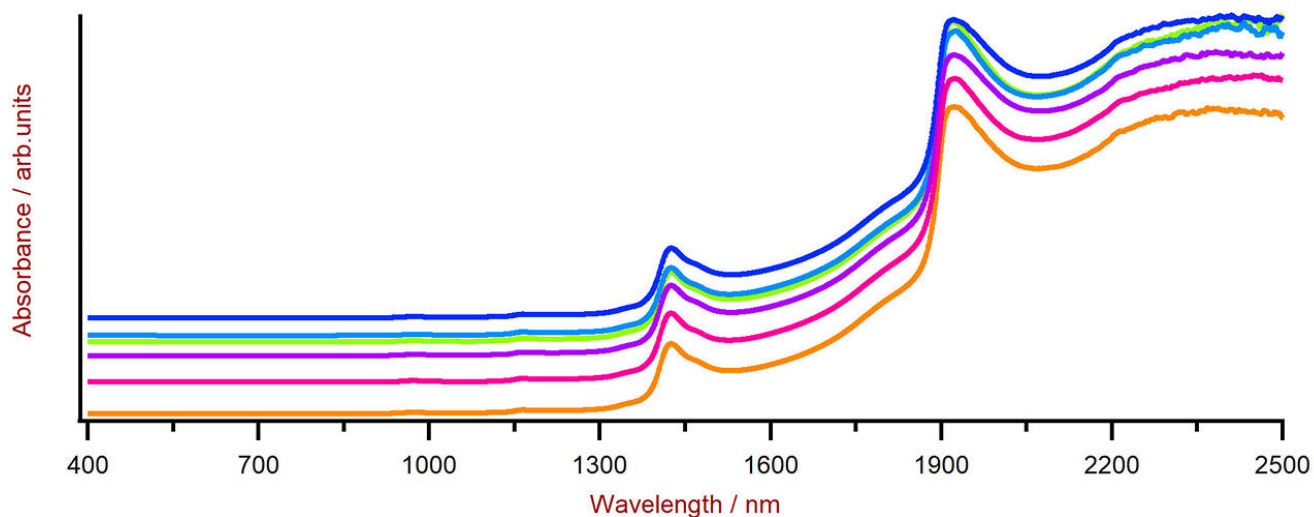
Ω **Metrohm**

Figure 2. Vis-NIR spectra of mixed acids solutions with varying acid content measured on a DS2500 Liquid Analyzer. For display reasons a spectra offset was applied.
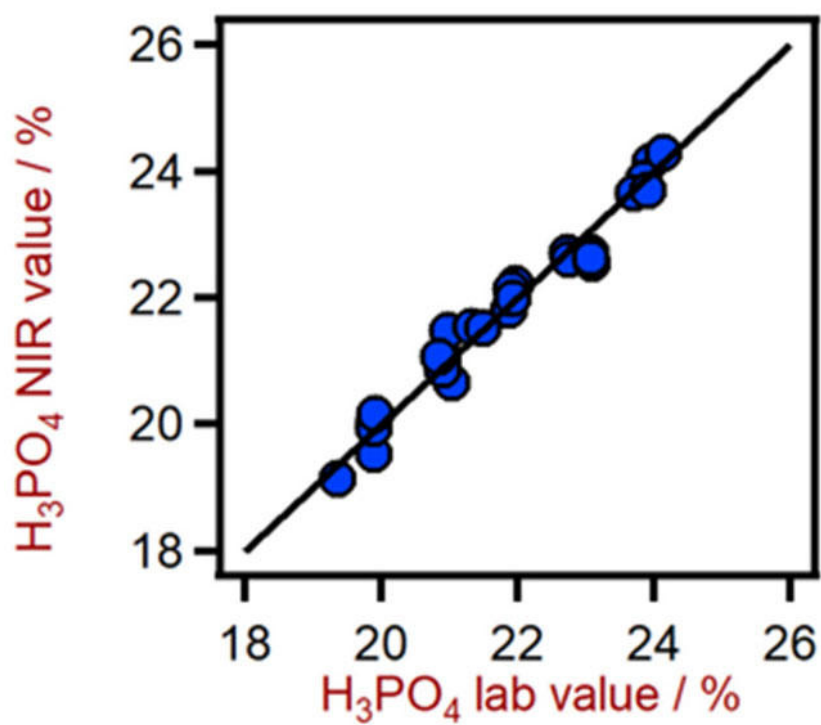

Figure 3. Correlation diagram for the prediction of H3PO4 content in a mixed acid solution using a DS2500 Liquid Analyzer.

Ω Metrohm

Table 2. Figures of merit for the prediction of H3PO4 content in a mixed acid solution using a DS2500 Liquid Analyzer.

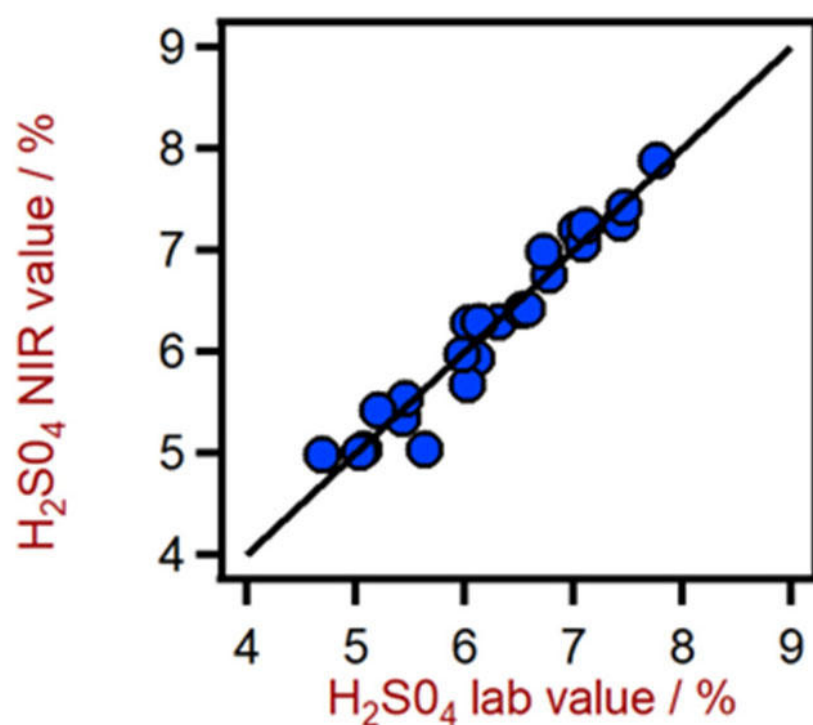| Figures of merit	Value
R^2	0.969
Standard error of calibration	0.290%
Standard error of cross-validation	0.410%

Figure 4. Correlation diagram for the prediction of H2SO4 content in a mixed acid solution using a DS2500 Liquid Analyzer.

Table 3. Figures of merit for the prediction of H2SO4 content in a mixed acid solution using a DS2500 Liquid Analyzer.

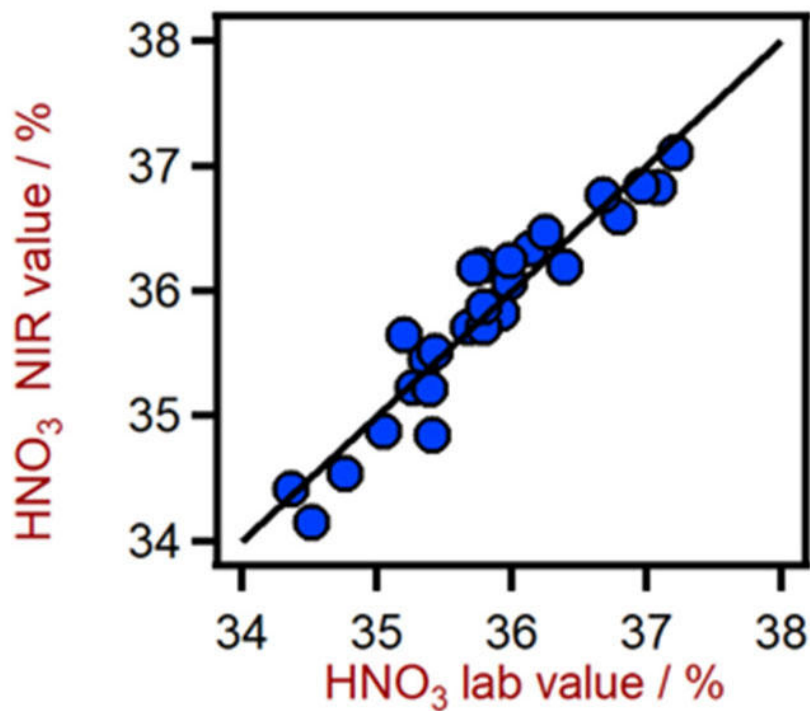| Figures of merit	Value
R^2	0.9448
Standard error of calibration	0.243%
Standard error of cross-validation	0.297%

Ω Metrohm

Figure 5. Correlation diagram for the prediction of HNO3 content in a mixed acid solution using a DS2500 Liquid Analyzer.

Table 4. Figures of merit for the prediction of HNO3 content in a mixed acid solution using a DS2500 Liquid Analyzer.

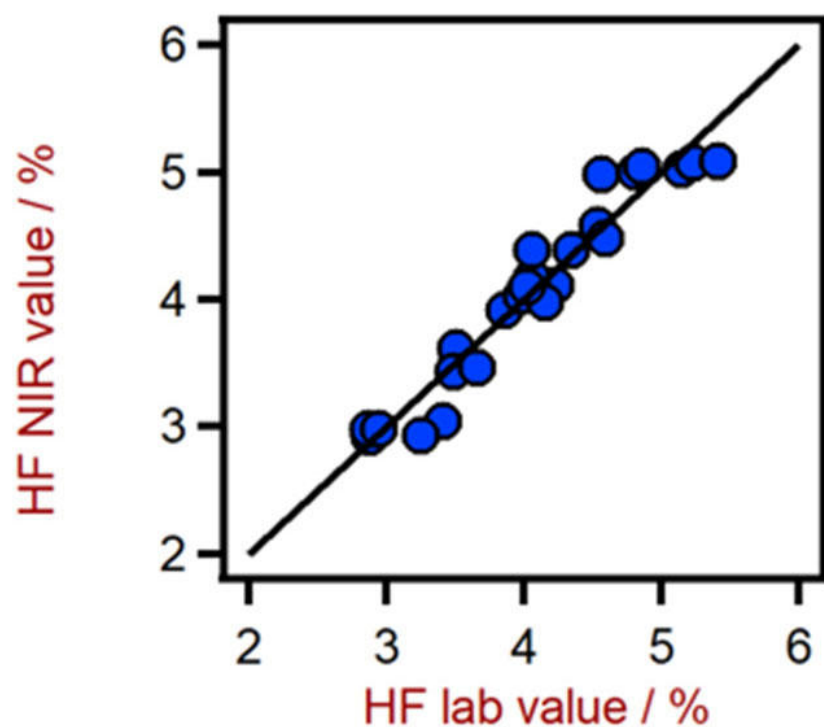| Figures of merit	Value
R^2	0.901
Standard error of calibration	0.279%
Standard error of cross-validation	0.345%

Ω **Metrohm**

Figure 6. Correlation diagram for the prediction HF content in a mixed acid solution using a DS2500 Liquid Analyzer.

Table 5. Figures of merit for the prediction of HF content in a mixed acid solution using a DS2500 Liquid Analyzer.

Figures of merit	Value
R^2	0.936
Standard error of calibration	0.211%
Standard error of cross-validation	0.276%

CONCLUSION

This application note demonstrates the feasibility of the DS2500 Liquid Analyzer for the determination of individual acid concentrations in a mixed acid solution. Vis-NIR spectroscopy enables fast determinations with high accuracy, and therefore represents a suitable alternative to the standard method (Table 6).

Ω Metrohm

Table 6. Time to result for the acid content determination of a mixed acid solution using thermometric titration and NIR spectroscopy.

Parameter	Method	Time to result and workflow
H_3PO_4, H_2SO_4, HNO_3, and HF content	Thermometric titration (three-fold determination)	~25 min. preparation for the determination of the titer and blank value + 12 min. (3 times 4 min.) for the titration measurement
H_3PO_4, H_2SO_4, HNO_3, and HF content	NIR Spectroscopy	1 minute for NIR spectroscopy measurement

Internal reference: AW NIR CN-0018-092020

CONTACT

117702
100085

marketing@metrohm.com.cn

DS2500 Liquid Analyzer
固的近外光,用于生境和室中的量。

DS2500 Liquid Analyzer 是一成熟且活的解决方案,其用于在整个生中行液体常分析。其固的使 DS2500 Liquid Analyzer 不受灰、潮湿、振的影,因此非常用于在劣的生境中使用。

DS2500 Liquid Analyzer 覆盖 400 至 2500 nm 的整个光范,将品加至 80°C 高温,并与各不同的一次性小瓶和石英比色皿兼容。因此,DS2500 Liquid Analyzer 可的个性化品要求,助在一分內得精和具有可重性的果。借助集成的品架装置和自的 Vision Air 件,保了用能松和安全地行操作。

如果是大的品量,可通将流通池与一个 Metrohm 机器人自器搭配使用的方法著提高生率。

Ω Metrohm

DS2500 2 mm
直径 2 mm 且更加智能的一次性玻璃小瓶支架

2 mm
200 个可封的玻璃(硼硅)一次性品瓶,直径 2 mm,用于分析透射中的液体。
兼容:
  - 支架 OMNIS NIR,小管,2 mm (**6.07401.050**)
  - DS2500 支架用于流池(**6.7492.000**)

Vision Air 2.0 Complete
Vision Air – 通用的光分析件。

Vision Air Complete 是用于管范境的先易用的件解决方案。

Vision Air 点一:
  - 便捷的件用和配的用界面保了直的操作方式
  - 操作程的建与方式
  - SQL 数据,可安全且地管理数据

Vision Air Complete (66072208) 版本包含所有用于可近外光分析量保程的用:
  - 器和数据管理用
  - 方法用
  - 常分析用

其它 Vision Air Complete 解决方案:
  - 66072207 (Vision Air Network Complete)
  - 66072209 (Vision Air Pharma Complete)
  - 66072210 (Vision Air Pharma Network Complete)

Ω Metrohm