A very sensitive LC-MS/MS assay was developed implementing a liquid-liquid extraction step followed by mass spectrometry which was operated in both positive and negative ion modes. The assay was calibrated with readily available commercial calibrators and compared with international reference standards. This data is also presented in "Sensitive Simultaneous Quantitation of Testosterone and Estradiol in Serum by LC-MS/MS without Derivatization and Comparison with the CDC HoSt Program" (Schofield et al., 2017). This article includes the comparison of the LC-MS/MS assay with a commonly available chemiluminescent immunoassay for the quantitation of both estradiol and testosterone. In addition we show baseline separation of estradiol and testosterone from other structurally related and/or isobaric compounds that could potentially interfere with the assay. In addition, various calibrator materials were tested and compared with internationally-recognized reference materials.

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Subject area | Clinical Chemistry
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More Specific subject area | Mass spectrometry and steroid measurement
Type of data | Graph, table and chromatogram
How data was acquired | LC-MS/MS and chemiluminescent immunoassay
Data format | Raw and processed data
Experimental factors | For LC-MS/MS analysis a serum sample was extracted with organic solvent. The organic extract was dried, reconstituted and injected into the LC/MS-MS system for analysis [1]. For immunoassay analysis human serum was placed onto the analyzer according to the manufacturer’s guidelines [2,3].
Experimental features | Specimens analyzed by LC-MS/MS for estradiol and testosterone were compared with a chemiluminescent immunoassays. Structurally related and/or isobaric compounds of estradiol and testosterone were evaluated for baseline separation. Various calibration materials were tested and compared with international reference standards.
Data source location | New York, NY, United States
Data accessibility | Data are available with this article

Value of data

- The data shows a comparison of testosterone by LC-MS/MS with a readily available chemiluminescent immunoassay at concentrations spanning the analytical measurement range.
- The data shows a comparison of estradiol by LC-MS/MS with a readily available chemiluminescent immunoassay at concentrations spanning the analytical measurement range.
- The data shows baseline separation of estradiol and testosterone from potentially interfering structurally related and/or isobaric compounds.

1. Data

The following data, Figs. 1 and 2, show the correlation between the LC-MS/MS assay and the chemiluminescent immunoassay for estradiol and testosterone. Fig. 3 shows baseline separation of estradiol and testosterone from potentially interfering structurally related and/or isobaric compounds. Figs. 4 and 5 show a comparison of calibration materials with international reference materials for estradiol and testosterone.

![Fig. 1.](image-url) (A) Correlation between the newly developed HPLC-MS/MS assay and the Architect i2000 immunoassay for testosterone; (B) Correlation between the newly developed HPLC-MS/MS assay and the Architect i2000 immunoassay at the low end of the analytical measurement range.
2. Experimental design, materials and method

2.1. Method comparison

The LC-MS/MS assay used to obtain the data set is given in [1]. The immunoassay used for testosterone is the Abbott Architect 2nd Generation Assay (Chicago, IL, USA). The assay is a chemiluminescent micro particle immunoassay for the quantitative determination of testosterone in human serum or plasma. This assay releases testosterone from binding proteins and measures the total testosterone level. This assay was performed according the manufacturer's guidelines [2]. The estradiol assay was from Abbott Diagnostic and was also a chemiluminescent immunoassay used for the quantitative determination of estradiol in human serum or plasma. This assay was performed according the manufacturer's guidelines [3].

2.2. Chromatographic separation

The LC-MS/MS parameters used in this study are given [1]. In order to rule out interference from structurally related and/or isobaric compounds, Chromsystems level III quality control from their MassCheck® Steroid Panel 1 [4] and Panel 2 [5] were combined. The analytes in this mixture were optimized for peak identification to ensure baseline separation from estradiol and testosterone.

2.3. Calibrator comparison with international reference material

For estradiol, certified reference material BCR 576, 577, and 578 was from the European Commission Institute for Reference Materials and Measurements (Geel, Belgium). For testosterone, standard reference materials SRM 971F and 971M were from the National Institute of Standards & Technology (Gaithersburg, MD, USA). Estradiol and testosterone were obtained from Sigma-Aldrich (Saint-Louis, MO, USA) and the calibrators were prepared gravimetrically. The commercially available Abbott Diagnostics (Chicago, IL, USA) and Chromsystems (Grafelfing, Germany) calibrators were prepared according to the manufacture’s instructions [2,3,6].
Fig. 3. (A) A positive ion chromatogram showing baseline separation of isobaric and structurally related compounds that have the potential to interfere with estradiol and testosterone. (B) A negative ion chromatogram showing baseline separation of isobaric and structurally related compounds that have the potential to interfere with estradiol and testosterone.
Funding sources

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Transparency document. Supporting information

Transparency data associated with this article can be found in the online version at https://doi.org/10.1016/j.dib.2018.08.077.

Fig. 4. Comparison of various calibrator materials to the European Commission Institute for Reference Materials and Measurements (IRMM) certified reference material (BCR 576, 577, and 578) for estradiol in human serum.

| Sample ID | Certified Value (pg/mL) | Measures value (N = 3) (pg/mL) | % Bias | CV |
|-----------|-------------------------|--------------------------------|--------|----|
| BCR 576   | 31.1                    | 31.3 (95% CI: 29.6-32.9)        | 0.7    | 0.6 |
| BCR 577   | 188.0                   | 186.7 (95% CI: 181.7-191.6)     | -0.7   | 2.3 |
| BCR 578   | 365.0                   | 358.8 (95% CI: 350.3-367.3)     | -1.7   | 2.1 |

Sigma-Aldrich Calibrators

| Samples   | Certified Value (pg/mL) | Measures value (N = 3) (pg/mL) | % Bias | CV |
|-----------|-------------------------|--------------------------------|--------|----|
| BCR 576   | 31.1                    | 30.9 (95% CI: 30.4-31.4)        | -0.5   | 1.3 |
| BCR 577   | 188.0                   | 190.6 (95% CI: 180.8-200.5)     | 1.4    | 4.6 |
| BCR 578   | 365.0                   | 365.5 (95% CI: 351.0-379.9)     | 0.1    | 3.5 |

Abbott Calibrators

| Samples   | Certified Value (pg/mL) | Measures value (N = 3) (pg/mL) | % Bias | CV |
|-----------|-------------------------|--------------------------------|--------|----|
| BCR 576   | 31.1                    | 38.0 (95% CI: 37.3-38.6)        | 22.3   | 1.6 |
| BCR 577   | 188.0                   | 189.2 (95% CI: 183.5-194.8)     | 0.6    | 2.6 |
| BCR 578   | 365.0                   | 361.5 (95% CI: 338.8-384.1)     | -1.0   | 5.5 |

Fig. 5. Comparison of various calibrator materials to the National Institute of Standards & Technology (NIST) standard reference material (SRM 971F and SRM 971M) for testosterone in human serum.

| Samples   | Certified Value (ng/dL) | Measures value (N = 3) (ng/dL) | % Bias | CV |
|-----------|-------------------------|--------------------------------|--------|----|
| SRM 971 F | 27.7                    | 27.6 (95% CI: 27.1-28.1)        | -0.4   | 1.5 |
| SRM 971 M | 642.9                   | 624.7 (95% CI: 624.8-656.4)     | -0.4   | 2.2 |

Sigma-Aldrich Calibrators

| Samples   | Certified Value (ng/dL) | Measures value (N = 3) (ng/dL) | % Bias | CV |
|-----------|-------------------------|--------------------------------|--------|----|
| SRM 971 F | 27.7                    | 27.5 (95% CI: 27.0-28.0)        | -0.7   | 1.2 |
| SRM 971 M | 642.9                   | 631.4 (95% CI: 615.8-647.0)     | -1.8   | 1.6 |

Abbott Calibrators

| Samples   | Certified Value (ng/dL) | Measures value (N = 3) (ng/dL) | % Bias | CV |
|-----------|-------------------------|--------------------------------|--------|----|
| SRM 971 F | 27.7                    | 30.3 (95% CI: 29.8-30.8)        | 9.4    | 0.3 |
| SRM 971 M | 642.9                   | 692.2 (95% CI: 675.1-709.3)     | 7.7    | 1.7 |

Funding sources

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[5] MassCheck® Steroid Panel 2 Serum Control, Level III [Package Insert], Chromsystems, Grafelfing, Germany, 2014.
[6] Placebo's Multilevel Serum Calibrator Set MassChrom® Steroid Panel 2 [Package Insert], Chromsystems, Grafelfing, Germany, 2014.